

Electrochemical Production of Transition Metal-Based Electrocatalysts for Hydrogen Production

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ABSTRACT

Energy demand increases with increasing population and industrial developments. Unfortunately, non-renewable sources are used to meet a large portion of the demand. As a result of the negative effects, researchers have begun to investigate renewable sources by working in a variety of fields in order to ensure the energy and humanity's long-term sustainability. Renewable energy sources are very important for the future of the world. In this regard, we can claim that the convenient solution is “hydrogen” which is a promising energy carrier. In this study, alkaline electrolysis system was preferred due to the advantage of being easily integrated with renewable energy sources to meet the power requirements of the system. The platinum (Pt) and stainless steel (SS) electrodes were used as anodes. Graphite (G) and ternary transition metals (nickel, copper, cobalt) modified graphite (G/NiCoCu) electrodes were used as cathode. The modified electrodes were prepared by using a galvanostatic method. The various operation voltages were applied that varied from 2.3V to 3V in order to realize water splitting reaction. The amount of produced hydrogen gas was increased with increasing operation voltage. According to the experimental results, at 3V for 15 minutes of electrolysis time, the produced H₂ gas values were 15 mL, 11.13 mL and 13.12 mL for Pt - G (Cell-1), SS - G (Cell-2) and SS - G/NiCoCu (Cell-3), respectively.

Hidrojen Üretimi için Geçiş Metali İhtiva Eden Elektrokatalizörlerin Elektrokimyasal Üretimi

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ÖZET

Artan nüfus ve endüstriyel gelişmelerle birlikte enerji talebi de artmaktadır. Ne yazık ki, talebin büyük bir kısmını karşılamak için yenilenemeyen kaynaklar kullanılmaktadır. Olumsuz etkilerin bir sonucu olarak, araştırmacılar enerjinin ve insanlığın uzun vadeli sürdürülebilirliğini sağlamak için çeşitli alanlarda çalışarak yenilenebilir kaynakları araştırmaya başlamışlardır. Yenilenebilir enerji kaynakları dünyanın geleceği için çok önemlidir. Bu bağlamda, uygun çözümün gelecek vadeden bir enerji taşıyıcısı olan “hidrojen” olduğunu söyleyebiliriz. Bu çalışmada, sistemin güç ihtiyacını karşılamak için yenilenebilir enerji kaynakları ile kolayca entegre edilebilme avantajından dolayı alkali elektroliz sistemi tercih edilmiştir. Anot olarak platin (Pt) ve paslanmaz çelik (SS) elektrotlar kullanılmıştır. Katot olarak grafit (G) ve üçlü

(nikel, bakır, kobalt) geçiş metalleriyle modifiye edilmiş grafit (G/NiCoCu) elektrotlar kullanılmıştır. Modifiye elektrotlar, galvanostatik yöntem kullanılarak hazırlanmıştır. Suyun ayrışma reaksiyonunu gerçekleştirmek için 2,3V ile 3V arasında değişen çeşitli çalışma potansiyelleri uygulanmıştır. Artan uygulama potansiyeli ile üretilen hidrojen gazı ($H_{2(g)}$) miktarı artmıştır. Deneysel sonuçlara göre, 3V'de 15 dakikalık elektroliz süresi boyunca üretilen $H_{2(g)}$ miktarları, Pt - G (Hücre-1), SS - G (Hücre-2) ve SS - G/ NiCoCu (Hücre-3) için sırasıyla 15 mL; 11,13 mL ve 13,12 mL'dir.

