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Research Paper

The Influence of Catalyst Loading on Electrocatalytic Activity and Hydrogen Production in PEM Water Electrolysis

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Abstract
The climate change caused by the widespread and continuous use of fossil fuels is a problem that needs to be addressed urgently. One of the solutions offered is through an energy transition towards the use of new or renewable and low-carbon fuels. Hydrogen gas as a carrier of energy is an alternative solution that has attracted the attention of researchers, due to its high combustion energy and environmental friendliness. The production of hydrogen gas using the Proton Exchange Membrane Water Electrolysis (PEMWE) method is considered effective for large-scale production. This study investigates the impact of catalyst loading and various current densities on hydrogen production in the PEM water electrolysis process, utilizing the Cu_2O/C catalyst. This study investigates the impact of catalyst loading and different current densities on hydrogen production in the PEM water electrolysis process, utilizing the Cu_2O/C catalyst. The electrode catalytic properties were evaluated using the Cyclic Voltammetry (CV) method to determine the Electrochemical Surface Area (ECSA) and the Electrochemical Impedance Spectroscopy (EIS) method to determine the electrical conductivity. The ECSA and EIS measurements demonstrated that the best results were obtained at a higher catalyst loading of 2 mg/cm^2 with an ECSA value of 0.21 m^2/g and electrical conductivity of 3.04×10^{-6} S/cm. The production of hydrogen results showed that the highest hydrogen production rate was 3.75 mL/s with a catalyst loading of 2.5 mg/cm^2, indicating that increasing the load could lead to a higher rate of hydrogen gas production, but this is highly dependent on the surface area utilized. Additionally, at higher current densities, the cell resistance in the electrolysis process may decrease, leading to reduced electrode efficiency for hydrogen production. Thus, the use of high currents may not always be advantageous in hydrogen production using the PEM water electrolysis method.

Keywords
Cu_2O/C, PEMWE, Electrolysis, Hydrogen Production

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1. INTRODUCTION

The source of energy is currently undergoing a transition from the use of fossil fuels towards the use of hydrogen as an alternative energy source. Besides its depleting availability, environmental issues have also become a major concern. The use of hydrogen as a clean and versatile energy carrier is gaining more attention due to its potential to reduce greenhouse gas emissions and support the transition towards a more sustainable and low-carbon energy system (Kovač et al., 2021). Hydrogen is an ideal energy carrier for sustainable energy due to its high energy density, cleanliness, and low emissions (Xu et al., 2022), and it is a renewable energy source if the hydrogen is produced from water (Lai et al., 2015). One method for hydrogen production is Proton Exchange Membrane Water Electrolysis (PEMWE) (Russel, 2018). Hydrogen production using water electrolysis is very effective because it utilizes water and the by-products produced are oxygen (Carmo et al., 2019). The other method of water electrolysis is alkaline electrolysis, which still produces residues from the basic raw materials and generates hydrogen gas with low purity levels, the same goes for Microbial electrolysis (Kadier et al., 2016; Kumar and Himabindu, 2019). The advantages of PEMWE, include high cell efficiency, a greater rate of hydrogen production, high purity, and the possibility of further conversion into electricity. In PEMWE, water molecules will dissociate into oxygen and

hydrogen under the influence of an electric current (Kumar and Himabindu, 2019). The amount of current needed for mass transport, especially on the anode side where water will be transported to the catalyst layer. Besides the use of a catalyst also affects the rate of hydrogen production. The more catalysts used, the greater the hydrogen production rate. This is because many catalyst active sites participate in reacting on the surface of the electrodes, causing the electrochemical reaction to occur faster (Yu et al., 2021).

This study conducted water electrolysis experiments using Cu_2O as a catalyst and Vulcan carbon matrix as a support. Copper catalysts are often used as doping agents in Pt/C catalysts to enhance catalytic activity in PEMFCs. However, copper metal converted into the oxide form (Cu_2O) has been found to exhibit nearly comparable abilities to Pt/C catalysts in water electrolysis, while being more economical when utilized in hydrogen gas production on a pilot scale (Sun et al., 2022). Cu_2O, also known as copper oxide, is one of the three stable oxides of copper, where copper exhibits an oxidation state of +1. In addition to being used as an electrocatalyst in water electrolysis (Wang et al., 2023), this compound is also utilized as a catalyst for converting CO_2 through electrochemical reduction with satisfactory results (Bugayong, 2014), and (Rohendi et al., 2022). Furthermore, the advantages of the catalyst Cu_2O demonstrated superior catalytic activity compared to Cu nanoparticles due to its semiconductor properties with a band gap of 2.2 eV (Sasmal et al., 2016). The catalyst is advantageous in facilitating the redox reaction on H_2O, is non-toxic (Bagal et al., 2019), cost-effective, easy to mix with other polymers, and is chemically and physically stable (Rohendi et al., 2015). The interaction of Vulcan carbon as a catalyst support and active substance Cu_2O can increase the surface area and electronic conductivity when combined with metal oxides and has catalytic activity, selectivity, stability, and long service life (Maillard et al., 2005; Sun et al., 2022). The addition of Cu-based carbon is also used as an electrocatalyst for the hydrogen evolution reaction (HER) and oxygen reduction reactions (ORR). In this manuscript, The Cu_2O/C catalyst is utilized at both the anode and cathode and subsequently combined with the Nafion-117 membrane to form a membrane electrode assembly (MEA).

