



**TÜRKİYE CUMHURİYETİ  
ADANA ALPARSLAN TÜRKER SCIENCE AND TECHNOLOGY  
UNIVERSITY**

**GRADUATE SCHOOL  
MECHANICAL ENGINEERING DEPARTMENT**

**PREDICTION AND OPTIMIZATION OF HYDROGEN PRODUCTION**

**ŞEKİP CANER ESERLİ**

**M.Sc.**



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**M.Sc.**

**THESIS ADVISOR  
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**ADANA, 2024**

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06/12/2024

[Signature]

Şekip Caner ESMERLİ

# ÖZET

## HİDROJEN ÜRETİMİNİN TAHMİNİ VE OPTİMİZASYONU

Şekip Caner ESMERLİ

Yüksek Lisans, Makine Mühendisliği Anabilim Dalı

Danışman: Doç. Dr. Ceyla GÜNGÖR

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Modern dünyanın en önemli iki sorunu, bir yandan uygun fiyatlı, kolay ulaşılabilir ve bol enerji kaynaklarının bulunması, diğer yandan ise sanayi devriminden bu yana sürekli artan çevre kirliliğidir. Fosil enerji kaynakları dünya enerji talebinin büyük bir kısmını karşılayabilmesine rağmen, fosil kaynakların sınırlı olması ve çevreyi ciddi şekilde kirletmesi gibi sorunlar nedeniyle alternatif enerji kaynaklarına yönelmek zorunlu hale gelmiştir. Bu nedenle günümüzde yenilenebilir enerji kaynaklarına olan ilgi artmaktadır. Bu kaynaklar arasında başlıca güneş enerjisi, rüzgâr enerjisi ve hidrojen enerjisi yer almaktadır. Yenilenebilir enerji kaynaklarının en büyük zorluklarından biri depolama sorunudur. Bu soruna çözüm olarak hidrojen, uzun vadede önemli bir alternatif olarak öne çıkmaktadır. Diğer yenilenebilir enerji kaynakları kullanılarak hidrojeni üretmek ve depolamak mümkündür. Suyun elektroliz yoluyla parçalanmasıyla elde edilen hidrojene yeşil hidrojen denir. Fosil yakıtlarla üretilen hidrojene kıyasla, yeşil hidrojen çok daha az emisyon gazı üretir.

Bu tezin amacı tepki yüzey yöntemi (RSM) ile suyun elektroliz yoluyla ayrıştırılarak hidrojen üretiminde farklı elektrotlar kullanılarak elde edilen sonuçların optimize edilmesidir. Bu çalışmada alkalın elektroliz hücresi kullanılarak hidrojen gazı üretimi için laboratuvar ölçeğinde iki elektrotlu bir sistem kurulmuştur. İlk başta grafit anot, hafif çelik elektrotlar ise katot olarak kullanılmıştır. Daha sonra ise bir soygun metal olan platin anot olarak kullanılmış, grafit elektrotlar ise katot olarak tercih edilmiştir. Bu grafit elektrotlar, bir galvanostatik yöntemle nikel-kobalt-molibden (NiCoMo) ile modifiye edilmiştir. Her iki çalışmada da Elektrot voltajı ve molarite süresi bağımsız faktörler olarak seçilmiştir. Design-Expert programı (deneme sürümü) kullanılarak, bu bağımsız faktörlerin hidrojen üretimi üzerindeki etkileri

istatistiksel olarak yorumlanmıştır. Bu yöntemin başlıca avantajlarından biri, çeşitli parametreleri küçük bir deneysel performans sayısı ile inceleyebilmesidir.

Deneysel sonuçlar, yapılan tüm deneylerde voltaj değeri 2,5 volttan 3 volta çıktıkça üretilen hidrojen miktarının arttığını göstermiştir. Benzer şekilde tüm deneylerde molarite 0,5 molar konsantrasyondan 1,5 molar konsantrasyona çıktıkça üretilen hidrojen miktarında da artış gözlenmektedir. NiCoMo modifiye grafit elektrot için optimum arzu edilebilirlik 3V voltaj ve 1.5 M molalite gibi ideal koşullarda üretilen 10.67 mL hidrojenle 0.890 olarak bulunmuştur.  $R^2$  katsayısı açısından, düzeltilmiş  $R^2$  ve tahmin edilen  $R^2$  sırasıyla %98,81, %97,96 ve %91,63 olarak belirlenmiştir. Doğrulama test sonuçları, deneysel olarak elde edilen hidrojen hacimleri ile tahmin edilen hidrojen hacimleri arasında iyi bir uyum olduğunu göstermiştir.

**Anahtar Kelimeler:** Hidrojen, elektroliz, tahmin, optimizasyon, RSM.

# **ABSTRACT**

## **PREDICTION AND OPTIMIZATION OF HYDROGEN PRODUCTION**

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M.Sc., Department of Mechanical Engineering

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Two of the most important issues in the modern world are, on one hand, the availability of affordable, easily accessible, and abundant energy sources, and on the other hand, the continuously increasing environmental pollution since the Industrial Revolution. Although fossil energy sources meet a large portion of the world's energy demand, the limitations of these sources and their significant environmental pollution have made it necessary to seek alternative energy sources. As a result, there is a growing interest in renewable energy sources today. Among these sources, solar, wind, and hydrogen energy are prominent. One of the biggest challenges of renewable energy sources is the storage issue. Hydrogen emerges as a significant long-term alternative to address this problem. It is possible to produce and store hydrogen using other renewable energy sources. Hydrogen obtained through the electrolysis of water is known as green hydrogen. Compared to hydrogen produced from fossil fuels, green hydrogen produces significantly fewer emission gases.

This thesis aims to optimize the results obtained by using different electrodes in hydrogen production through the electrolysis of water with the response surface method (RSM). In this study, a laboratory-scale two-electrode system was set up for hydrogen gas production using an alkaline electrolysis cell. Initially, graphite was used as the anode and mild steel electrodes as the cathode. Later, platinum, a noble metal, was used as the anode, while graphite electrodes were preferred as the cathode. These graphite electrodes were modified with nickel-cobalt-molybdenum (NiCoMo) using a galvanostatic method. In both studies, electrode voltage and molarity time were selected as independent factors. Using the Design-Expert program (trial version), the effects of these independent factors on hydrogen production were statistically

interpreted. One of the main advantages of this method is that it allows for the examination of various parameters with a small number of experimental performances.

Experimental results have shown that the amount of hydrogen produced increased as the voltage value rose from 2.5 volts to 3 volts in all experiments. Similarly, an increase in hydrogen production was observed as the molarity increased from 0.5 M to 1.5 M across all experiments. For the NiCoMo-modified graphite electrode, the optimum desirability was found to be 0.890 with 10.67 mL of hydrogen produced under ideal conditions of 3V voltage and 1.5 M molarity. In terms of the coefficient of determination,  $R^2$ , the adjusted  $R^2$  and predicted  $R^2$  values were determined to be 98.81%, 97.96%, and 91.63%, respectively. Validation test results demonstrated a good agreement between the experimentally obtained hydrogen volumes and the predicted hydrogen volumes.

**Keywords:** Hydrogen, electrolysis, prediction, optimization, RSM.

### ***Dedication***

*I dedicate this thesis to my mother, who supported me throughout my graduate education, and to my nephews Bora and Kaan, who constantly encouraged me.*





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## LIST OF ABBREVIATIONS

|                |                                            |
|----------------|--------------------------------------------|
| RSM            | : Response Surface Methodology             |
| CCD            | : Central Composite Design                 |
| $E_d$          | : Decomposition Voltage                    |
| V              | : Voltage                                  |
| M              | : Molarity                                 |
| L              | : Liter                                    |
| mL             | : Milliliter                               |
| %              | : Percent                                  |
| G              | : Graphite                                 |
| Ni             | : Nickel                                   |
| Co             | : Cobalt                                   |
| Mo             | : Molybdenum                               |
| G/NiCoMo       | : Nickel cobalt molybdenum coated graphite |
| Pt             | : Platinum                                 |
| H <sub>2</sub> | : Hydrogen                                 |
| O <sub>2</sub> | : Oxygen                                   |
| KOH            | : Potassium Hydroxide                      |
| SEM            | : Scanning Electron Microscope             |
| EDX            | : Energy Dispersion X-ray                  |
| ANOVA          | : Analysis of Variance                     |
| R <sup>2</sup> | : Correlation Coefficient                  |

# 1. INTRODUCTION

Energy is the ability of a system or an object to do work. Energy can exist in various physical forms and can transform into new forms. Kinetic energy, or the energy of motion, potential energy, or the energy of position, thermal energy, chemical energy, electrical energy, and nuclear energy are the most prevalent types of energy.

According to the law of conservation of energy, energy can only be changed from one form to another and cannot be created or destroyed. This suggests that energy is a universal constant and that in a closed system, the total energy stays constant.

Energy also plays an important role in daily life. In our homes, we use electrical energy to illuminate, heat, and power electronic devices. In our cars, chemical energy is used as fuel, converting into kinetic energy and allowing us to move. Our bodies use the chemical energy they get from food to maintain their vital functions.

In the field of industry and technology, energy is also at the center of production processes. Factories use electricity and other forms of energy to run machines. The importance of renewable energy sources in lowering reliance on fossil fuels is growing and minimizing environmental damage. Renewable energy sources, including biomass, hydroelectric power, solar energy, and wind energy, are crucial to the generation of sustainable energy.

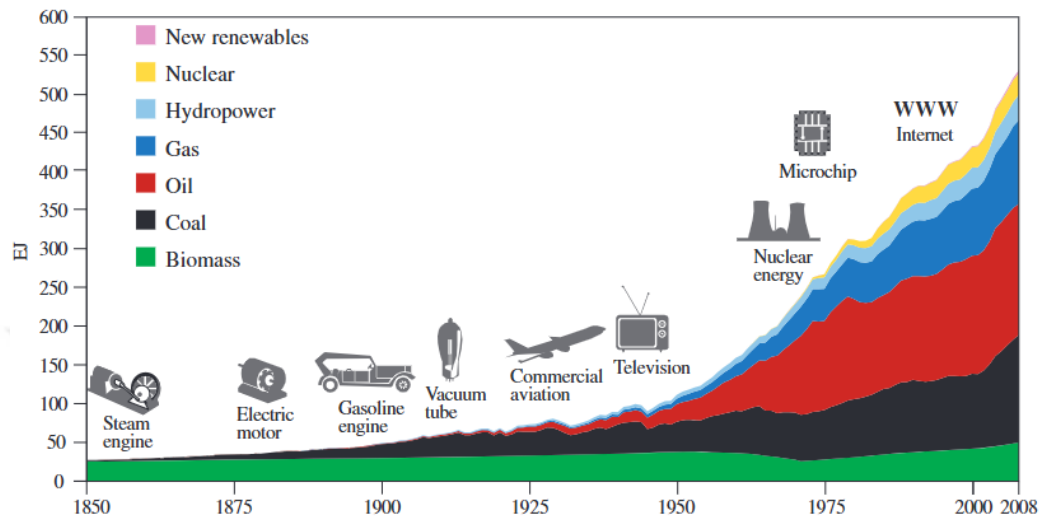
In conclusion, energy is a fundamental component of life and the universe. Defined as the ability to do work, energy can be found and transformed in different forms, making it a universal and vital concept.

## 1.1. History of Energy

The history of energy is a story intertwined with the development of humanity. Since the earliest times, humans have harnessed various energy sources to improve their living standards and advance civilizations. The discovery of fire marked the first major step in energy use, providing heat, light, and meeting basic needs such as cooking and protection (Bithas, 2016). Additionally, early societies converted wind and water power into mechanical energy through sailing ships and watermills.



During the Middle Ages, the use of water and wind power became more widespread. Watermills and windmills were employed for grinding grain and pumping water, providing mechanical energy for various processes. However, energy consumption remained limited and localized (Lucas, 2005).



**Figure 1.1.** History of world primary energy use, by Source (Grubler et al., 2012)

The Industrial Revolution in the late 18th century brought about a major transformation in energy use. The invention of the steam engine allowed coal to be utilized as a large-scale energy source, revolutionizing industries such as textile manufacturing, railways, and ships (Sicilia, 1986). This pivotal shift dramatically increased energy consumption and laid the foundation for industrialization and urbanization.

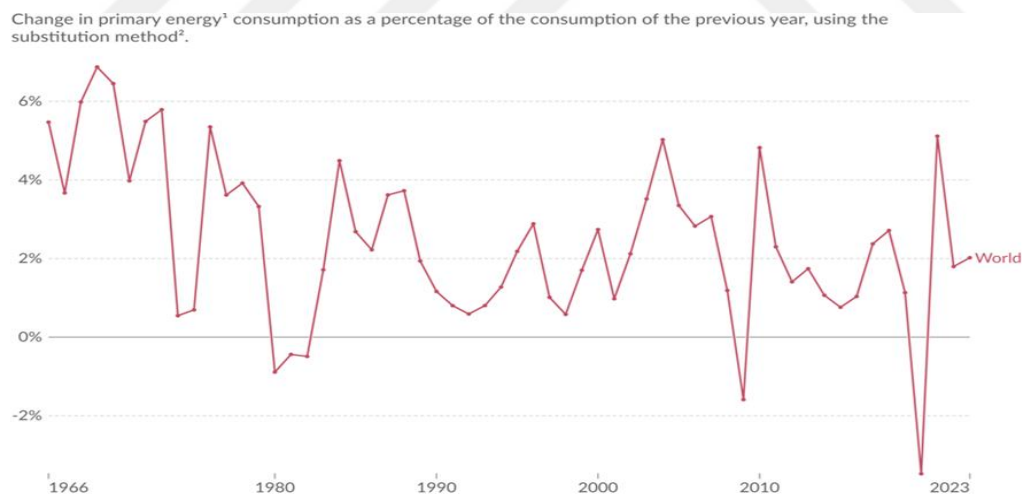
The late 19th and early 20th centuries saw the discovery and widespread use of electricity. Pioneers like Thomas Edison and Nikola Tesla played critical roles in developing the production and distribution of electricity, which began to power homes for lighting, heating, and various electrical devices (Gooday, 2009). This era also marked the rise of oil, particularly for transportation, as the internal combustion engine enabled the growth of automobiles and aviation (Yergin, 1991).

The mid-20th century introduced nuclear energy as a new frontier in electricity generation. Nuclear reactors, developed in the 1940s, offered the potential for producing large amounts of energy. However, concerns over environmental impact and safety, particularly after accidents

such as Chernobyl and Fukushima, have constrained the expansion of nuclear energy (Právělie, 2018; Rashad, 2000).

In recent decades, growing awareness of environmental impacts and the depletion of fossil fuels have driven increased interest in renewable energy sources, such as biomass, solar, wind, and hydropower (Holechek, 2022). Technological advancements have improved the efficiency of these renewable sources while reducing costs (Koroneos, 2003). Countries around the world have begun shifting toward low-carbon energy systems, driven by environmental policies and the need to combat climate change (Fouquet, 2010).

The 21st century is characterized by the rapid growth of renewable energy technologies and infrastructure. Innovations in solar and wind energy have made these sources increasingly competitive with fossil fuels. Advances in battery storage and smart grids are enabling better integration of renewables into the power grid, addressing challenges like intermittency (IRENA, 2020; IEA, 2020). This energy transition also has geopolitical implications, as countries rich in renewable resources gain strategic importance, while oil-exporting nations diversify their economies (Månberger & Stenqvist, 2018).



**Figure 1.2.** Annual change in primary energy consumption (Energy Institute - Statistical Review of World Energy 2024)

Financial development has been an essential driver of renewable energy growth, particularly in emerging economies where investments are needed to overcome the initial high costs of these

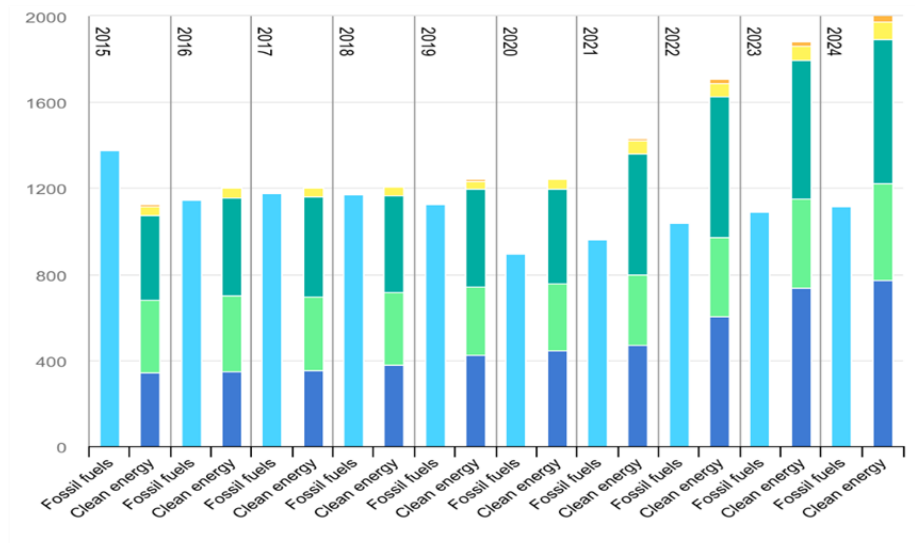
technologies (Kor, 2023). Technological advancement has sped up the use of renewable energy, particularly in nations like China, leading to increased energy security and sustainability (Shi, 2017). Digital technologies integrated into renewable energy systems have further optimized production and consumption (Su et al., 2022).