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Introduction

Energy is an important element needed in people's daily lives and is also the infrastructure of technology. Unfortunately, this energy that we use to meet our needs and to develop technology has a certain limit. The increase in population rates, the increase in settlements and the increase in the demand for energy accelerate fossil fuel consumption. Humanity has used oil, coal, wood, fossil fuels, in short, non-renewable energy resources to meet the increasing demand for the centuries. The harmful products resulting from the consumption of fossil fuels, unfortunately, pollute our world (Barbir et al., 1990; Shafiee and Topal, 2009; Andres et al., 2011; Hanif et al., 2019; Martins et al., 2019; Waheed et al., 2019). In this context, we need renewable and sustainable energy in order to meet the needs and reduce the harmful effects on the environment. Thus, we will be able to reduce the level of risk and the damage we cause to the environment. Our country's physical-geographical location is very suitable for renewable energy production, and we are in a very efficient position, especially in terms of geothermal, solar and wind energy (Şenel and Koç, 2015; Koç et al., 2018; Çetin et al., 2019; Bekar, 2020; Güllü and Kartal, 2021). We should give much effort in this field, and we should contribute to the development of Turkey. Hydrogen should be convenient option in the energy arena of the future. Because the combustion of hydrogen produces merely water. It provides zero carbon emissions and has a high energy density, making it the most promising form of energy for the future. Although there are many methods for the production of hydrogen gas, alkaline water electrolysis method comes to the fore (Suermann et al., 2016). Because almost 99.9% pure hydrogen can be produced by this method (de Fátima Palhares et al., 2018). For the establishment and dissemination of industrial-scale systems, the overvoltage of the electrodes should be low, and the investment costs should be appropriate. Electrode selection is extremely important in the water decomposition process. While designing the electrodes, price-performance research was conducted for oxygen and hydrogen production efficiency. In the literature, Platinum (Pt) based materials are known as the most efficient electrocatalysts for hydrogen production due to their low overvoltage and high current density. However, Pt material is not in a cost-effective position. Therefore, we should produce electrocatalysts which are used as Pt alternatives for alkaline electrolysis. An electrode material that can act as an alternative to Pt electrodes and function effectively for hydrogen production has been extensively investigated. In this context, we chose to use SS on the anode side. Due to the affordability, chemical stability and catalytic properties of SS, it contributes to reducing the overpotential of hydrogen production compared to other

transition metals (Kim et al., 2017). On the cathode side, we preferred graphite (G). With its electrical conductivity, high corrosion resistance and low cost, it meets exactly the criteria we are looking for. Instead of using G lonely, based on the superior performance of dual coatings combining Ni-Co and Ni-Cu in the literature, triple coating on graphite was preferred and Ni-Co-Cu was used. Ni is one of the promising non-new metals with the highest current exchange density to the hydrogen evolution reaction, and recently, Ni-based alloy catalysts have been investigated to obtain high catalytic activity by electrolysis in alkaline water (Greeley et al., 2006; Aydın, 2021; Farsak and Aydın, 2021). Cu is the most suitable for alloying with Ni due to its low cost, high corrosion resistance and environmental friendliness (Ahn et al., 2013; Kumar and Shetti, 2018).

The main purpose of this study is to produce hydrogen gas by alkaline electrolysis process with energy obtained from a renewable energy source. At the same time, different types of anode materials were studied to improve the study. Two different anode materials (i.e., Pt and SS) were used for the electrolysis cell. Graphite and ternary metal deposited (i.e., nickel, copper, cobalt) graphite were used as the cathode. The main objectives of this study are explained as follows:

- To produce hydrogen gas by alkaline electrolysis process through different anodes and cathodes,
- To discover the most suitable material for electrolysis by comparing the anodes and cathodes.

Material and Method

In this study, graphite (G) electrode was preferred as cathode in our system due to its low cost. Copper wire was passed to one end of a graphite rod to provide conductivity. The electrode is 5 cm in length and embedded in polyester. The surface area of the prepared graphite electrode, which was not covered with resin in contact with the solution, is 0.36 cm^2 . In Cell 1, a platinum (Pt) sheet with a surface area of 2 cm^2 was used as the anode electrode and graphite was used as the cathode. Stainless steel (SS) with a surface area of 0.78 cm^2 was used as anode electrode in Cells 2 and 3, graphite and modified graphite were used as cathodes, respectively. To ensure conductivity, a 5 cm long copper wire was passed to the end of the Pt plate and the SS electrode. The SS electrode is embedded in the polyester so that only the surface area remains free. Except for the Pt electrode, the electrode surface was abraded up to 1200 degrees with sandpaper before electrodeposition processes. Then, each electrode was washed with acetone/ethanol mixture and distilled water. Two electrode techniques were used, several anodes were examined. In this work, ternary coating was favored on graphite and Ni-Co-Cu was employed, based on the outstanding performance of binary coatings combining Ni-Co and Ni-Cu in the literature (Solmaz et al., 2008; Solmaz et al., 2009; Demirdelen et al., 2020). Considering the literature, the coating's Co and Cu mole ratios should be kept low in comparison to the Ni mole ratio (Mert and Kardaş, 2011; Sun et al., 2021). The electrodeposition of nickel, cobalt and copper was performed by galvanostatically using Iviumstat Electrochemical Interface with a three-electrode configuration. In this system graphite was used as working electrode, nickel as counter electrode, and