2. EXPERIMENTAL SECTION

2.1 Materials

Cu_2O Catalyst Powders (Sigma Aldrich), Carbon Vulcan XC-72R (Fuel Cell Store), Ammonium Bicarbonate (Sigma Aldrich), 2-Propanol (Sigma Aldrich), PTFE Emulsion 60%wt (Fuel Cell Store), Carbon Paper Avcarb P75T (Fuel Cell Store), Nafion-117 Membrane (Fuel Cell Store), Nafion Dispersion D2020 (Fuel Cell Store), H_2O_2 (Sigma Aldrich), And H_2SO_4 (Sigma Aldrich).

2.2 Methods

The water electrolysis was conducted using a specially built electrolyzer, initially designed as a Proton Exchange Membrane Fuel Cell (PEMFC) with an active area of 49 cm^2 and various Cu_2O/C catalyst loads ranging from 1, 1.5, 2, and 2.5 mg/cm^2 on both the cathode and anode. MEAs were prepared using the same method as in PEMFC, with catalyst electrodes hot-pressed on both sides of the Nafion-117 membrane. The electrodes were fabricated using a catalyst mixture of Copper Oxide, and Vulcan XC-72R carbon at a ratio of 40:60 between Cu_2O 2 g and Carbon 3 g. This catalyst was mixed using the mechanical alloying method with high-energy milling (HEM) Shaker E3D for 3 hours with an active and rest time of 3:1 minute. Methanol was added as a process control agent (PCA) to prevent cold welding from occurring on the walls of the HEM Shaker jar. The catalyst was then mixed with Nafion Dispersion D2020, Polytetrafluoroethylene (PTFE) Solution, and 2-propanol. The Cu_2O/C ink was sprayed onto the gas diffusion layer (GDL) using a spray gun with alternating horizontal and vertical movements, followed by sintering at 350° or 3 hours, allowing PTFE impregnation (Lee et al., 2013). The electrodes were characterized using the Cyclic Voltammetry (CV) method to determine ECSA, while their electrical conductivity properties were analyzed using the EIS method. The active surface area of the electrode with Cu_2O/C catalyst was quantified using the CV technique, involving the measurement of the voltage response of each cell current simultaneously. The ECSA value was determined from the voltammogram. For this, Cu_2O/C electrodes were used as the working electrode, Pt electrodes as the counter electrode, and Ag/AgCl electrodes as the reference electrode. CV measurements were conducted in a 1 M NaOH electrolyte solution from -1 to 1 V to identify the active area of the catalyst on the electrode (Rohendi et al., 2023).

The production of hydrogen from water electrolysis using PEM can be described as shown in Figure 1.

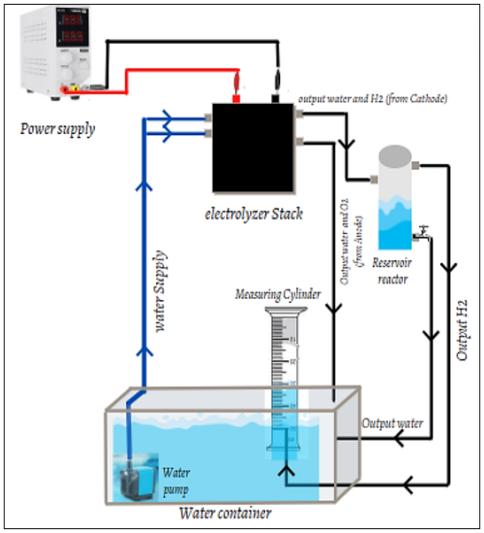

Figure 1. Experimental Setup of Hydrogen Production with PEMWE Methods

Water supplied through the anode and cathode chambers

undergo reduction and oxidation reactions as in Equation (1).

$$\begin{aligned}&\text{Anode:}\quad H_2O(l) \rightarrow 2H^+ + \frac{1}{2}O_2(g) + 2e^-\\&\text{Cathode:}\quad 2H^+ + 2e^- \rightarrow H_2(g)\\&\text{Overall Reaction:}\quad H_2O(l) \rightarrow H_2(g) + \frac{1}{2}O_2(g)\end{aligned} \qquad (1)$$

The hydrogen gas and water exit through the output channel to the reservoir reactor. There is a separation between water and hydrogen gas with the water remaining in the reservoir reactor, while the hydrogen gas exit through the channel leading to a measuring cup filled with water. Therefore, the H_2 gas will displace the water, and the reduced volume is calculated as the volume of hydrogen gas produced.