However, there are difficulties involved in making the switch to renewable energy. High upfront investment expenses, regulatory barriers, and the need for substantial infrastructure investments are some of the obstacles that prevent the general implementation of renewable energy (Maradin, 2021). Nevertheless, Governments have put in place programs including tax breaks, feed-in tariffs, and renewable portfolio standards to promote the private sector participation and increase investment (Kabel & Bassim, 2019).

Environmental considerations remain at the forefront of this energy transition. To cut greenhouse gas emissions, a switch to renewable energy sources is essential and mitigating climate change. Countries like India and China have already made significant strides in cutting carbon emissions through renewable energy adoption (Tabassum & Shastry, 2021).

As the world continues to move toward environmentally friendly and sustainable energy sources, smart grids, energy storage solutions, and energy efficiency technologies are becoming key areas of focus. Research into new energy sources, such as fusion energy, also holds promise for revolutionizing future energy production (Burkart, 2005).

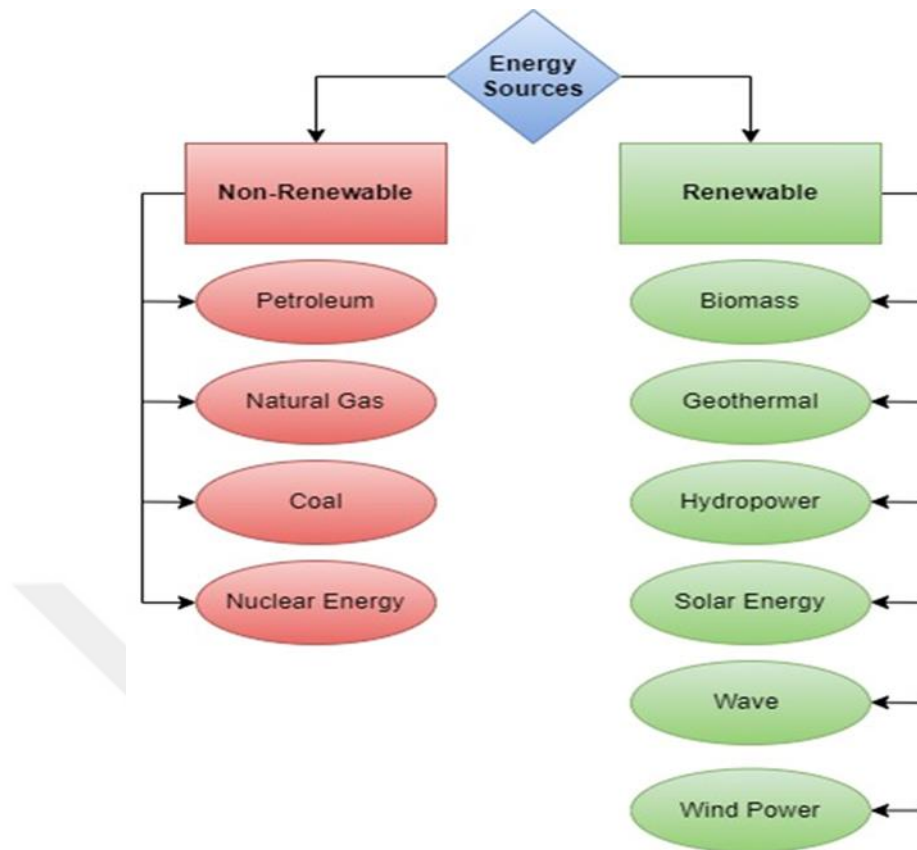
In conclusion, the history of energy reflects humanity's ongoing efforts to harness natural resources for progress. From the discovery of fire to the development of renewable energy, technological innovations have shaped the trajectory of energy use. Today, sustainability and environmental responsibility are fundamental elements shaping the energy policies of the future (Ribeiro et al, 2013). The path forward requires coordinated efforts to overcome existing challenges and fully realize the potential of renewable energy to achieve a sustainable and resilient energy future.



**Figure 1.3.** Global investment in clean energy and fossil fuels, (Internal Energy Agency 2015-2024)

## 1.2. Energy Resources

Energy resources can be broadly classified into two categories: renewable and non-renewable. Historically, non-renewable energy sources including coal, oil, and natural gas have dominated the world's energy production, particularly since the Industrial Revolution, because of their high energy density and ease of transport and storage (Smil, 2017). However, the environmental impacts of fossil fuels, especially their addition to greenhouse gas emissions and climate change, have led to a growing shift toward renewable energy sources, including solar, hydropower, wind, and biomass (IPCC, 2021).



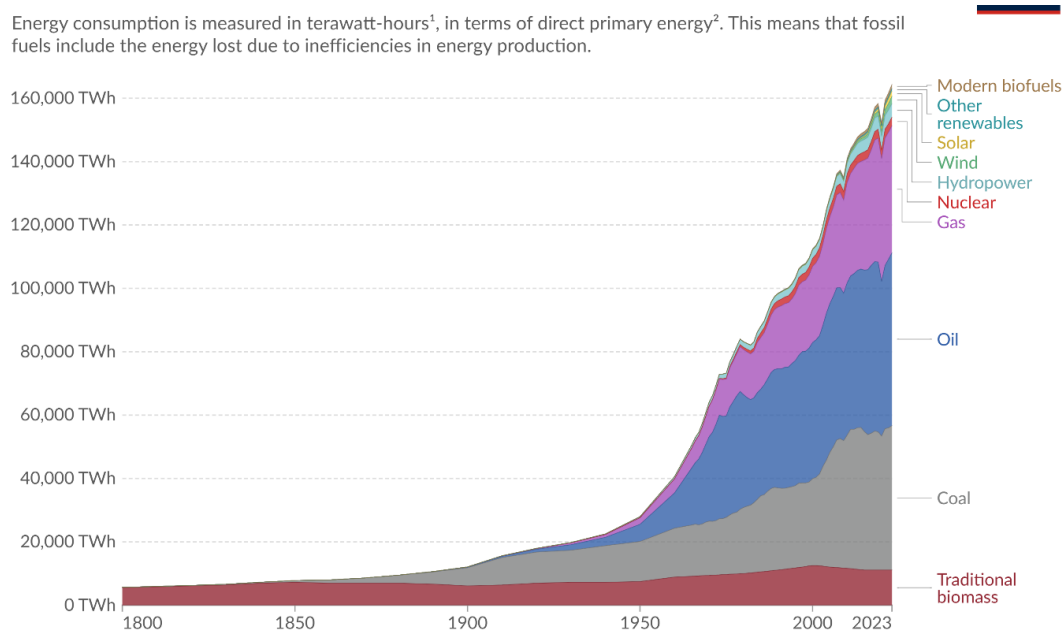
**Figure 1.4.** Diagram of energy resources

The exploration of energy sources is crucial in understanding the dynamics of energy production and consumption globally. Non-renewable energy sources, including fossil fuels, contribute significantly to environmental degradation through greenhouse gas emissions (Cîrstea et al., 2018; Gupta, 2023; Eboibi et al., 2018). In contrast, renewable energy sources, like solar, wind, hydro, geothermal, and biomass energy, come from naturally occurring processes that replenish themselves continuously (Bogaraj, 2018). This distinction is vital as the world grapples with the pressing must make the switch to more environmentally friendly energy systems to lessen the effects of climate change and reliance on fossil fuels (Cavalcanti, 2023; Xin et al., 2022).

### 1.2.1. Non-renewable energy sources

Non-renewable energy resources are finite natural sources that are consumed faster than they can be replenished. These resources have been the backbone of global energy systems since the Industrial Revolution, powering much of the world's transportation, industrial, and electricity

sectors. However, their finite nature and environmental impacts pose long-term challenges for sustainable energy development. The two primary categories of non-renewable energy resources are nuclear energy and fossil fuels, which include coal, oil, and natural gas.



**Figure 1.5.** Global direct primary energy consumption (Energy Institute - Statistical Review of World Energy, 2024; Smil, 2017)

Fossil fuels, which include coal, oil, and natural gas, are formed from the remains of fossilized plants and animals that were heated and compressed for millions of years in the Earth's crust. These fuels are highly energy-dense and have dominated global energy systems since the 18th century (Abas, 2015). However, their use contributes significantly to greenhouse gas accumulation in the atmosphere, leading to serious environmental issues including climate change and global warming (IPCC, 2021).

Coal is the most common and historically significant fossil fuel. It fueled the Industrial Revolution and continues to play a major role in electricity generation, particularly in countries like China, India, and the United States (Dai, 2017). But coal also produces the most pollutants of all the fossil fuels, including sulfur dioxide (SO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), and carbon dioxide (CO<sub>2</sub>). These pollutants lead to acid rain and air pollution. (Smil, 2017). Coal use has declined

in many areas due to environmental concerns and the availability of cleaner alternatives, but it remains an important energy source in many developing economies (Bilgen, 2016).

Oil, or crude oil, is a liquid fossil fuel that has been the world's primary energy source for transportation since the early 20th century (Heinberg, 2016). It is processed to create a variety of goods, including jet fuel, diesel, and gasoline, and petrochemicals. Oil is central to the global economy, and its production is concentrated in a few regions, notably the Middle East, which has led to significant geopolitical implications (Yergin, 1991). The burning of oil releases large amounts of CO<sub>2</sub>, contributing to climate change, while oil spills and extraction methods such as offshore drilling and hydraulic fracturing pose serious environmental risks.

Natural gas is primarily made up of the powerful greenhouse gas methane (CH<sub>4</sub>), and is considered the cleanest of the fossil fuels due to its lower CO<sub>2</sub> emissions when burned, relative to coal and oil. It is widely used for electricity generation, heating, and as an industrial fuel. The use of natural gas has surged in recent years, particularly in the United States, where advancements in hydraulic fracturing have unlocked vast reserves of shale gas. However, methane leaks during extraction, processing, and transportation are a major concern, because methane has a considerably greater potential to cause global warming than CO<sub>2</sub> over a shorter period of time (IEA, 2021).

Nuclear energy is another significant non-renewable resource, derived by means of nuclear fission. To do this, a heavy atom, usually uranium-235, must split its nucleus in order to release a significant quantity of energy. Nuclear energy is considered low carbon because it does not produce direct CO<sub>2</sub> emissions during electricity production, making it an ecologically friendly alternative to fossil fuels in terms of emissions. Despite its low-carbon benefits, nuclear energy faces several challenges. The most important is handling radioactive waste, which needs to be stored securely for a long time because it might remain dangerous for thousands of years. Another major issue is the risk of nuclear accidents, such as those that occurred at Chernobyl (1986) and Fukushima (2011), which have raised concerns about the safety of nuclear power. Additionally, the cost of constructing and maintaining nuclear power plants is high, and public opposition remains strong in many regions due to safety and environmental concerns (Garrick, 2008).

Non-renewable energy sources, like nuclear power and fossil fuels, emerge naturally over millions of years and cannot be replenished on a timeline that is suitable for humans. As these resources are depleted, energy production costs will rise, and the environmental consequences will become more severe (Sitbon et al., 2019). Although non-renewable energy sources still dominate the global energy supply, they do not offer a sustainable long-term solution due to their environmental impacts and limited reserves.

### **1.2.2. Renewable energy sources**

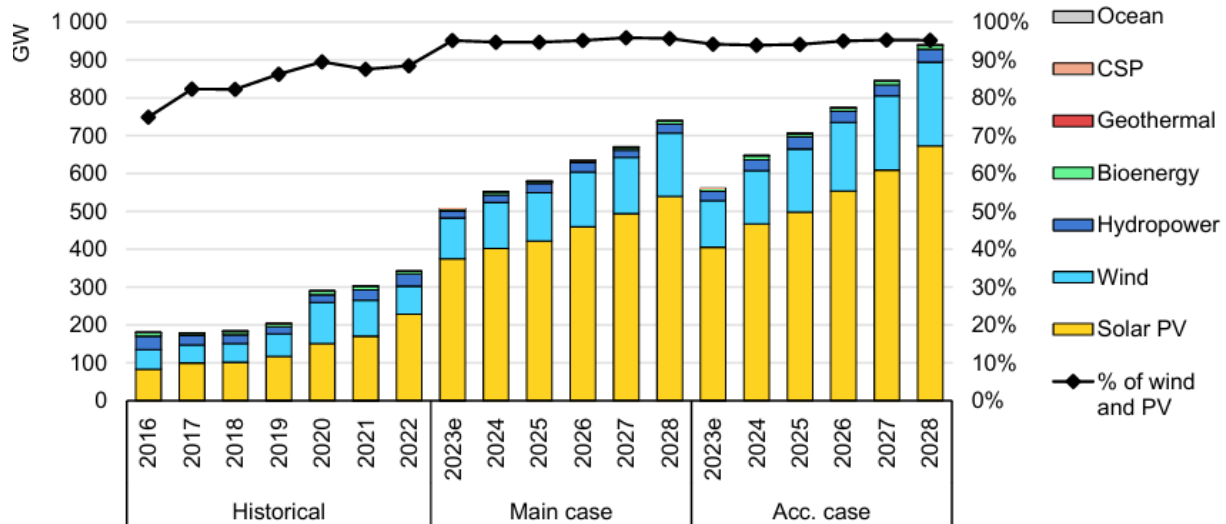
Renewable energy sources come from naturally occurring processes that regenerate themselves over time on human timescales. As opposed to fossil fuels, which form over millions of years, renewable resources are practically inexhaustible and have a much smaller environmental impact. They are essential to reducing the effects of climate change by providing clean alternatives to carbon-intensive energy sources. The main sources of renewable energy are geothermal, biomass, hydropower, solar, and wind. Each of these sources has unique characteristics, advantages, and challenges that influence their adoption and integration into energy systems worldwide.

The Intergovernmental Panel on Climate Change (IPCC) has identified six renewable energy sources with significant global potential: bioenergy, direct solar energy, geothermal energy, hydropower, ocean energy, and wind energy (Shields, 2014). These sources are considered inexhaustible due to their continuous regeneration, making them crucial for achieving sustainable development goals (Ravichandran et al., 2022).

Renewable energy sources are increasingly recognized for their potential to provide clean energy without the harmful emissions associated with fossil fuels. For instance, solar energy harnesses sunlight through photovoltaic cells or solar thermal systems, while wind energy captures kinetic energy from wind currents using turbines (Genç, 2023; Ayorinde & Olasebikan, 2020). Hydropower, another significant renewable source, utilizes flowing water to generate electricity, contributing to a substantial portion of the energy mix in many countries (Shcherbak et al., 2021; Pedraza, 2014). Biomass energy, derived from organic materials, also plays a crucial role, particularly in rural areas where it serves as a primary energy source for cooking and heating (Okoye et al., 2017; Sultana et al., 2018). The diversification of energy



sources is essential for enhancing energy security and reducing vulnerability to supply disruptions (Yuaningsih et al., 2020).