an Ag/AgCl (3 M KCl) electrode was used as the reference electrode. A current density was 50 mA cm⁻² and a coating thickness was 10 μm. Chemical composition of coating baths were; nickel bath 30% NiSO₄.7H₂O, 1% NiCl₂.6H₂O, and 1.25% H₃BO₃; cobalt bath 30% CoSO₄.7H₂O, 1% CoCl₂.6H₂O, 1.25% H₃BO₃; copper bath 26.67% CuSO₄.5H₂O, 1.25% H₃BO₃. NiCoCu ternary deposition was prepared by mixing in appropriate volumes to contain [Ni²⁺], [Co²⁺], [Cu²⁺] ions at different concentrations in the deposition bath which was 50 mL. [Ni²⁺]: [Co²⁺]: [Cu²⁺] mole ratio is 99: 0.5: 0.5.

Electrolysis was carried out using a direct current source and the amount of hydrogen produced both experimentally and theoretically at constant voltage. The theoretical calculations obtained by current-voltage curves with the help of Faraday Laws. A multimeter was used to determine the current flowing through the system. The cell components are given in Table 1.

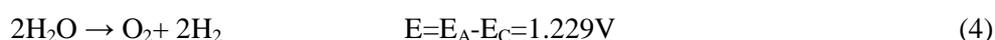
Table 1. Working electrodes used in the electrolysis system in three different cells

Cell	Anode	Cathode
Cell-1	Pt	G
Cell-2	SS	G
Cell-3	SS	G/NiCoCu

The minimum potential required for electrode reactions to start is called the decomposition voltage. The theoretical decomposition voltage (E_d) can be found by calculating the electrode potentials corresponding to the equilibrium state of the anode and cathode with the Nernst Equation in Eq. (1) (Yazici et al., 1995);

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

Theoretically, at 25 °C, the potential required for water to decompose is 1.23 V, where the evolution of hydrogen gas begins (Yörük et al., 2019). However, a higher potential must be applied because of the over voltages caused by the solution and electrode metal and the internal resistance of the voltage source and the cables used during the experiment. During the electrolysis of alkaline water, oxygen is released at the anode as in Eq. (2) and hydrogen gas is released at the cathode as in Eq. (3) (Kardaş et al., 2003).



In order to determine the decomposition voltages of the cells, Pt and SS electrodes on the anode sides; G and modified G electrodes on the cathode side were used in 1 M KOH solution. By using a direct current source, voltages between 1.3 V and 3 V were applied to the electrodes and applied to the system at 0.1 V intervals, and current potential curves were obtained by determining the current values

passing through the system. Since the theoretical split voltage is about 1.3 V, the studies started with 1.3 V and continued. After the decomposition voltage was determined as 1.8 V for Cell-1 and 1.9 V for Cell-2-3, hydrogen production volumes were observed at different voltage values. The amount of hydrogen production was investigated by placing the cylinder upside down on the cathode electrode where the hydrogen gas generation was located. The volumes of hydrogen produced were measured from 2.3 V to 3 V by increasing 0.1 V. The volume amounts displayed in the cylinder were noted at 3-minute intervals at each potential. Each voltage was applied to the system for 15 minutes. Considering that water vapor was collected in the cylinder in the same environment, the volume correction was made as follows (Kardaş et al., 2003);

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

When the vapor pressure of water (P_{H_2O}) under these conditions is 23,756 mm Hg and the total atmospheric pressure (P_T) is 756 mm Hg, the pressure of hydrogen (P_{H_2}) gas under these conditions is determined as 732,244 mm Hg. Hydrogen volume (V_{H_2}) is;

$$V_{H_2} = \frac{732,244}{756} \times V_{\text{measuring}} \quad (6)$$

determined by the relation. The $V_{\text{measuring}}$ here is the total volume of gas collected experimentally in the cylinder.