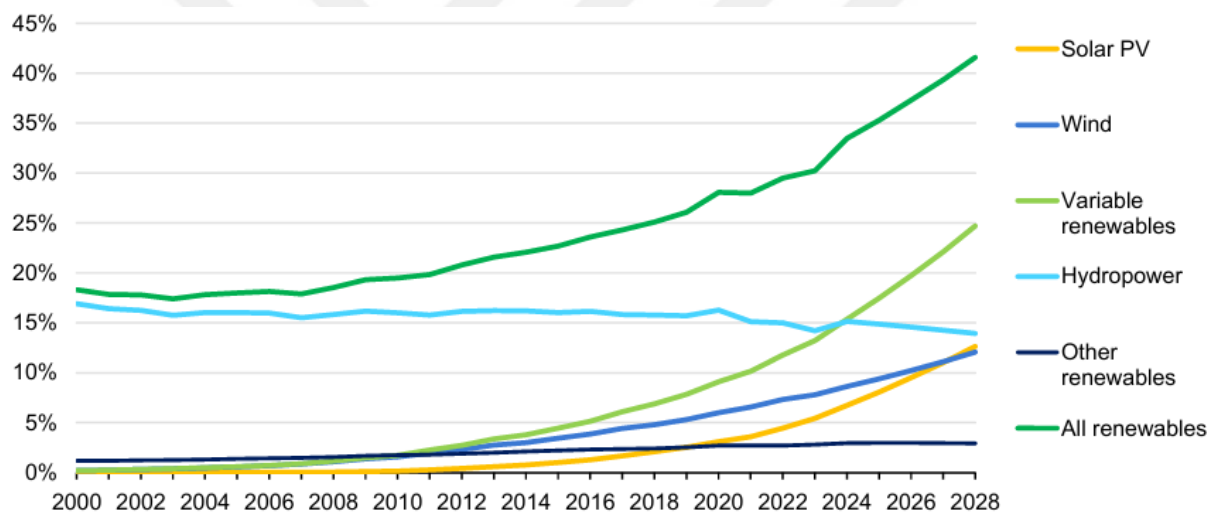
**Figure 1.6.** Renewable electricity capacity additions by technology and segment, (IEA, 2023)

In order to achieve sustainability, renewable energy is essential because it offers plentiful, clean, and low-carbon substitutes for fossil fuels, hence reducing environmental degradation and mitigating climate change. As renewable energy sources like solar, wind, hydropower, and geothermal emit little to no greenhouse gases during operation, they are essential in limiting global warming to well below 2°C, as outlined in the Paris Agreement (UNFCCC, 2015). Additionally, renewable energy also strengthens energy security by reducing reliance on fossil fuels, which are limited and geopolitically sensitive, fostering energy independence for many countries. Beyond environmental benefits, the deployment of renewable technologies creates economic opportunities, generating green jobs in the manufacturing, installation, and maintenance sectors. However, ensuring the sustainability of renewable energy requires addressing challenges such as resource extraction for materials like lithium for batteries, land use for wind and solar farms, and managing the lifecycle of renewable technologies (IRENA, 2018). Thus, renewable energy is central to the transition to a more sustainable, resilient, and equitable global energy system.

The integration of these renewable energy sources into existing energy systems presents both opportunities and challenges. The transition to renewable energy is driven by the need to reduce

greenhouse gas emissions, enhance energy security, and promote sustainable development (Huseynli, 2024; Sipa & Gorzeń-Mitka, 2021). However, the intermittent nature of solar and wind energy necessitates the development of energy storage solutions and smart grid technologies to ensure reliability and stability in energy supply (Sayed et al., 2023; Balsalobre-Lorente et al., 2018).

In conclusion, the utilization of renewable energy sources is vital in tackling worldwide energy issues and advancing sustainable development. The diverse range of renewable technologies offers various pathways for energy generation, each with its advantages and challenges. As technological advancements continue and policy frameworks evolve, the potential for renewable energy to contribute significantly to the global energy mix is increasingly recognized.



**Figure 1.7.** Electricity generation by technology, 2000-2028 (IEA, 2023)

Reducing the use of fossil fuels and nuclear energy and replacing it with renewable energy sources has become one of the main goals of global energy policies. In this context, increasing investments in renewable energy sources will play a critical role in building a sustainable future.

Renewable energy sources, such as solar, wind, hydro, and biomass energy, provide sustainable alternatives to non-renewable energy sources that can be renewed over time without causing the same level of environmental degradation ("Advanced Energy Sources", 2019).

Renewable energy resources are essential in the global energy landscape, providing sustainable alternatives to traditional fossil fuels. These resources include wind energy, solar energy (comprising thermal, photovoltaic, and concentrated forms), hydroelectric power, tidal energy, geothermal energy, biofuels, and the renewable component of waste (Radu et al., 2019).

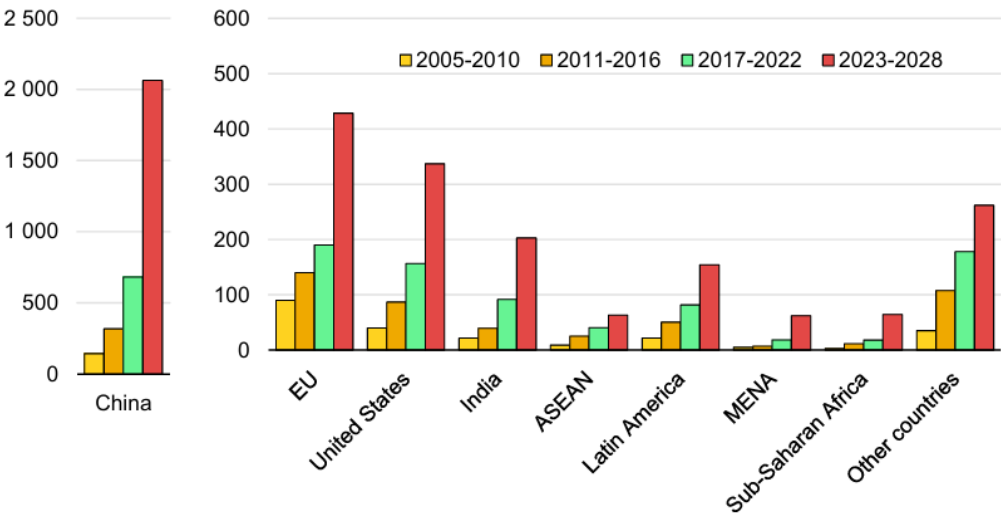
There are advantages and disadvantages to integrating renewable energy sources into the global energy grid. On one hand, the adoption of renewables significantly reduces greenhouse gas emissions and air pollutants, leading to improvements in public health and environmental quality (Kor, 2023; Maradin, 2021). On the other hand, the intermittent nature of some renewable sources, such as solar and wind, poses challenges for grid stability and energy reliability (Sayed et al., 2023; Joshi et al., 2022). This necessitates the development of advanced energy management systems, energy storage solutions, and hybrid systems that combine multiple energy sources to ensure a consistent power supply and optimize renewable energy use (Anyaeche et al., 2018; Kumar & Kumar, 2017).

Technological advancements have been pivotal in making renewable energy more accessible and less costly. The rapid decline in the cost of solar panels and wind turbines has made these technologies increasingly competitive with fossil fuels (Nkordeh, 2023; Karabegović, 2021). Additionally, innovations in energy storage, such as battery technologies, are addressing the variability of renewable energy generation (Sayed et al., 2023; Wu et al., 2022). Governments and policymakers play a critical role in supporting this transition by implementing policies, incentives, and regulations that foster the development and integration of renewable energy sources (Radu et al., 2019; Agustina, 2023).

The world's transition to renewable energy sources is not uniform across regions. While countries in Europe and North America are leading the way, many developing nations, particularly in Africa, still rely heavily on fossil fuels due to economic constraints and energy access challenges (Nkordeh, 2023; Yuaningsih et al., 2020). Tailored approaches that account for local contexts and resource availability are essential for promoting renewable energy solutions in these regions. The environmental benefits of renewable energy extend beyond emissions reductions. For instance, sustainable management of natural resources, such as forests for biomass energy and water bodies for hydropower, can enhance biodiversity and ecosystem services (Grilli et al., 2016; Humeniuk & Humeniuk, 2023).

The transition to renewable energy is also closely linked to economic development, as the renewable sector has the potential to create millions of jobs in manufacturing, installation, and maintenance (Xin et al., 2022; Agustina, 2023). This job creation is particularly vital in developing countries, where energy access and economic opportunities are critical for sustainable development (Bishoge et al., 2018). Despite these benefits, challenges such as technological limitations, high initial investment costs, and regulatory barriers continue to hinder the widespread adoption of renewable energy. Overcoming these challenges requires coordinated efforts from governments, industries, and communities to create an enabling environment for renewable energy development (Maradin, 2021).

Technological advancements have played a key role in the evolution of renewable energy systems, reducing costs and increasing efficiency. The integration of digital technologies into renewable energy systems has enhanced their viability, enabling better management and optimization of energy production and consumption (Su et al., 2022). Policy frameworks have also been instrumental in driving renewable energy adoption, with governments implementing measures such as feed-in tariffs, tax incentives, and renewable portfolio standards to stimulate investment (Kabel & Bassim, 2019). However, the effectiveness of these policies varies across regions, depending on local economic conditions, governance, and public acceptance (Pan et al., 2022).



**Figure 1.8.** Renewable electricity capacity growth by country/region, main case (IEA, 2023)

Note: ASEAN = Association of Southeast Asian Nations. MENA = Middle East and North Africa. Capacity additions refer to net additions.

As countries continue to transition towards renewable energy, the lessons learned from past experiences will be crucial in shaping future strategies for sustainable energy development (Alsagr et al., 2021). The shift towards renewables offers a pathway to achieve carbon neutrality and address climate change, but it also requires substantial infrastructure investments and policy reforms to ensure reliability and efficiency in energy systems (Lund, 2007). In conclusion, the switch to renewable energy is a difficult one, multifaceted process influenced by technological advancements, financial developments, policy frameworks, and environmental imperatives. As the renewable energy sector continues to evolve, overcoming existing challenges will be essential for achieving a sustainable and resilient global energy future.

#### **1.2.2.1.      *Solar energy***

Solar energy, a renewable energy source, produces electricity and heat using sunlight. The most common way for generating solar power is through photovoltaic (PV) cells, which convert sunlight directly into electricity using semiconductor materials, such as silicon. Another technology is called Concentrated Solar Power (CSP), which employs mirrors to concentrate sunlight and create heat that a steam turbine uses to generate energy. Solar energy is considered one of the most abundant and clean energy sources, with the potential to provide large amounts of electricity, especially in areas receiving high solar radiation, such as North Africa, the Middle East, and parts of Asia. The rapid decline in the cost of solar PV—by about 85% between 2010 and 2020—has made it increasingly competitive with fossil fuels, leading to widespread adoption (IRENA, 2020). However, challenges remain, such as the intermittency of solar power (since it only produces electricity when the sun is shining) and the large land area required for solar farms. Advances in energy storage technologies, like lithium-ion batteries, are helping to mitigate these challenges by allowing excess electricity generated during the day to be stored for use at night or during cloudy periods (IEA, 2020).

Solar energy employs thermal solar panels or photovoltaic cells to capture sunlight. One of the most plentiful renewable energy sources on the planet, it has the capacity to supply a sizable amount of the world's energy requirements. Over time, solar panel efficiency has significantly increased, making solar energy more affordable and widely available. Countries like Germany

and China have made substantial investments in solar technology, leading to significant increases in solar energy production (Nadarajah & Vakeesan, 2016; Filho et al., 2018). Solar energy systems can be deployed at various scales, from small residential installations to large utility-scale solar farms, providing flexibility in energy generation (Jarmouni et al., 2021).

#### **1.2.2.2. *Wind energy***

Wind energy is a sustainable energy source that uses wind turbines to transform the kinetic energy of the wind into electrical power. With the widespread deployment of both onshore and offshore wind farms, wind power is one of the renewable energy sources with the quickest rate of growth. Although they are more expensive to build, offshore wind farms are becoming more and more popular because of the higher wind speeds across seas, but onshore wind farms are the most prevalent and are found in regions with strong and steady winds. Wind power is a major source of electricity generation in nations like China, the US, and Germany, which lead the world in wind energy output. Wind energy is cost-competitive with fossil fuels, and technological advancements have made modern wind turbines more productive and able to produce large amounts of power. However, the intermittency of wind, meaning it only generates power when the wind is blowing, and concerns about its environmental impact, particularly on wildlife such as birds and bats, pose challenges to its expansion (IRENA, 2020). Notwithstanding these obstacles, wind energy continues to play a significant role in international initiatives to switch to greener energy sources and lower greenhouse gas emissions.

Another well-known renewable resource is wind energy, which is produced by wind turbines, which transform wind energy from kinetic energy into electrical power. Wind energy has grown rapidly globally, particularly in regions with favorable wind conditions, such as the North Sea and parts of the United States (Simionescu et al., 2020; Kor, 2023). The development of wind energy technology has produced bigger, more effective turbines that can produce electricity even at lower wind speeds. However, challenges such as visual impact, noise, and the need for suitable locations for wind farms remain significant considerations in wind energy development (Tutar & Ataş, 2022; Megawati, 2024).

#### **1.2.2.3.      *Hydropower***

A sustainable energy source called hydropower, sometimes known as hydroelectric power, produces electricity by using the force of flowing water, usually from rivers or artificial reservoirs. It contributes significantly to the production of renewable electricity worldwide, making it one of the most established and popular renewable energy sources. Large hydropower dams, like the Three Gorges Dam in China and the Itaipu Dam in South America, generate substantial amounts of electricity by releasing water through turbines. Hydropower is particularly valued for its ability to provide consistent baseload power and its capability to adjust quickly to fluctuations in electricity demand. Small-scale hydropower systems, which have a lower environmental impact, are also being increasingly deployed in remote or rural areas. Large hydropower projects, however, may have detrimental effects on the environment and society, such as the destruction of natural habitats, interruptions to fish migration, and uprooting of local populations. Additionally, the effectiveness of hydropower can be affected by climate change, as alterations in precipitation patterns and prolonged droughts can reduce water availability (IPCC, 2021). Despite these challenges, hydropower remains a reliable and essential renewable energy source in many countries.

One of the earliest and most well-known kind of renewable energy is hydropower, which is generated by the flow of water. It accounts for a substantial share of the world's electricity production, particularly in countries with abundant water resources, such as Norway and Canada (Kukharets, 2023; Pedraza, 2014). Large dams and small run-of-the-river systems are two sizes of hydropower facilities that can be used with flexibility in their installation. However, environmental concerns related to ecosystem disruption and the displacement of communities are critical issues that must be addressed in hydroelectric projects (Shcherbak et al., 2021).

#### **1.2.2.4.      *Biomass***

A renewable energy source, biomass energy comes from organic resources including waste, residues from agriculture, and wood. It can be transformed into biofuels like ethanol and biodiesel for transportation, or it can be used straight away for heating and the production of electricity. Because the carbon dioxide (CO<sub>2</sub>) absorbed by plants during their growth roughly offsets the CO<sub>2</sub> released after combustion, biomass is frequently regarded as carbon-neutral.

However, because large-scale biomass production can result in deforestation, degraded soil, and competition with food crops, the sustainability of biomass energy depends on how the feedstocks are managed. While traditional biomass, such as firewood, is still widely used in rural areas of developing countries, modern biomass technologies are gaining traction in industrialized nations. Advanced biomass systems include biogas plants and combined heat and power (CHP) facilities that efficiently convert organic waste into energy. Despite its benefits, challenges like land use, feedstock supply, and emissions from biomass combustion need to be carefully managed to ensure that biomass remains a sustainable energy option (IRENA, 2020).

Plant and animal waste are among the organic resources used to produce biomass energy. It may be transformed into a variety of energy sources, including heat, electricity, and biofuels. Biomass is particularly valuable in rural areas where agricultural residues can be utilized for energy production, thus providing local energy solutions and reducing waste (Okoye et al., 2017; Genç & Akilli, 2019). However, the sustainability of biomass energy depends on responsible sourcing and management practices to avoid deforestation and competition with food production (Okoye et al., 2017).

#### **1.2.2.5. Geothermal**

Geothermal energy is a renewable energy source that converts heat stored beneath the Earth's surface into power and direct heating. Drilling wells into geothermal reservoirs that hold hot water or steam allows one to extract this energy, which can subsequently be used to heat buildings and industrial operations or to turn turbines in geothermal power plants. Using geothermal energy for district heating systems and electricity generation has been pioneered by countries with substantial geothermal potential, including areas of the United States, Indonesia, and Iceland. Unlike solar and wind energy, which are impacted by the weather and time of day, geothermal energy is extremely dependable and capable of providing baseload electricity. Unfortunately, because it works best in areas with a lot of tectonic or volcanic activity, its application is location-specific. Moreover, there may be environmental drawbacks to geothermal development, including the possibility of induced seismicity (earthquakes) and the emission of gasses like hydrogen sulphide. Despite these challenges, geothermal remains a critical part of the renewable energy mix in areas where the resource is available (IRENA, 2020).



Geothermal energy harnesses heat from the Earth's interior, providing a reliable and consistent energy source. It is particularly effective in regions with high geothermal activity, such as Iceland and parts of the United States (Genç, 2023). Because they can produce baseload electricity, geothermal power plants are a reliable supplement to intermittent renewable energy sources like wind and solar power. However, the geographical limitations and high upfront costs of geothermal projects can pose challenges to widespread adoption (Genç, 2023).

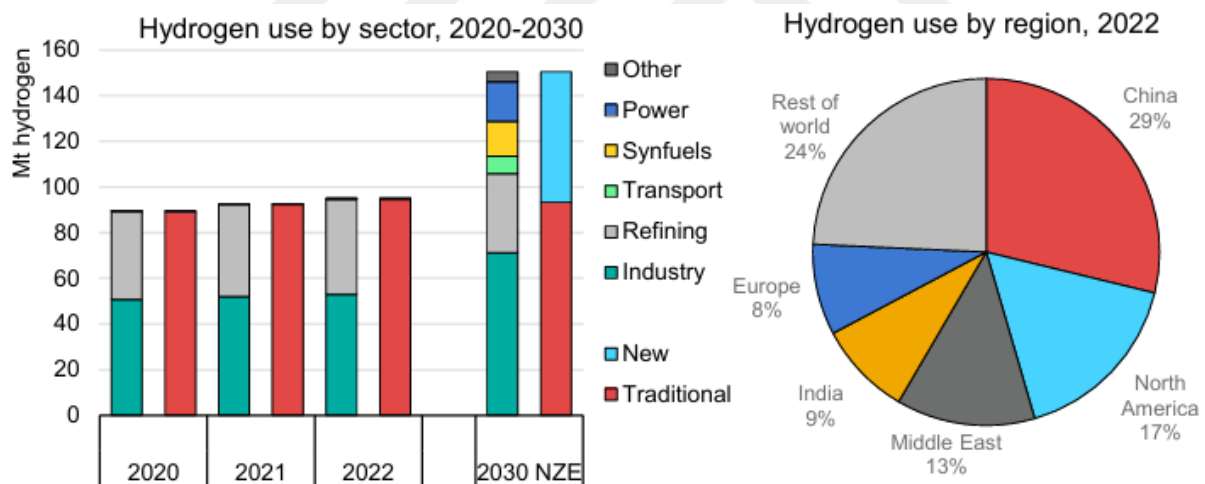
#### **1.2.2.6. *Tidal and wave***

Marine energy, which combines tidal and wave energy, uses the ocean's power to create electricity. Underwater turbines positioned in tidal streams or tidal barrages can be used to harvest tidal energy, which is produced by the moon and sun's gravitational influence on the tides' predictable movement. Surface waves provide wave energy, which is harnessed by apparatuses that transform water oscillation into electrical current. Because of the huge energy capacity of the oceans, both wave and tidal energy offer significant potential, especially in coastal regions like the United Kingdom and Canada that experience large tidal motions. Since tidal energy is more predictable than solar or wind energy, it is particularly appealing as a power source. However, the development of marine energy faces challenges, including the high costs of construction and maintenance in harsh marine environments, as well as concerns over the potential impact on marine ecosystems (IRENA, 2014). Despite these challenges, advancements in technology are making marine energy an increasingly viable option for diversifying the global renewable energy mix (Lewis, 2011).

Ocean energy encompasses various technologies that capture energy from the sea, including tidal, wave, and ocean thermal energy conversion (OTEC). These technologies are still in the developmental stages compared to other renewable sources, but they hold significant potential due to the vast energy resources available in oceans (Shields, 2014). The challenges associated with ocean energy include high costs, technological complexity, and environmental impacts on marine ecosystems (Shields, 2014).

### 1.3. Hydrogen

Hydrogen power refers to the use of hydrogen as a clean energy carrier that can be utilized for electricity generation, heating, and transportation, and plays a key role in renewable energy systems (Shinnar, 2003; Melis et al., 2001; Ball et al., 2015). While hydrogen is not a primary energy source, it holds significant potential as a flexible energy carrier, similar to electricity, that can support decarbonization efforts across multiple sectors by reducing greenhouse gas emissions and enhancing energy security (Møller et al., 2017; Hosseini et al., 2020). The primary advantage of hydrogen is that it produces zero carbon emissions at the point of use, with water being the only byproduct when used in fuel cells (Singla et al., 2021; Brouwer, 2010). Hydrogen can be produced through several methods, including water electrolysis powered by renewable sources like wind, solar, and hydroelectric power, as well as through fossil fuel reforming, though the latter results in associated carbon emissions (Brauns & Turek, 2020; Zhang et al., 2023). This versatility positions hydrogen as a crucial component part of the shift toward a future powered by renewable energy.



**Figure 1.9.** Hydrogen use by sector and by region, historical and in the Net Zero (IEA, 2023)  
Notes: NZE = Net Zero Emissions by 2050 Scenario. “Other” includes buildings and biofuels upgrading.

Transportation, energy production, and industrial operations are just a few of the uses for hydrogen. Hydrogen fuel cell vehicles (FCVs) are becoming more and more popular in the transportation industry as a greener substitute for traditional gasoline and diesel vehicles. These

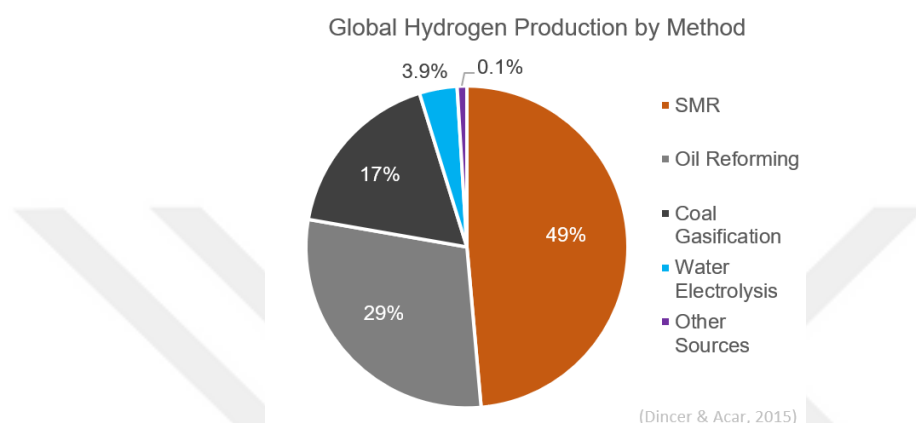
vehicles use hydrogen to generate electricity through a fuel cell, emitting only water vapor as a byproduct (Pankhedkar et al., 2023; Yuvaraj, 2023; Açıkgöz & Celik, 2012). The development of hydrogen refueling infrastructure is crucial for the widespread adoption of FCVs, as it enables drivers to refuel their vehicles similarly to traditional gasoline stations (Oueslati, 2023; Yuvaraj, 2023; Eberle et al., 2012). Furthermore, hydrogen can also be blended with natural gas for use in existing gas infrastructure, providing a transitional pathway towards a hydrogen economy (Pelaz et al., 2023; Guerra et al., 2019).

Hydrogen power offers enormous possibilities not only for transportation but also for the energy industry. In renewable energy systems, hydrogen can be utilized for energy storage, which helps to balance supply and demand. By converting excess electricity into hydrogen, it can be stored and later converted back into electricity when needed, thus enhancing grid stability and reliability (Song et al., 2022; Wang et al., 2022). This capability is particularly valuable in regions with high penetration of variable renewable energy sources, such as wind and solar, where energy production does not always align with consumption patterns (Wu et al., 2018; Pelaz et al., 2023). Moreover, hydrogen can be used in combined heat and power (CHP) systems, where it can contribute to both electricity generation and heating, further improving energy efficiency (Maniyali et al., 2013; Pelaz et al., 2023; Zhong et al., 2022).

The economic viability of hydrogen power is a critical consideration as well. While the production costs of hydrogen have historically been high, advancements in technology and economies of scale are expected to reduce these costs significantly in the coming years (Iqbal, 2023; Guerra et al., 2019; Zhang et al., 2020). The integration of hydrogen into existing energy systems can also lead to cost savings by optimizing the use of renewable resources and reducing reliance on fossil fuels (Iqbal, 2023; Guerra et al., 2019; Zhang et al., 2020). Additionally, hydrogen's versatility as an energy carrier allows it to serve multiple sectors, including transportation, heating, and industrial processes, thereby enhancing its overall economic potential (Pelaz et al., 2023; Guerra et al., 2019; Brown et al., 2018).

The hydrogen economy has many benefits, but in order to reach its full potential, a number of issues need to be resolved. Among these is the requirement for large-scale infrastructure investments, such as hydrogen production facilities, storage systems, and distribution networks (Pelaz et al., 2023; Guerra et al., 2019; Zhang et al., 2020). Furthermore, the environmental

impact of hydrogen production methods, particularly those that rely on fossil fuels, must be carefully managed to ensure that hydrogen truly serves as a clean energy solution (Iqbal, 2023; Pelaz et al., 2023; Guerra et al., 2019). Research and development efforts are ongoing to improve hydrogen production methods, enhance storage technologies, and develop efficient fuel cells, all of which are essential for the successful implementation of hydrogen power (Song et al., 2022; Brauns & Turek, 2020; Iqbal, 2023; Pelaz et al., 2023; Guerra et al., 2019).

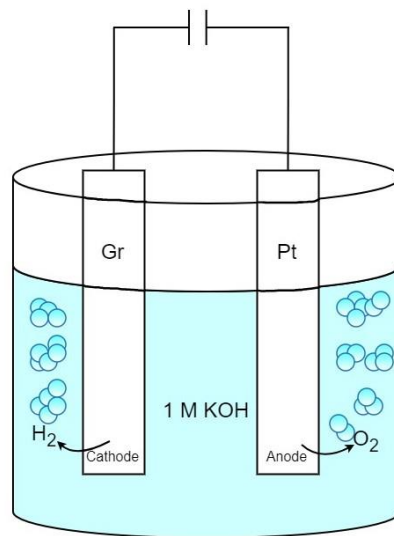


**Figure 1.10.** Global hydrogen production by method (Dincer & Acar, 2015)

The various processes that can be used to manufacture hydrogen are classified according to their carbon intensity and each has a distinct effect on the environment. Currently, the most popular approach is steam methane reformation (SMR), which produces gray hydrogen from natural gas, though it results in significant CO<sub>2</sub> emissions since methane is a fossil fuel. While gray hydrogen is currently the least expensive method, its high carbon footprint limits its environmental benefits (IEA, 2019). Blue hydrogen is also produced through SMR but with carbon capture and storage (CCS) technologies that reduce the associated CO<sub>2</sub> emissions, making it a lower-carbon alternative (IEA, 2021). Green hydrogen, the cleanest and most sustainable option, is produced by using electrolysis using clean energy from solar or wind power to split water into hydrogen and oxygen. It emits no greenhouse gases during production or use but is currently the most expensive method due to the high cost of electrolyzers and the need for large-scale renewable energy capacity (IRENA, 2020). There are other, less popular ways to manufacture hydrogen, like using biomass or thermochemical and biological processes. Research is still being done to find more economical and environmentally friendly ways to produce hydrogen.

### 1.3.1. Hydrogen production by electrolysis

One of the most promising methods for hydrogen production is water electrolysis, which involves splitting water into hydrogen and oxygen using electricity (Chi, 2018). When powered by renewable energy sources like wind or solar, this process generates "green hydrogen," the cleanest and most sustainable option as it emits no greenhouse gases during production or use (Brauns & Turek, 2020; Hong, 2024). Although green hydrogen is currently the most expensive method due to the high cost of electrolyzers and the need for large-scale renewable energy capacity, recent advancements in electrolysis technology have improved efficiency, making it a viable option for large-scale hydrogen production (Zhang et al., 2023; Hong, 2024; Ursúa et al., 2012). The integration of hydrogen production with renewable energy sources also addresses the intermittency issues associated with these systems, allowing excess energy generated during peak times to be stored as hydrogen for later use (Song et al., 2022; Wu et al., 2018; Pelaz et al., 2023).



**Figure 1.11.** Schematic of the experimental setup

## 1.4. Response Surface Method

A comprehensive set of statistical and mathematical methods known as Response Surface Methodology (RSM) are used to describe and optimize intricate processes in which several

variables affect an outcome of interest. It is particularly valuable for improving product quality, efficiency, and process performance in fields like pharmaceuticals, chemical engineering, agriculture, and food science. By designing and conducting well-planned experiments, RSM helps to identify optimal operating conditions that either maximize or minimize responses such as yield, quality, or cost (Box & Draper, 1987; Myers, Montgomery, & Anderson-Cook, 2016).

At the heart of RSM is the concept of the response surface, a graphical representation showing the relationship between input factors (independent variables) and the outcome or response (dependent variable). These factors are controlled during experimentation, while the response is the measured outcome that RSM seeks to optimize. The response surface allows researchers to visually explore how different levels of input variables affect the response, often through tools like contour plots and 3D surface graphs (Montgomery, 2017). Central Composite Design (CCD) and Box-Behnken Design (BBD) are two of the most commonly used experimental designs in RSM. These designs help collect data efficiently, enabling researchers to fit second-order polynomial models (quadratic models) to describe the nonlinear interactions between variables (Bezerra et al., 2008).

The primary goal of RSM is to identify the optimal factor settings to achieve the desired outcome. Techniques like gradient ascent (for maximization) or gradient descent (for minimization) are employed to explore the fitted response surface and determine optimal conditions (Myers et al., 2016). Applications of RSM are broad, including the optimization of pharmaceutical formulations, improving agricultural yields, and enhancing food processing techniques. For example, in a chemical reaction, RSM can be used to find the ideal combination of temperature and pressure to maximize product yield (Bas & Boyaci, 2007; Montgomery, 2017).

Despite its many advantages, RSM does have limitations. Its effectiveness relies heavily on the quality of experimental design and the accuracy of the fitted model. Poor experimental design or inadequate models can lead to erroneous conclusions. Furthermore, RSM is best suited for continuous variables, making it less effective for categorical variables (Giunta et al., 2003). Nonetheless, its ability to model complex, nonlinear relationships and provide clear visualizations makes RSM an essential tool in process optimization across numerous industries (Bezerra et al., 2008).

## 2. LITERATURE REVIEW

Doner et. al. (2012) applied nickel and cobalt to the carbon felt surface to produce hydrogen electrocatalytically. Because of its porous structure, carbon has a high specific surface area. Additionally, when nickel and cobalt atoms are deposited, the surface of the carbon is exposed to numerous active sites.

Fang et al. (2014) improved the electrocatalytic performance of glassy carbon electrodes by depositing Ni and S atoms on their surface using the electrodeposition method. Glassy carbon exhibited good performance of electrocatalytic hydrogen production in acidic solutions because of the formation of a thin layer of S and Ni atoms on its surface.

Fan et al. (2016) carbonized MOF structural materials to produce graphite carbon modified with Ni atoms. Due to the Ni atom change, this electrocatalyst produced hydrogen with exceptional efficiency by using the improved electron transportation abilities on the graphite carbon surface.

Sivasakthi et al. (2017) employed a pulse electrodeposition approach to deposit a nanostructured NiCeO<sub>2</sub>Gd doped nanocomposite on copper foam, that they were then employed in the hydrogen evolution process (HER) as an electrocatalyst. Using SEM, TEM, EDAX, XRD, and XPS, the surface form, preferred orientation, chemical composition, and oxidation states of the as-prepared Ni and Ni-CeO<sub>2</sub>Gd doped nanocomposite were investigated." The Ni and Ni-CeO<sub>2</sub>Gd doped nanocomposite for HER was investigated for stability and electrocatalytic activity using Linear Sweep Voltammetry (LSV). The results show that the Ni-CeO<sub>2</sub>Gd doped nanocomposite exhibits higher catalytic activity compared to nickel, with a larger exchange current density (0.1231 mA/cm<sup>2</sup>), a lower onset overpotential (160 mV), and a lower Tafel slope (102 mV/dec).

Allam et al. (2018) examined the electrocatalytic characteristics and corrosion resistance of NiWMo thin films electrodeposited on copper sheet for HER. The three-element composite's synergistic influence on hydrogen production has led to positive results.

Mert and Dođru Mert (2018) developed a triple coating of nickel-cobalt-bismuth (NiCoBi) on a copper (Cu) surface using electrochemical methods (Cu/NiCoBi) and studied its performance

in hydrogen gas production through the electrolysis of alkaline water. According to their results, the hydrogen overpotential of the Cu/NiCoBi electrode is significantly lower compared to Cu and other binary coatings (Cu/NiCo, Cu/NiBi). The Cu/NiCoBi electrode maintained its effectiveness throughout a 168-hour electrolysis period.

Kim et al. (2018) demonstrated that hydrogen could be produced electrocatalytically by applying a single atom of the precious metal Pt to the surface of other carrier materials. It was noted that doped carbon and faulty graphene materials might act as carriers for single atoms of valuable metals.

Koca et al. (2019) examined the effects of nickel (Ni) and a small quantity of gallium (Ga) placed on carbon felt electrodes (CF/Ni and CF/NiGa). They also assessed the hydrogen evolution performance of CF/Ni and CF/NiGa in 1 M KOH, finding that Ni modification lowered over the potential value of the CF cathode and that Ga improved the performance of hydrogen production. After 30 minutes in 1 M KOH, the CF/NiGa electrode generated 28.9 mL H<sub>2</sub>(g).

Jin et al. (2021) have also synthesized Ni-Mo-O/Ni<sub>4</sub>Mo nanointerface electrocatalyst (Ni-MoO/Ni<sub>4</sub>Mo@NC) supported by N-doped carbon using electrodeposition–calcination–electrodeposition techniques, and have examined their electrocatalytic activity toward the hydrogen evolution reaction in an alkaline solution (1M KOH). The N-doped carbon layers in the produced Ni-Mo-O/Ni<sub>4</sub>Mo@NC electrocatalyst have resulted in a 50% reduction in overpotential, measuring 61 mV at a geometric current density of 10 mA.cm<sup>-2</sup>, compared to Ni-Mo-O's 120 mV.