Results and Discussion

Theoretically, the potential required for water decomposition at 25°C is 1.229 V. When the O₂ overvoltage (0.47 V) on Pt as the anode is added to this value, a potential of at least 1.70 V must be applied to the system to initiate the hydrogen gas evolution. The hydrogen gas was measured for the Cell-1 in 1 M KOH. Experimental decomposition voltage was calculated for each electrode in order to compare the volumes and the effectiveness of the deposition with each other. The decomposition voltage (E_d) for the Cell-1 (Pt anode-G cathode) electrode was 1.8 V and the overvoltage E_{over} was 0.5V in Eq. (7).

$$E_{\text{over}} = E_d - 1.3 = 1.7 - 1.3 \quad (7)$$

$$E_{\text{over}} = 0.4 \text{ V}$$

Hydrogen production volumes of Cell-1 at different voltage values are given in Table 2. The volumes of hydrogen produced were measured from 2.3 V to 3 V by increasing 0.1 V and the current values passing through the system at each potential are given. After 15 minutes, it was found to produce 15 mL of hydrogen with a Pt electrode.

Table 2. Total charge (Q) during 15 minutes electrolysis, theoretical (Vt) and experimental (Ve) hydrogen volumes at the end of 15 minutes for various voltages (E) for G electrode in case of Cell-1

E/V	Q (Coulomb)	Vt (mL)	Ve (mL)
2.3	39.0	4.54	3.50
2.4	50.0	5.79	4.75
2.5	63.0	7.29	6.25
2.6	73.5	8.54	7.50
2.7	86.5	10.04	9.00
2.8	99.5	11.54	10.50
2.9	129.5	15.04	14.00
3.0	138.0	16.04	15.00

The hydrogen gas was measured for the Cell-2 in 1 M KOH and given in Equation (11). The decomposition voltage was calculated for each electrode in order to compare the volumes and the effectiveness of the deposition with each other. The decomposition voltage (E_d) for the Cell-2 (SS anode-G cathode) electrode was 1.9 V and the overvoltage E_{over} was 0.6 V in Eq. (8).

$$E_{over} = E_d - 1.3 = 1.9 - 1.3 \quad (8)$$

$$E_{over} = 0.6 \text{ V}$$

Hydrogen production volumes of Cell-2 at different voltage values are given in Table 3. The volumes of hydrogen produced were measured from 2.3 V to 3 V by increasing 0.1 V and the current values passing through the system at each potential are given. After 15 minutes, it was found to produce 11.13 mL of hydrogen with an SS electrode.

Table 3. Total charge (Q) during 15 minutes electrolysis, theoretical (V_t) and experimental (V_e) hydrogen volumes at the end of 15 minutes for various voltages (E) for G electrode in case of Cell-2

E/V	Q (Coulomb)	Vt (mL)	Ve (mL)
2.3	23.88	2.77	2.42
2.4	33.48	3.88	3.58
2.5	56.52	6.56	6.30
2.6	65.16	7.56	7.26
2.7	74.52	8.65	8.13
2.8	82.80	9.61	9.20
2.9	88.36	10.26	10.16
3.0	97.74	11.34	11.13

The hydrogen gas was measured for the Cell-3 in 1 M KOH. Experimental dissociation voltage was calculated for each electrode in order to compare the volumes and the effectiveness of the coatings with each other. The decomposition voltage (E_d) for the Cell-3 (SS anode-G/NiCoCu cathode) electrode was 1.9 V and the overvoltage E_{over} was 0.6 V in Eq. (9).

$$E_{over} = E_d - 1.3 = 1.9 - 1.3 \quad (9)$$

$$E_{over} = 0.6 \text{ V}$$

Hydrogen production volumes of Cell-3 at different voltage values are given in Table 4. The volumes of hydrogen produced were measured from 2.3 V to 3 V by increasing 0.1 V and the current values passing through the system at each potential are given. After 15 minutes, it was found to produce 13.12 mL of hydrogen with an SS electrode.