Yang et al. (2021) developed a morphology-controlled nanocrystal  $\beta$ -Ni(OH)<sub>2</sub>/NF (nickel hydroxide/nickel foam) electrocatalyst for the electrolysis of water using a hydrothermal method. The study aimed to investigate the morphology-dependent electrocatalytic characteristics of the  $\beta$ -Ni(OH)<sub>2</sub>/NF catalyst in an alkaline environment for the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Additionally, it was associated with the mechanism of the morphology-dependent electrocatalytic properties of the  $\beta$ -Ni(OH)<sub>2</sub>/NF catalyst.



Mert (2022) examined the electrodeposition of Ni and Co particles on the Ni foam (NF) electrode, which was accomplished using a suitable catalyst placed with Ag nanoparticle size after the galvanostatic method with different chemical compositions. The characterisation was accomplished by means of energy-dispersive X-ray (EDX), field emission scanning electron microscopy (FESEM), and X-ray diffraction (XRD) techniques. The hydrogen generation performance of electrodes in the alkaline medium was investigated using Tafel polarization measurements, electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), linear sweep voltammetry (LSV), and Tafel polarization measurements. Chronoamperometry (CA) was used to investigate long-term stability after the convenient electrode was identified.

Xu et. al. (2022) created a highly effective bifunctional electrocatalyst using NiCoP nanoflakes (NiCo(nf)-P) generated from the 2D metal-organic framework (MOF) for general water splitting in alkaline environments. Utilizing a three-electrode cell assembly and the linear sweep voltammetry (LSV) technique, the produced electrocatalyst's performance was assessed at a potential cycling rate of  $2 \text{ mV.s}^{-1}$  in an electrolyte solution of 1M KOH. The generated NiCo(nf)-P was shown to have improved electrocatalytic activity with a lower onset potential of 37 mV and 1.435 V compared to commercial 5% Pt/C (43 mV) and IrO<sub>2</sub> (1.504 V) at the current density of  $100 \text{ mV.cm}^{-2}$  towards the HER and OER.

Alotaibi et al. (2024) doped graphene oxide with molybdenum oxide (MoO<sub>3</sub>) at concentrations ranging from 1 mM to 5 mM to create an extraordinary performance, self-supporting electrode meant for OER and HER. The catalytic efficacy of MoO<sub>3</sub>/rGO-1000 was tested at annealing temperatures ranging from 600 to 1000 °C. Notably, the maximum HER and OER activity was shown by MoO<sub>3</sub>/rGO-1000. These materials demonstrated remarkable stability; current densities of  $10 \text{ mA.cm}^{-2}$  were achieved at low over potentials of 13 mV and 250 mV, respectively.

Mu et. al. (2009) used Response Surface Methodology (RSM) with central composite design to optimize hydrogen production from glucose. They assessed how pH, temperature, and glucose concentration affected hydrogen generation alone and in combination. It was discovered that 38.8 °C, pH 5.7, and 9.7 g/L of glucose were the optimum values for maximum H<sub>2</sub> yield.

Lotfi et. al. (2019) investigated the electrocatalytic activity of a Ni foam electrode modified with dendritic Ni-Cu for "HER" in an alkaline solution. To improve the stability and electrocatalytic activity of the Ni foam substrate, the study attempts to optimize the synthesis and modification process of the dendritic Ni-Cu electrode. The mechanism underlying the dendritic Ni-Cu modified Ni foam substrate for HER's enhanced electrocatalytic activity was also investigated. They also used RSM analysis to investigate and reveal the experimental HER efficiency.

Ayodele et al. (2019) investigated the optimization of hydrogen production through photocatalytic steam methane reforming on lanthanum-modified TiO<sub>2</sub> using response surface methodology. Through the use of photocatalytic steam methane reforming, hydrogen production was optimized. The rate at which hydrogen is produced was investigated in relation to the following factors: irradiation period (10-150 minutes), metal loading (1-3%), methane content (10-50%), and steam concentration (0.5-1.5%). By applying the response surface methodology, four model formulations were developed, of which it was discovered that the quadratic model best fit the experimental data.

Umer et. al. (2019) employed response surface methodology (RSM) to maximize the impact of titanium dioxide (TiO<sub>2</sub>) nanoparticles modified by carbon nanotubes on photocatalytic hydrogen generation activity. Methods including Raman spectroscopy, transmission electron microscopy (TEM), and X-ray diffraction (XRD) were used to clarify the composition, shape, and crystal structure of the carbon nanotube/TiO<sub>2</sub> composites. Furthermore, RSM was used to optimize process variables such reaction time, carbon nanotube modification, and calcination temperature in order to determine the the maximum photocatalytic hydrogen production activity.

Kowthaman et al. (2021) used a designed micro-patterned graphite electrode to study ways to increase the hydrogen production rate during the water electrolysis process. They used alkaline sodium hydroxide as an electrolyte to enhance the amount of hydrogen produced during continuous electrolysis. The regression model for the hydrogen manufacturing process was developed using a four-factor, three-level full factorial design, which was based on the Box-Behnken design. The combined effects of electrode distance, electrolyte molarity, and voltage supplied on the rate of hydrogen production were examined using the RSM.

Rothan et. al. (2021) performed optimization for hydrogen yield from ammonia using the response surface methodology. Response surface methodology was used in studies to examine the variables taken into consideration in hydrogen yields, including electric voltage (90-110 V), ammonia flow rate (30-150 L/hour), and upstream pressure (0-100 kPa).

Esmailzadeh et. al (2021) studied the RSM determined by CCD was used to pulse plate NiSe on nickel foam at the ideal pH and Se/Ni ratio. Additionally, after determining the ideal values for Se/Ni and pH using RSM analysis, the influence of temperature was investigated in order to create an electrocatalyst with high electrocatalytic properties. Lastly, LSV, CV, EIS, SEM, EDX, and XRD characterisation were used to conduct the electrochemical studies of the ideal electrode. The results show that the electrode electrodeposited at room temperature has the best electrocatalytic performance for OER and HER. It has a large electrochemically active surface area of 8960 and low Tafel slopes of 35 mV/dec and 86 mV/dec, respectively.

Özgür and Mert (2022) looked into the hydrogen production efficiency of Ni and Cu-deposited Ni foam electrodes in different composition proportions. They were selected three factors as independent variables: catalyst amount, electric voltage, and electrolysis time. They used the Design-Expert program to statistically analyze the effects of these independent variables on hydrogen generation. The results showed that the developed model determined that 3 V of electricity, 11.35  $\mu\text{g}$  of catalyst, and 30 minutes of electrolysis time would yield the best hydrogen generation.

Özgür and Mert (2023) developed a two-electrode method on a laboratory scale using an alkaline electrolysis cell to produce hydrogen gas. The anode was the noble metal platinum, and the cathode was nickel foam electrodes modified with nickel-copper-molybdenum (NiCuMo) via a galvanostatic process. They tested the system at three different operation potentials (2.4, 2.7, and 3 V). Based on the increasing application potentials, the study's findings show that after 30 minutes of electrolysis, 77.30, 90.67, and 105.08 mL of hydrogen gas were produced, respectively. The system was optimized using response surface methodology (RSM). The electrolysis potential and time were selected as parameters for analyzing system efficiency, and their impacts on the volume of hydrogen gas produced and the amount of charge were examined. The hydrogen volume and charge quantity obtained respective  $R^2$  values of 0.9956 and 0.9955.

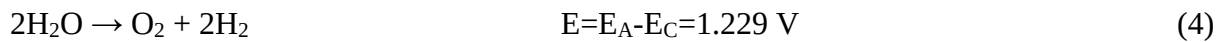
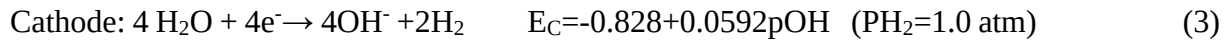
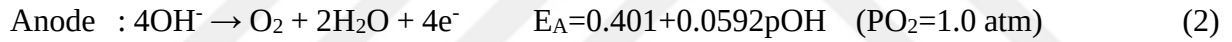
### 3. MATERIAL AND METHOD

#### 3.1. Electrochemical Part

The Nernst equation can be used to calculate the electrode potentials corresponding to the equilibrium states of the cathode and anode in order to calculate the theoretical separation stress.

$$E_d = E_{\text{anode}} - E_{\text{cathode}} \quad (1)$$

The  $E_d$  value obtained here is the theoretical separation voltage calculated under the assumption that there is no overvoltage at the anode and cathode. Theoretically, 1.23 V is needed for the breakdown of water at 25 °C. However, in order to start the evolution of hydrogen gas in the system, a minimum potential of 1.70 V needs to be applied, taking into account the overvoltage of  $O_2$  on Pt (0.47 V) as an anode addition. Higher potential is needed during the experiment because of overvoltages from the solution and electrode metal, as well as the internal resistance of the voltage source and the experiment's connections. Alkaline water is electrolyzed to produce hydrogen gas at the cathode and oxygen gas at the anode.



Electrolysis was performed using the two-electrode technique with a precise current source to obtain current-voltage curves and determine hydrogen volumes at a specific constant potential. The current meter section of the multimeter was used to measure the current flowing through the system. In the system where platinum was selected as the anode and graphite as the cathode, decomposition voltages in a 1 M KOH solution were determined using the two-electrode technique. For this purpose, potentials ranging from 1.3 V to 3 V in 0.1 V increments were applied from a direct current source, and current values through the system were recorded to generate current-potential curves. The reason for not starting from 0 V is that the theoretical decomposition voltage is approximately 1.3 V. The decomposition voltages of the electrolysis

system were determined from intersection points by extrapolating the linear portions of the curves. This experiment was repeated 5 times, yielding consistent results.



**Figure 3.1.** Experimental Setup

After determining the decomposition voltage, the second part of our experiment, which involved determining hydrogen volumes, began. In this section, volumes of hydrogen generation were noted at various voltage levels. The graphite electrode and platinum electrode were immersed in a 1 M KOH solution at a fixed distance of 2 cm. The cathode, from which hydrogen gas was evolved, was placed in the cylinder by inverting it. This setup allowed the measurement of the produced hydrogen amount. Starting from a decomposition voltage of 2.3 V and increasing in 0.1 V increments up to 3 V, the volume of hydrogen produced and the current values through the system were measured for all voltage values. The system was operated for 30 minutes at each voltage value. The volume amounts depicted in the cylinder were recorded at 5-minute intervals for each voltage since it was anticipated that water vapor would also accumulate in the cylinder under the same circumstances. Volume corrections were made as detailed below.

$$P_{H_2} = P_T - P_{H_2O} \quad (5)$$

Under these conditions, with the vapor pressure of water ( $P_{(H_2O)}$ ) at 23.756 mmHg and the total atmospheric pressure ( $P_T$ ) at 756 mmHg, the pressure of hydrogen gas ( $P_{(H_2)}$ ) is determined to be 732.244 mmHg. The volume of hydrogen is as follows ( $V_{(H_2)}$ ):

$$V_{H_2} = \frac{732.244}{756} \times V_{measured} \quad (6)$$

The value determined here is calculated using the relationship. Here, the volume refers to the entire amount of gas that was experimentally gathered in the cylinder. Table 3.1. shows Experimental hydrogen volumes after 30 minutes of electrolysis at various voltages (E) for the G electrode.

**Table 3.1.** Experimental Hydrogen Volumes

| E (V) | V <sub>e</sub> (mL) |
|-------|---------------------|
| 2.3   | 7                   |
| 2.4   | 9.5                 |
| 2.5   | 12.5                |
| 2.6   | 15                  |
| 2.7   | 18                  |
| 2.8   | 21                  |
| 2.9   | 28                  |
| 3.0   | 30                  |

### 3.1.1. Setting up electrolysis system

#### 3.1.1.1. Mild steel electrode

The working electrode was a sample of mild steel. A thin disk of mild steel with a surface area of 0.5 cm<sup>2</sup> was cut from a mild steel rod (weight percentages): 0.17% C, 0.59% Si, 1.60% Mn, 0.04% P, and the remainder Fe. After being connected via a copper wire for electrical conductivity, the samples were mounted on a polyester block. Prior to the experiments, the exposed surface of the working electrode was polished with 1200-grit sandpaper, then cleaned with distilled water and dried with filter paper.



**Figure 3.2.** Mild steel electrode

### **3.1.1.2. Modified graphite electrode**

The coatings were performed using analytical grade chemicals (Sigma, Adana) with the Ivium stat electrochemical workstation via the galvanostatic method at 5 mA/cm<sup>2</sup> current density, using a 5 µg/cm<sup>2</sup> catalyst amount and a Ni:Co:Mo molar ratio of 0.9:0.5:0.5. The chemical compositions of the plating baths are nickel plating bath: %30 NiSO<sub>4</sub>·7H<sub>2</sub>O, %1 NiCl<sub>2</sub>·6H<sub>2</sub>O, % 1,25 H<sub>3</sub>BO<sub>3</sub>, cobalt plating bath: % 30 CoSO<sub>4</sub>·7H<sub>2</sub>O, % 1 CoCl<sub>2</sub>·6H<sub>2</sub>O, % 1,25 H<sub>3</sub>BO<sub>3</sub>, molybdenum plating baths: 30 % MoNa<sub>2</sub>O<sub>4</sub>, 2,0 % NaCl, 1,25 % H<sub>3</sub>BO<sub>3</sub>. The substrate used was a graphite rod, with a counter electrode of Pt plate (1\*1\*0.1 cm), and an Ag/AgCl (3 M KCl) electrode was used as the reference electrode.



**Figure 3.3.** Modified Graphite Electrode

Energy-dispersive X-ray (EDX) methods and field emission scanning electron microscopy (FE-SEM) were used to characterize the samples.

The amount of hydrogen gas generated measured using a two-electrode electrolysis cell, where the working electrode was used as the cathode and Pt as the anode. Over the cathode was an inverted graduated cylinder that was filled with 1 M KOH. When the system was being set up, different constant potential values (2.5, 2.75, and 3.0 V) were applied. The amount of hydrogen gas gathered in the graduated cylinder over a 15-minute period was recorded for each potential.

### 3.2. RSM Technology

In response surface methodology, designing experiments is a crucial step. This process involves selecting variables, determining experimental strategies, performing mathematical and statistical analyses on collected experimental data, evaluating model fit, and obtaining optimized values for each variable (Bashir et al., 2015). The trial version of Design Expert software was utilized for data analysis in this study, and central composite design (CCD) was selected as the method for second-order model creation. Electrode voltage and molarity were selected as independent parameters, while hydrogen volume was considered as the dependent variable. Table 3.2 shows the independent parameters and their respective levels. To determine the effects and interactions of these factors, a central composite experimental design with 13 combinations was created, as presented in Table 3.3. and Table 3.4. for mild steel electrode and modified graphite electrode, respectively. The independent variables chosen were voltage (coded as A: 2.5, 2.75, 3.0 V) and molarity (coded as B: 0.5, 1.0, 1.5 M). The response function measured was hydrogen volume (mL). The first stage of response surface methodology (RSM) involves establishing an appropriate relationship between input and output parameters. To achieve this relationship, a second-order equation model, as shown in Equation 7, was applied (Kahveci and Taymaz, 2022).

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{i < j}^k \beta_{ij} X_i X_j + \epsilon \quad (7)$$

The second-degree polynomial model's regression coefficients are as follows:  $\beta_0$  represents the constant term,  $\beta_i$  denotes a linear effect,  $\beta_{ii}$  denotes a quadratic impact, and  $\beta_{ij}$  denotes an interaction effect.

The significance of the linear, interaction, and quadratic terms as well as the applicability of the fitting models for the experiments were assessed in the last stage using the analysis of variance



(ANOVA) for the response surface quadratic model. Second-order polynomial regression was developed using the sequential F-test, lack-of-fit test, and extra CCD adequacy measurements.

Statistical software was used to calculate the coefficient values. Multiple regressions were used to assess the experimental data from CCD. A minimum coefficient of determination ( $R^2$ ) of 0.8 is necessary for a model fit to be considered acceptable. This made the quality assessment of the fitted models possible. Using Fisher's F-test, the statistical significance of the regression coefficients was assessed. Overall, a high correlation coefficient (correlation coefficient) and a substantial F-value suggested that the model was useful and effective. Following that, response surface diagrams and statistical computations were conducted using the regression results.

The model terms were assessed using the 95% confidence level and P-value (probability of error, P-value 0.05). Significant model terms are indicated by P-values ( $\text{Prob} > F$ )  $< 0.050$ . Conditions for the highest hydrogen volume were predicted by using the Design-Expert program's quadratic equation/numerical optimization function. The voltage and molarity were changed to create three-dimensional graphs and the accompanying contour plots. The experimental design matrix was created using the Design-Expert software program (version trial Stat-Ease, Inc. Minneapolis, USA), analyze statistical data, and optimize or respond to surface analysis. Figure 1 shows a flowchart representing the steps in the response surface methodology.

**Table 3.2.** Independent parameters and their corresponding levels

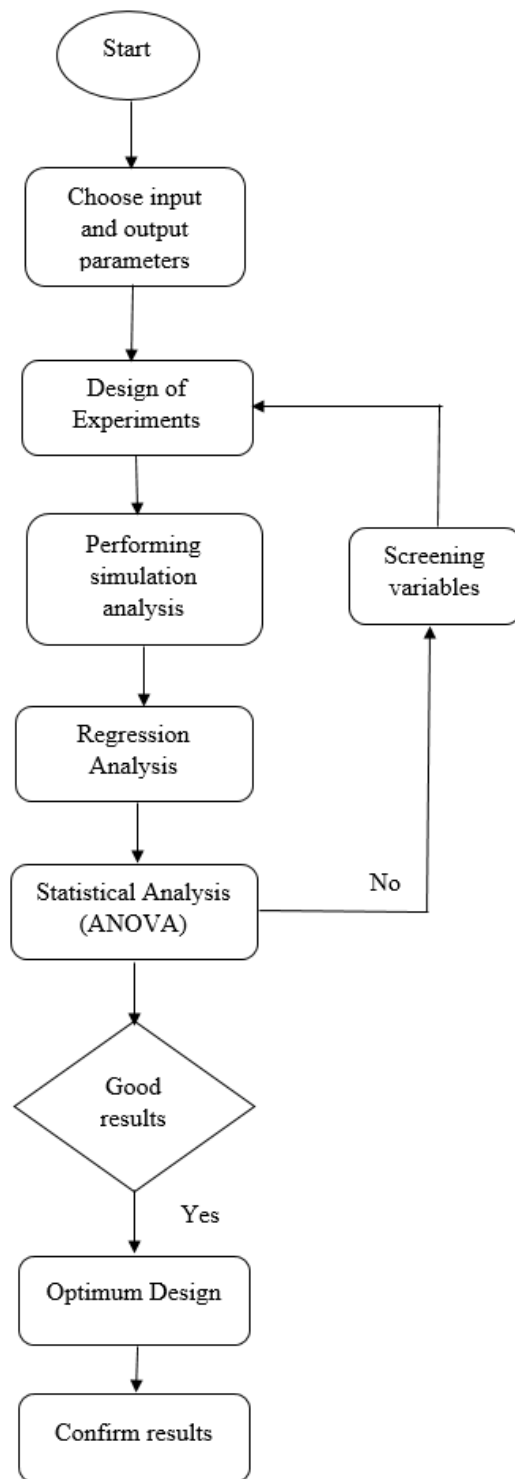
| Symbol | Parameter         | Unit | Level |      |     |
|--------|-------------------|------|-------|------|-----|
|        |                   |      | -1    | 0    | +1  |
| $X_1$  | Electrode voltage | V    | 2.5   | 2.75 | 3.0 |
| $X_2$  | Molarity          | M    | 0.5   | 1.0  | 1.5 |

**Table 3.3.** Comparison of experimental and predicted values for hydrogen production for mild steel electrode

| No | X <sub>1</sub> :Electrode<br>voltage (V) | X <sub>2</sub> :Molarity<br>(M) | Experimental<br>Hydrogen<br>Volume (mL) | RSM<br>Hydrogen<br>Volume (ml) |
|----|------------------------------------------|---------------------------------|-----------------------------------------|--------------------------------|
| 1  | 2.75                                     | 1.0                             | 4.55                                    | 4.54                           |
| 2  | 3.0                                      | 1.5                             | 8.23                                    | 8.25                           |
| 3  | 2.75                                     | 0.5                             | 2.91                                    | 2.91                           |
| 4  | 2.5                                      | 0.5                             | 2.13                                    | 2.08                           |
| 5  | 3.0                                      | 1.0                             | 6.3                                     | 6.23                           |
| 6  | 2.5                                      | 1.5                             | 3.78                                    | 3.70                           |
| 7  | 2.75                                     | 1.5                             | 5.81                                    | 5.88                           |
| 8  | 3.0                                      | 0.5                             | 3.87                                    | 3.92                           |
| 9  | 2.5                                      | 1.0                             | 2.91                                    | 3.04                           |
| 10 | 2.75                                     | 1.0                             | 4.55                                    | 4.54                           |
| 11 | 2.75                                     | 1.0                             | 4.55                                    | 4.54                           |
| 12 | 2.75                                     | 1.0                             | 4.55                                    | 4.54                           |
| 13 | 2.75                                     | 1.0                             | 4.55                                    | 4.54                           |

**Table 3.4.** Comparison of experimental and predicted values for hydrogen production for modified graphite electrode

| No | X <sub>1</sub> :Electrode<br>voltage (V) | X <sub>2</sub> :Molarity<br>(M) | Experimental<br>Hydrogen<br>Volume (mL) | RSM<br>Hydrogen<br>Volume (mL) |
|----|------------------------------------------|---------------------------------|-----------------------------------------|--------------------------------|
| 1  | 2.5                                      | 1.0                             | 5.81                                    | 6.08                           |
| 2  | 3.0                                      | 0.5                             | 6.97                                    | 6.89                           |
| 3  | 2.5                                      | 1.5                             | 6.97                                    | 6.79                           |
| 4  | 2.75                                     | 0.5                             | 5.62                                    | 5.79                           |
| 5  | 2.75                                     | 1.0                             | 8.23                                    | 8.12                           |
| 6  | 3.0                                      | 1.0                             | 9.2                                     | 9.47                           |
| 7  | 2.5                                      | 0.5                             | 4.07                                    | 3.99                           |
| 8  | 2.75                                     | 1.5                             | 8.72                                    | 9.08                           |
| 9  | 2.75                                     | 1.0                             | 8.23                                    | 8.12                           |
| 10 | 2.75                                     | 1.0                             | 8.23                                    | 8.12                           |
| 11 | 3.0                                      | 1.5                             | 10.85                                   | 10.67                          |
| 12 | 2.75                                     | 1.0                             | 8.23                                    | 8.12                           |
| 13 | 2.75                                     | 1.0                             | 8.23                                    | 8.12                           |



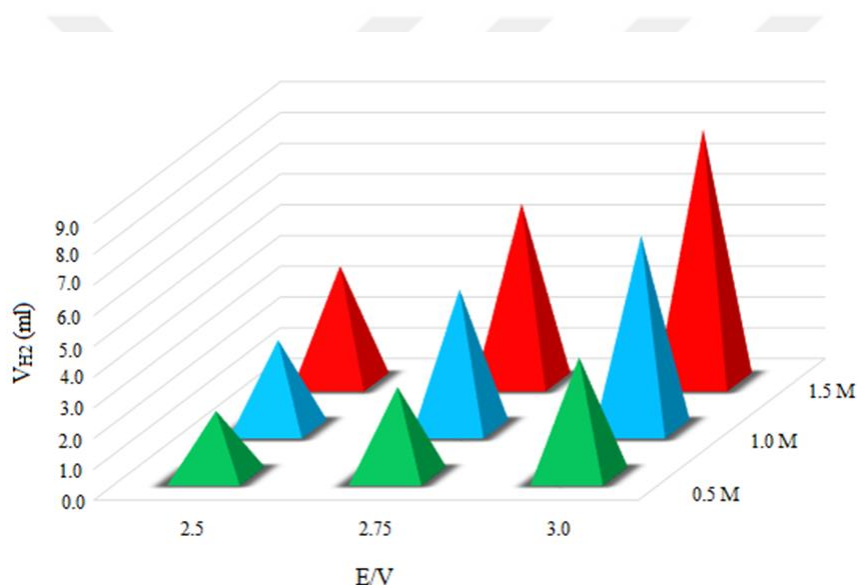
**Figure 3.4.** The stages of the response surface methodology

## 4. RESULTS AND DISCUSSION

### 4.1. Electrochemical Measurements

#### 4.1.1. Mild steel electrode

Figure 4.1. shows the volumes of hydrogen produced by the mild steel electrode. Hydrogen gas production ranged from 2.5 to 3.0 V as the potential increased. Given that the vapor pressure of water was 23.756 mmHg and the total atmospheric pressure was 756 mmHg, the pressure of hydrogen gas under laboratory conditions was determined to be 732.244 mmHg. The hydrogen volume ( $V_{H_2}$ ) was determined using the Equation 6.[37-38]

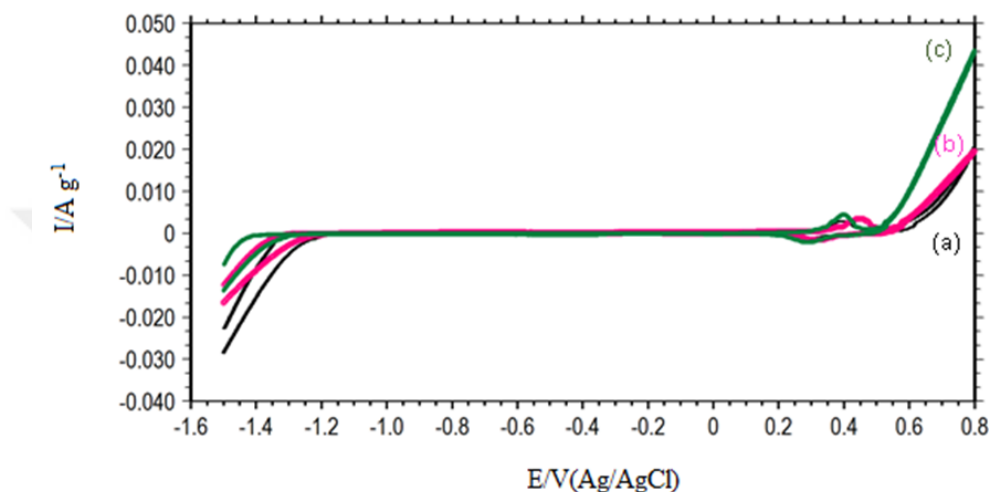


**Figure 4.1.** Hydrogen volumes for mild steel electrode over 15 minutes at various voltages

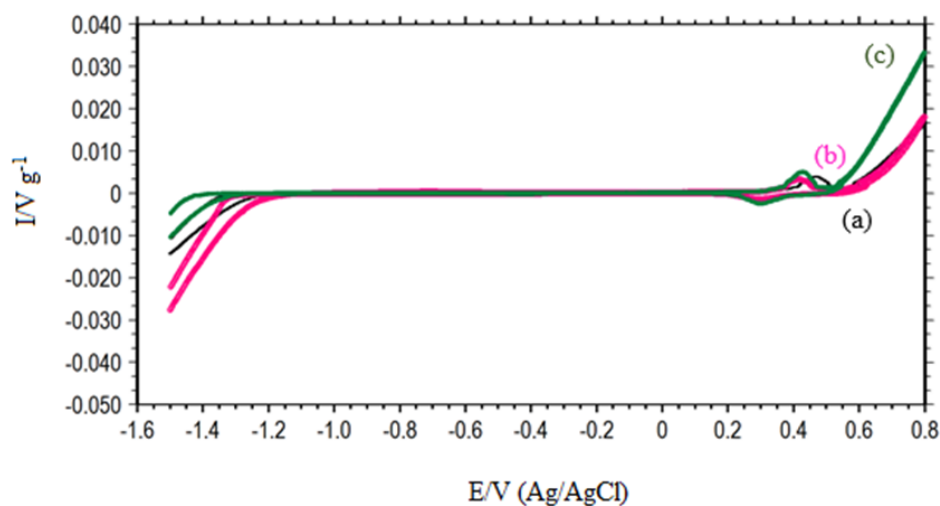
#### 4.1.2. Modified graphite electrode

Cyclic voltammograms were used as LSV measurements in a 1 M KOH solution. The chronoamperometric approach was used to test the ideal electrode's long-term performance. By using the following symbols, the electrodes were coded based on different quantities of catalyst: G/NiCoMo- I 5 microgram/ $\text{cm}^2$ , G/NiCoMo- II 7,5 microgram/ $\text{cm}^2$ , G/NiCoMo- III 10 microgram/ $\text{cm}^2$ . Figure 4.2. illustrates the increase in current values with rising basic conditions in the anodic regions. At 0.6 V, the current values were 3.6 mA, 3.9 mA, and 8.2 mA for the

G/NiCoMo-I electrode in (a) 0.5 M, (b) 1 M, and (c) 1.5 M KOH solutions, respectively, as shown in Figure 4.1. For the same solution conditions, the current values in the cathodic region at -1.4 V were -16 mA, -9.4 mA, and -5.1 mA. Figure 4.3. shows the current values for the G/NiCoMo-II electrode at 0.6 V in (a) 0.5 M, (b) 1 M, and (c) 1.5 M KOH solutions. The values were 3.7 mA, 4.1 mA, and 6.6 mA, respectively. The values obtained for the same solutions were -8.1 mA, -16.7 mA, and -3.8 mA at -1.4 V.

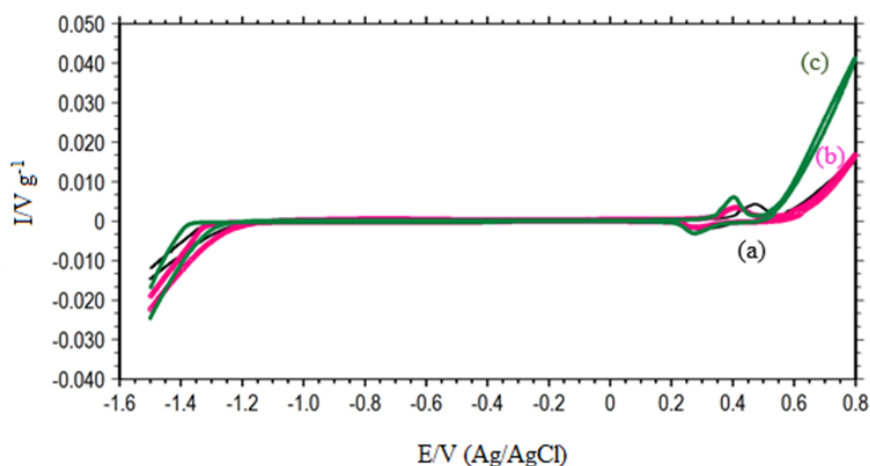


**Figure 4.2.** The I-V curves of G/NiCoMo-I electrode in 0.5 M (a), 1 M (b), and 1.5 M (c) KOH solutions with scan rate: 0.1 V s<sup>-1</sup>



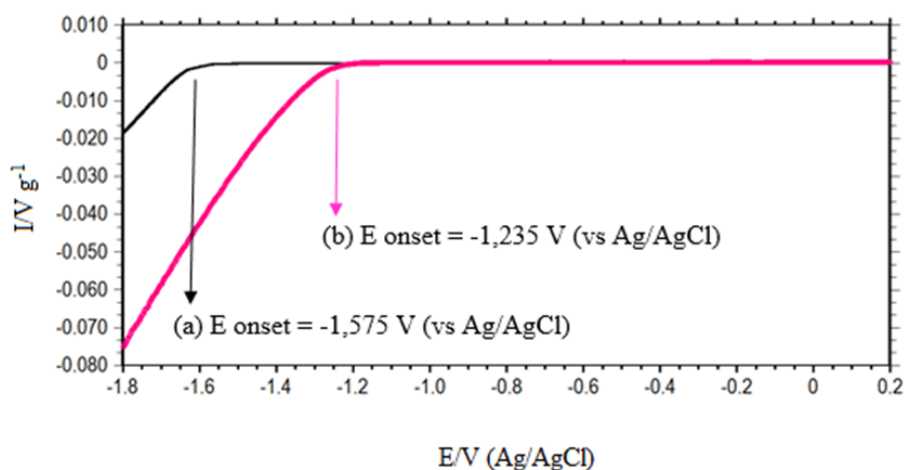
**Figure 4.3.** The I-V curves of G/NiCoMo-II electrode in 0.5 M (a), 1 M (b), and 1.5 M (c) KOH solutions with scan rate: 0.1 V s<sup>-1</sup>

In Figure 4.4, systematically increasing the catalyst amount resulted in current values at 0.6 V in the anodic region of 3.8 mA, 4.8 mA, and 7.3 mA corresponding to increasing base concentrations. In the cathodic region at -1.4 V, the current values were -9 mA, -13.3 mA, and -3.7 mA for the same sequence of catalyst amounts.