Table 4. Total charge (Q) during 15 minutes electrolysis, theoretical (V_t) and experimental (V_e) hydrogen volumes at the end of 15 minutes for various voltages (E) for SS electrode in case of Cell-3

E/V	Q (Coulomb)	V_t (mL)	V_e (mL)
2.3	52.83	6.14	5.94
2.4	67.68	7.86	7.60
2.5	73.98	8.60	8.30
2.6	84.42	9.80	9.48
2.7	91.26	10.60	10.25
2.8	99.63	11.56	11.20
2.9	107.73	12.50	12.10
3.0	116.82	13.56	13.12

Hydrogen production of each cell was observed with a graduated cylinder filled with 1 M KOH electrolyte. The hydrogen performance of Pt-G (Cell-1), SS-G (Cell-2), and SS-G/NiCoCu (Cell-3) is compared in Figure 1. According to the study of Koca et al. (2019), in the alkaline electrolyte, Platinum (Pt) was utilized as the anode, CF, CF / Ni, CF / NiGa were utilized as the cathode electrodes, and 3V was operated in the cell for 30 minutes. The highest hydrogen production efficiency was detected for the Pt anode - CF/NiGa cathode which produced almost 0.92 mL hydrogen gas per minute. For the comparison of this study, the operation time was chosen as 15 minutes by the way the produced gas volume almost 13.75 mL. According to the study of Kaya et al. (Kaya et al., 2017), for steel and graphite electrodes at 4 V for concentration 5 wt. %, 10 wt. % and 15 wt. % KOH, 0.58 mL/min, 0.95 mL/min, 1.18 mL/min, hydrogen gas was accumulated, respectively. In this study, 15 mL, 11.13 mL and 13.12 mL hydrogen gas were produced, respectively, for 15 minutes by applying a voltage of 3 V. Although Cell-1 has the highest hydrogen production values at higher potentials (2.9 V and 3 V), for the lower potentials (from 2.3 V to 2.8 V), the Cell-3 was more favorable. Consequently, in the range of 2.3 V and 2.8 V, modified cathode (G/NiCoCu) enlarged catalytic activity.

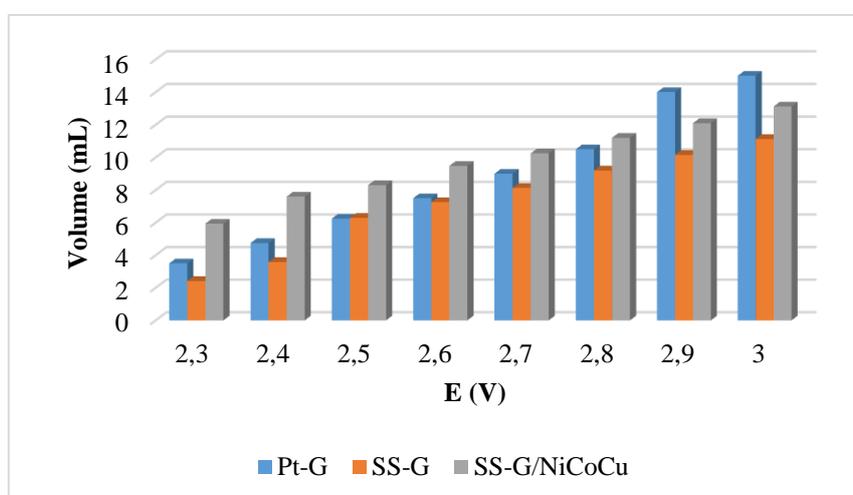


Figure 1. Comparison of hydrogen amounts produced by Pt-G, SS-G, and SS-G/NiCoCu electrodes after 15 minutes at 2.3 V to 3 V

Conclusion

We examined the several electrodes for producing hydrogen, which is trend topic and has recently attracted the concern of academics interested in renewable and sustainable energy. Results showed that, produced hydrogen gas values were 15 mL; 11.13 mL and 13.12 mL at 3V for 15 minutes electrolysis period, by using Pt-G (Cell-1); SS-G (Cell-2) and SS-G/NiCoCu (Cell-3), respectively. Although Cell-1 exhibited better efficiency than other electrodes at higher potentials, due to the high cost of Pt anode, SS anode was preferable. We recommend SS anode for hydrogen production, both in terms of cost and corrosion resistance. And we suggest the SS-G/NiCoCu electrode due to high hydrogen evolution performance, especially at lower potentials. In comparison to all cells, Cell-3 was observed to be the most convenient setup for alkaline electrolysis.

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Statement of Conflict Interest

The authors have no conflicts of interest to declare.

Author's Contributions

The experimental studies were done by Eren ERGEN, discussion and writing of the manuscript was achieved by all author.

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