**Figure 4.4.** The I-V curves of G/NiCoMo-III electrode in 0.5 M (a), 1 M (b), and 1.5 M (c) KOH solutions with scan rate: 0.1 V s<sup>-1</sup>

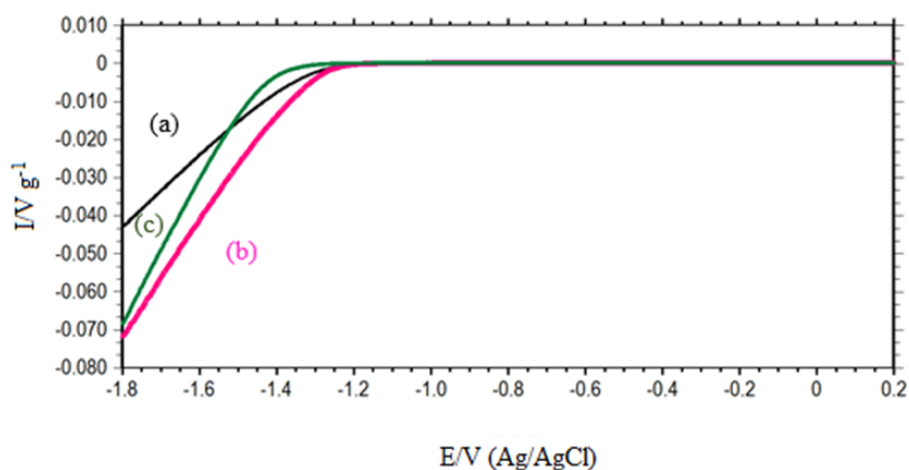
Comparing Figures 4.2 to 4.4 shows that increasing the catalyst amount (G/NiCoMo-I, G/NiCoMo-II, and G/NiCoMo-III) results in a higher current density in the anodic area of the 0.5 M solution. This observation implies a relationship with the existence of metal oxides that have formed. Additionally, upon evaluating the electrochemical behavior, it is observed in Figures 4.2-4.4 that during the positive scan direction, two anodic peaks are formed. The first anodic peak represents the Ni/NiII transition. Additionally, the conversion from  $\alpha$ -Ni(OH)<sub>2</sub> to  $\beta$ -Ni(OH)<sub>2</sub> occurs in the potential range of -0.600 V to 0.300 V. The second anodic peak corresponds to the transition from Ni(OH)<sub>2</sub> to NiOOH (NiII/NiIII) (Mert and Gardaş, 2011; Solmaz et. al., 2008; Solmaz et. al., 2009). The cathodic peak observed during the reverse scan corresponds to the reduction of NiII/NiIII (Mert and Gardaş, 2011; Solmaz et. al., 2010). Using linear sweep voltammetry (LSV) analysis, the hydrogen gas production performance of a bare graphite electrode (G) and electrodes modified with different concentrations of catalyst were assessed. Figure 4.5. shows the LSV measurements obtained in 1 M KOH solution.



**Figure 4.5.** The I-V curves for (a) G and (b) G/NiCoMo-I electrodes in 1 M KOH solutions with scan rate: 0.05 V s<sup>-1</sup>

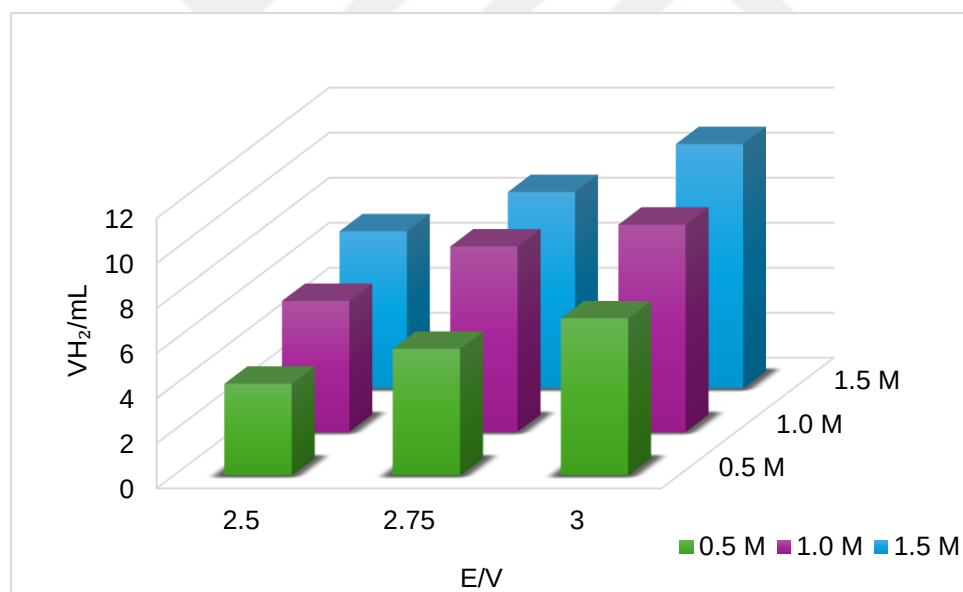
The hydrogen evolution potentials for the bare G and G/NiCoMo-I electrodes produced in 1 M KOH solutions differ significantly, as shown in Figure 4.5. When comparing the current measurements at -1.8 V, it can be seen that while the bare G electrode detects 18 mA, the G/NiCoMo-I electrode detects 76 mA, which is approximately four times greater. Additionally, the impact of catalyst quantity and electrolyte concentration has been studied. Figure 4.6 displays LSV readings in several solutions for comparison purposes. Figure 4.6 illustrates that a 1 M KOH solution is an appropriate electrolyte.





**Figure 4.6.** The I-V curves of G/NiCoMo-I electrode in 0.5 M (a), 1 M (b), and 1.5 M (c) KOH solutions with scan rate: 0.05 V s<sup>-1</sup>

Figure 4.7. illustrates the volumes of hydrogen produced by the G/NiCoMo-I electrode.

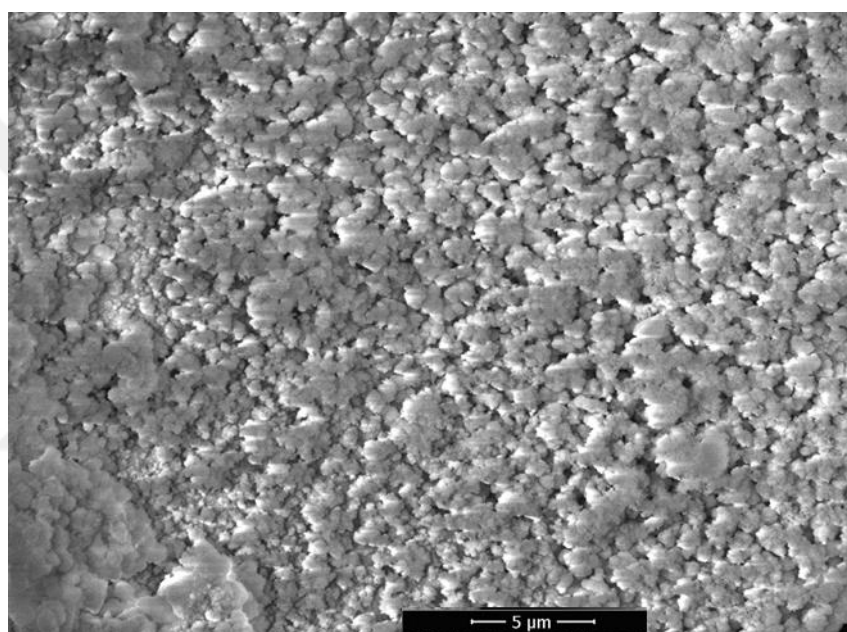


**Figure 4.7.** Hydrogen volumes for G/NiCoMo-I electrode over 15 minutes at various voltages

Figure 4.7. shows that the amount of hydrogen produced increases with applied voltage and the molar concentration of the KOH solution, based on data for the G/NiCoMo-I electrode. In particular, hydrogen production gradually increases from 4.07 mL at 2.5 V to 6.97 mL at 3.0 V with a constant molar concentration of 0.5 M. Similarly, at a concentration of 1 M, hydrogen

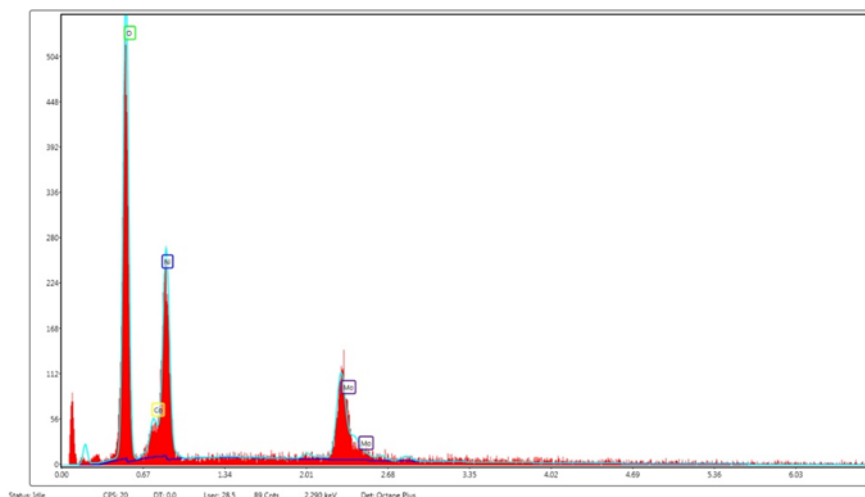
production rises from 5.81 mL at 2.5 V to 9.20 mL at 3.0 V. At a concentration of 1.5 M, the same pattern is observed, with hydrogen generation increasing from 6.97 mL at 2.5 V to 10.85 mL at 3.0 V.

As shown in Figure 4.8, a large surface area and uniform shape are desirable characteristics for an effective electrocatalyst surface. This image, taken with a scanning electron microscope at 10,000x magnification, reveals that the surface of the G/NiCoMo-I electrode is remarkably consistent, rugged, and suitable. It features spherical particles and deep fissures, which create an optimal surface for hydrogen gas evolution.



**Figure 4.8.** SEM image of G/NiCoMo-I electrode

Although the SEM study can reveal the surface shape, it cannot determine the elemental composition of the surface. Therefore, EDX analysis is necessary. The EDX spectrum of the G/NiCoMo-I electrode is shown in Figure 4.9. Peaks for Ni, Co, and Mo are detected on the graphite surface, indicating that the ternary coating was successfully deposited. Table 4.1. lists the elemental compositions determined by the EDX analysis presented in Figure 4.9.



**Figure 4.9.** EDX analysis of the G/NiCoMo-I electrode

**Table 4.1.** Chemical composition of G/NiCoMo-I electrode

| Element | Weight (%) |
|---------|------------|
| O K     | 41.77      |
| Co L    | 3.06       |
| Ni L    | 31.97      |
| Mo L    | 23.20      |

The proportions by weight are approximately 32% Nickel (Ni), 23% Molybdenum (Mo), and 3% Cobalt (Co). The presence of oxide peaks on the surface results from the coating being exposed to surrounding air, which leads to the formation of metal oxides after deposition. This finding is consistent with previous research. Importantly, the existence of these peaks indicates that the ternary coating on the graphite surface was successfully formed.

## 4.2. Response Surface Methodology

### 4.2.1. Model equation

Equations 3 and 4 provide the coded values for the model equation developed using the surface response technique for mild steel and modified graphite electrodes, respectively. The formula used to describe the electrode voltage and molarity was A and B, respectively.

$$Y = +4.54 + 1.60A + 1.48B + 0.6775AB + 0.0972A^2 - 0.1478B^2 \quad (8)$$

$$Y = 8.12 + 1.69A + 1.65B + .02450AB - 0.3533A^2 - 0.6883B^2 \quad (9)$$

#### 4.2.2. Analysis of variance (ANOVA)

Tables 4.2 and 4.3 present the model summary statistics for H<sub>2</sub> production for mild steel and modified graphite electrodes, respectively. To accommodate the experiment result, this study used mathematical models based on linear, two-factor interaction, quadratic and cubic terms.

**Table 4.2.** Model Summary for mild steel electrode

| Source           | F-value | P-value | R <sup>2</sup> | Adjusted R <sup>2</sup> | Predicted R <sup>2</sup> | Significance |
|------------------|---------|---------|----------------|-------------------------|--------------------------|--------------|
| <b>Linear</b>    | 73.54   | <0.0001 | 0.9363         | 0.8239                  | 0.8205                   |              |
| <b>2FI</b>       | 159.38  | <0.0001 | 0.9966         | 0.9955                  | 0.9916                   | Suggested    |
| <b>Quadratic</b> | 6.09    | 0.0297  | 0.9979         | 0.9979                  | 0.9879                   | Suggested    |
| <b>Cubic</b>     | 15.47   | 0.0072  | 0.9996         | 0.9996                  | 9799                     | Aliased      |

**Table 4.3.** Model Summary for modified graphite electrode

| Source           | F-value | P-value | R <sup>2</sup> | Adjusted R <sup>2</sup> | Predicted R <sup>2</sup> | Significance |
|------------------|---------|---------|----------------|-------------------------|--------------------------|--------------|
| <b>Linear</b>    | 52.20   | <0.0001 | 0.9126         | 0.8951                  | 0.8440                   |              |
| <b>2FI</b>       | 0.7278  | 0.4117  | 0.9191         | 0.8922                  | 0.7458                   |              |
| <b>Quadratic</b> | 20.28   | 0.0012  | 0.9881         | 0.9796                  | 0.9163                   | Suggested    |
| <b>Cubic</b>     | 0.1714  | 0.8472  | 0.9889         | 0.9733                  | -0.2941                  | Aliased      |

The ANOVA results from the central composite design indicate that the RSM model has statistical significance. The effects of electrode voltage and molarity on hydrogen production efficiency were examined using the ANOVA method. Using this method, the Fisher (F-test)

was used to assess the statistical significance of the square impacts of each component on the replies at a 95% confidence level. An increase in Fisher values (F) indicates greater importance of terms in a model, while a decrease in p-values is also expected. Variables in the model that have values higher than 0.05 are eliminated because they are deemed insignificant. The fit quality of compatible models is assessed using the coefficient of determination,  $R^2$ . For an ideal model fit, the  $R^2$  value should be approximately 1.0 (Singh vd., 2019). Tables 4.4 and 4.5 present the results of the analysis of variance (ANOVA) that was utilized to ascertain the models' significance for mild steel and modified graphite electrodes, respectively. According to Table 4.4. and 4.5., the F values are 1122.15 and 116.25, respectively the p-value is  $<0.0001$ , indicating that the designed models are statistically significant.

**Table 4.4.** Variance Analysis for mild steel electrode

| Source                         | Sum of Squares | df | Mean Square | F-value | P-Value   | Significance |
|--------------------------------|----------------|----|-------------|---------|-----------|--------------|
| <b>Model</b>                   | 30.43          | 5  | 6.09        | 1122.15 | $<0.0001$ | Significant  |
| <b>A-Electrode Voltage (V)</b> | 15.30          | 1  | 15.30       | 2820.41 | $<0.0001$ |              |
| <b>B-Molarity (M)</b>          | 13.23          | 1  | 13.23       | 2439.70 | $<0.0001$ |              |
| <b>AB</b>                      | 1.84           | 1  | 1.84        | 338.54  | $<0.0001$ |              |
| <b>A<sup>2</sup></b>           | 0.0261         | 1  | 0.0261      | 4.82    | 0.0643    |              |
| <b>B<sup>2</sup></b>           | 0.0603         | 1  | 0.0603      | 11.12   | 0.0125    |              |
| <b>Residual</b>                | 0.0380         | 7  | 0.0054      |         |           |              |
| <b>Lack of Fit</b>             | 0.0380         | 3  | 0.0127      |         |           |              |
| <b>Pure Error</b>              | 0.0000         | 4  | 0.0000      |         |           |              |
| <b>Cor Total</b>               | 30.47          | 12 |             |         |           |              |

**Table 4.5.** Variance Analysis for modified graphite electrode

| Source                         | Sum of Squares | df | Mean Square | F-value | P-Value | Significance |
|--------------------------------|----------------|----|-------------|---------|---------|--------------|
| <b>Model</b>                   | 36.28          | 5  | 7.26        | 116.25  | <0.0001 | Significant  |
| <b>A-Electrode Voltage (V)</b> | 17.24          | 1  | 17.24       | 276.17  | <0.0001 |              |
| <b>B-Molarity (M)</b>          | 16.27          | 1  | 16.27       | 260.64  | <0.0001 |              |
| <b>AB</b>                      | 0.2401         | 1  | 0.2401      | 3.85    | 0.0906  |              |
| <b>A<sup>2</sup></b>           | 0.3447         | 1  | 0.3447      | 5.52    | 0.0511  |              |
| <b>B<sup>2</sup></b>           | 1.31           | 1  | 1.31        | 20.96   | 0.0025  |              |
| <b>Residual</b>                | 0.4369         | 7  | 0.0624      |         |         |              |
| <b>Lack of Fit</b>             | 0.4369         | 3  | 0.1456      |         |         |              |
| <b>Pure Error</b>              | 0.0000         | 4  | 0.0000      |         |         |              |
| <b>Cor Total</b>               | 36.72          | 12 |             |         |         |              |

The correlation coefficient ( $R^2$ ), adjusted coefficient of determination (Adjusted  $R^2$ ), anticipated coefficient of determination (anticipated  $R^2$ ), adequate precision, and coefficient of variation (CV) are among the statistical parameters used to assess the model fit. The model's evaluation results are shown in Table 4.6.

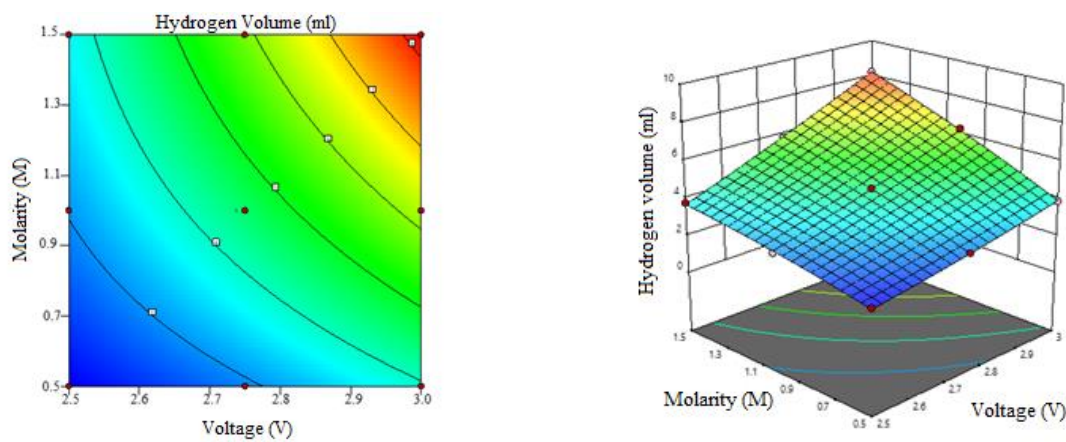
**Table 4.6.** Model evaluation

| <b>For mild steel electrode</b>        |        |                                |        |
|----------------------------------------|--------|--------------------------------|--------|
| <b>Std. Dev.</b>                       | 0.0736 | <b>R<sup>2</sup></b>           | 0.9988 |
| <b>Mean</b>                            | 4.51   | <b>Adjusted R<sup>2</sup></b>  | 0.9979 |
| <b>C.V. %</b>                          | 1.63   | <b>Predicted R<sup>2</sup></b> | 0.987  |
| <b>For modified graphite electrode</b> |        |                                |        |
| <b>Std. Dev.</b>                       | 0.2498 | <b>R<sup>2</sup></b>           | 0.9881 |
| <b>Mean</b>                            | 7.64   | <b>Adjusted R<sup>2</sup></b>  | 0.9796 |
| <b>C.V.%</b>                           | 3.27   | <b>Predicted R<sup>2</sup></b> | 0.9163 |

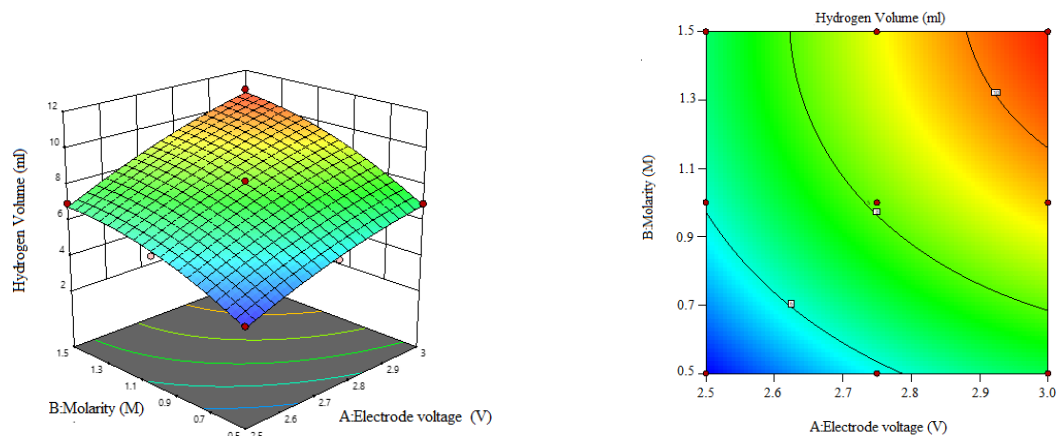
The experimental values' high level of accuracy and dependability is shown by the coefficient of variation (CV) of 1.6 and 3.27 for mild steel and modified graphite electrode, respectively, which is less than 10. The  $R^2$  regression coefficient of the models are quite high at 0.9988 and 0.9881 for mild steel and modified graphite electrodes, respectively. This suggests that every variable in the experimental design may have response factors that may be precisely calculated using the second-order regression model. Furthermore, the closeness of the  $R^2$  and adjusted  $R^2$  values suggests that the proposed model fits the experimental data well (Özgür, mert, 2022).

#### 4.2.3. Effects of parameters on hydrogen production

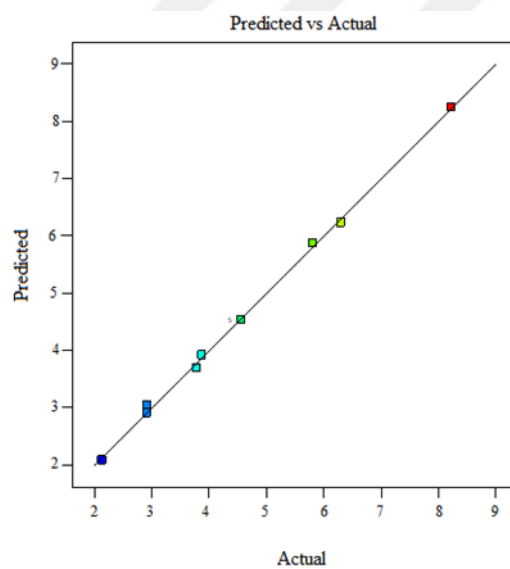
Dependent variables are used to measure the effects of independent variables on hydrogen generation. Therefore, identifying these dependent variables is essential (Özgür and Mert, 2022). The 2D and 3D response surface plots of electrode voltage and molarity against hydrogen volume are shown in Figures 4.10 and 4.11, for mild steel and modified graphite electrodes, respectively. The positivity of the A and B values in Equations 8 and 9 indicate that increasing voltage and molarity enhances the hydrogen volume. The actual values from the hydrogen model equation were used to generate predicted outcomes. Figures 4.12 and 4.13 illustrate the relationship between these predictions and the test results. The correlation between the experimental and model-predicted hydrogen production volumes was significantly consistent, as shown in Figures 4.12 and 4.13.



**Figure 4.10.** 2D and 3D Response Surface Plot: Electrode Voltage (V) and Molarity (M) for mild steel electrode

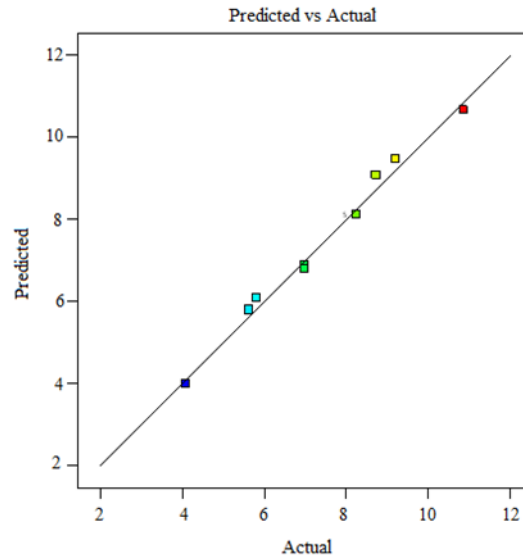


**Figure 4.11.** 2D and 3D Response Surface Plot: Electrode Voltage (V) and Molarity (M) for modified graphite electrode



**Figure 4.12.** Comparison between the predicted and actual results for mild steel electrode



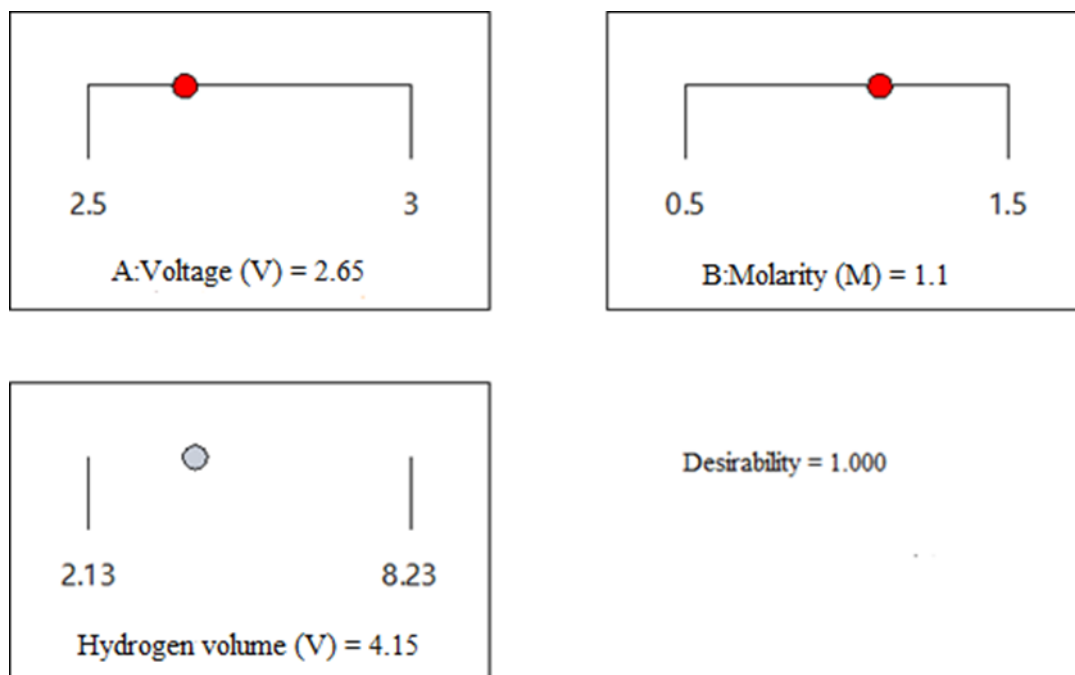


**Figure 4.13.** Comparison between the predicted and actual results for modified graphite electrode

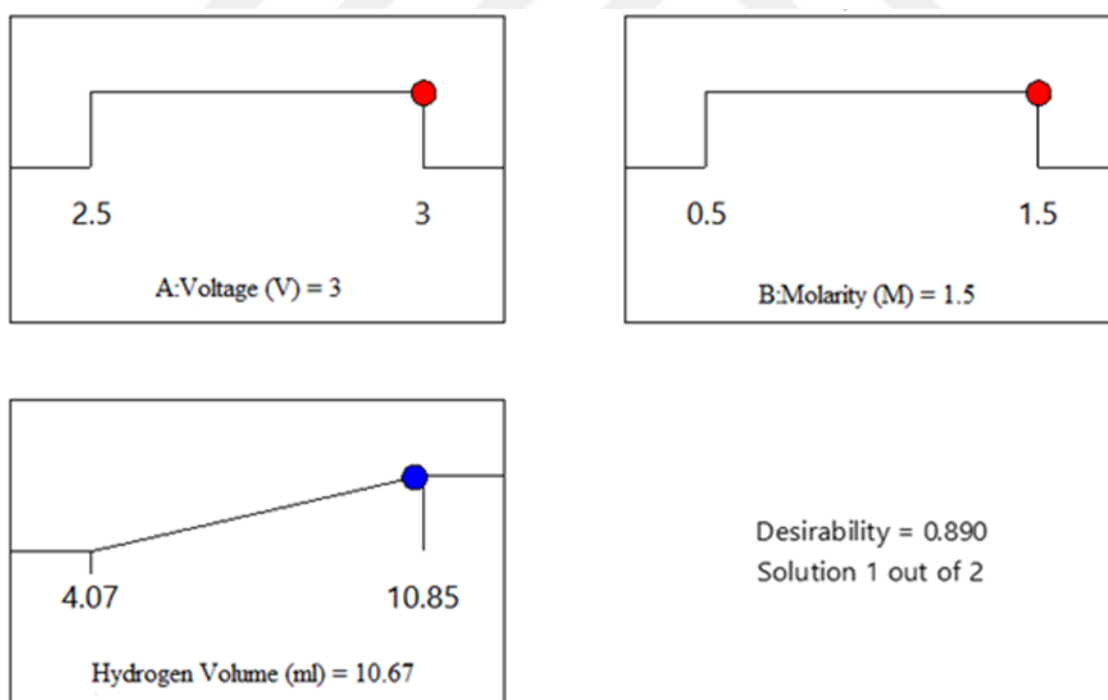
#### 4.2.4. Optimization and validation

RSM optimization uses a regression model to identify the input parameters that maximize the hydrogen output for an integrated system. In this process, while two factors are kept within specific ranges, the response variable is adjusted to reach its maximum level. A suitable solution has been obtained using the desirability approach with Design Expert software. The desirability value of the preferred solution is 1.0, indicating it is closest to the optimization criteria. The set of ideal conditions is more desirable the higher its desirability.

The optimal electrode voltage and molarity for hydrogen production are 2.65 V and 1.1 M for mild steel electrodes and 3 V and 1.5 M for modified graphite electrodes, respectively. This solution is based on the desirability approach. The optimization criteria solution is illustrated in Figure 4.14. and 4.15. for mild steel and modified graphite electrodes, respectively. Table 4.7 presents the results of a verification experiment that was carried out to validate the optimization outcomes. The experimental results showed a good agreement between the actual hydrogen production values and the values predicted by the statistical model for mild steel and modified graphite electrodes.



**Figure 4.14.** RSM Optimization results for mild steel electrode



**Figure 4.15.** RSM Optimization results for modified graphite electrode

**Table 4.7.** Validation tests

| For mild steel electrode        |              |           |                         |
|---------------------------------|--------------|-----------|-------------------------|
| Electrode Voltage<br>(V)        | Molarity (M) | Value     | Hydrogen volume<br>(mL) |
| 2.65                            | 1.1          | Optimum   | 4.15                    |
|                                 |              | Actual    | 4.085                   |
|                                 |              | Error (%) | 1.5                     |
| For modified graphite electrode |              |           |                         |
| Electrode Voltage<br>(V)        | Molarity (M) | Value     | Hydrogen volume<br>(mL) |
| 3                               | 1.5          | Optimum   | 10.67                   |
|                                 |              | Actual    | 10.85                   |
|                                 |              | Error (%) | 1.7                     |

## 5. CONCLUSION

Due to the rapidly depleting fossil fuel supplies and the damaging effects of fossil fuel combustion on the environment, finding alternative energy sources has become a priority. Utilizing sustainable fuels like hydrogen is one appropriate solution to this issue.

In this study, a laboratory-scale, two-electrode system has been set up for hydrogen gas production using an alkaline electrolysis cell. Firstly, graphite was used as the anode, while mild steel electrodes were used as the cathode. Then platinum, a noble metal, was used as the anode, while graphite electrodes were preferred as the cathode. These graphite electrodes were modified with nickel-cobalt-molybdenum (NiCoMo) using a galvanostatic method.

This study aims first to identify the purpose and parameters of process design and then to work with a suitable process model to create a performance index. Sensitivity analysis is used to identify the effective design variables, while response surface methodology (RSM) and experimental design (DoE) are employed to optimize the system. To identify specific responses, experiments were conducted using the trial version of Design-Expert software.

According to the response surface method, under optimal conditions of 2.65 V for electrode voltage and 1.1 M for molarity, a total of 4.15 ml of hydrogen was obtained with a desirability level of 1 for mild steel electrodes.

The optimal production parameters for the modified graphite electrode were 1.5 M molarity and 3 V electrode voltage. Under these ideal conditions, a hydrogen volume of 10.67 mL was achieved. The experimentally obtained hydrogen volume of 10.85 mL was consistent with the expected value. The error, determined by comparing the predicted and experimental values from the RSM model, was 1.7%. The response surface methodology's desirability method achieved the best desirability of 0.890 for hydrogen production.

The  $R^2$ , Adj  $R^2$ , and Pred.  $R^2$  values obtained for hydrogen volume using the response surface methodology are 0.9988, 0.9979, and 0.9879 for mild steel electrodes and 0.9881, 0.9796, and 0.9163 for modified graphite electrodes, respectively.

The validation test results have demonstrated a good agreement between the experimentally obtained hydrogen volume results, and the predicted hydrogen volume results.

Based on the results of this investigation, it can be concluded that optimizing operating parameters using response surface methodology (RSM) is essential for maximizing hydrogen production performance and saving time.



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