3. RESULTS AND DISCUSSION

3.1 Determination of ECSA Value Using the CV Methods

This analysis was performed to produce a voltammogram curve reflecting the presence of an oxidation-reduction reaction. The values extracted from the curve were then utilized to determine the ECSA value (Rohendi et al., 2015). The voltammogram curve indicates the occurrence of redox reactions on electrodes containing Cu_2O/C catalysts, as demonstrated by the formation of an anodic peak on the upper curve signifying an oxidation reaction, and a cathodic peak on the lower curve indicating a reduction reaction at the electrode (Sharma et al., 2022). Figure 2 shows the voltammogram curve for electrodes containing Cu_2O/C catalyst with various loadings of 1, 1.5, 2, and 2.5 mg/cm^2.

The presence of cathodic and anodic peaks on the voltammogram curve indicates that electrodes with Cu_2O/C catalyst undergo reduction and oxidation reactions, as shown in Figure 2. Furthermore, the ECSA results provide insight into the effective charge transfer resistance of an electrode, where a larger ECSA value implies lower resistance (Xue et al., 2022). This resistance can impact the proton conductivity of the MEA. Table 1 shows the results of the ECSA calculations for the four electrodes with Cu_2O/C catalyst at varying loadings using a common Equation (Eris et al., 2018; Yıldız et al., 2016; Juodkazytė et al., 2013).

Table 1. The ECSA Values for Electrodes with Variation in Catalyst Loading

Catalyst Loading (mg/cm^2)	ECSA (m^2/g)
1	0.040
1.5	0.097
2	0.210
2.5	0.090

Based on the calculation results, the Cu_2O/C catalyst with a loading of 2 mg/cm^2 demonstrated the highest ECSA value of 0.21 m^2/g. This suggests that at this loading, there are numerous active sites on the electrodes, leading to faster electrochemical reactions. From a kinetics perspective, lowering the loading reduces the reduction in intrinsic activity, resulting in higher actual intrinsic activity. This is because an increase in loading results in the accumulation of catalysts, increasing the distance of electron transport through the electrode. As the charge increases, the total charge transferred to the electrodes also increases, requiring faster ion diffusion into the electrolyte (Yu et al., 2021). Additionally, the use of carbon as catalyst support contributes to good catalyst dispersion in the kinetic control region and improves mass transport in both affected regions and mass transport regions, leading to an increase in ECSA value (Saadi et al., 2022).

An electrode with a catalyst loading of 2.5 mg/cm^2 exhibits a reduction in the area on the voltammogram curve, resulting in a lower ECSA value of 0.09 m^2/g. This is attributed to the possibility of accumulation of Cu_2O/C catalyst on the electrode, which reduces the availability of active sites for the reaction. Moreover, a high loading of catalyst can lead to inactive catalyst surfaces, particularly in regions far from the electrolyte interface, as not all surfaces can be wetted by the electrolyte. The loading of the catalyst also affects electron and ion transport through the electrodes, thereby impacting the reaction kinetics (Yu et al., 2021). Furthermore, with an increase in the electrode layer, the charge transfer coefficient will decrease proportionally, which is directly related to the amount of loading used (Xue et al., 2022; Soderberg et al., 2006).

3.2 Determination of Electrical Conductivity Value by EIS Methods

The purpose of measuring the electrical conductivity value of the Cu_2O/C electrode is to assess its conductivity capacity. The measurement yields real impedance values (Z') and imaginary impedance (Z"), which are plotted on a Nyquist Plot using NOVA software. The X-axis represents the real impedance, while the Y-axis represents the imaginary impedance. Subsequently, the Nyquist Plot is analyzed to determine the desired equivalent circuit. The results of the EIS measurement for Cu_2O/C electrodes at catalyst loadings of 1, 1.5, 2, and 2.5 mg/cm^2 are shown on the Nyquist curve in Figure 3, and the results of EIS measurements and the conductivity values of the electrodes are shown in Table 2. Calculation of the electrical conductivity of the electrode follows Zhang et al. (2022) and Wang et al. (2017).

The conductivity values are in the range of 10^{-6}-10^{-5} S/cm. A material whose conductivity value is in the range of 10^{-6} to 10^{2} S/cm is classified as a semiconducting material (Sharaf, 2020). In the measurement, the highest electrical conductivity value was obtained for the electrode with Cu_2O/C catalyst at a loading of 2 mg/cm^2 of 3.04×10^{-6} S/cm. This indicates that the electrode with Cu_2O/C catalyst at a loading of 2 mg/cm^2 can conduct electricity better than other loadings because this value can facilitate the occurrence of redox reactions,

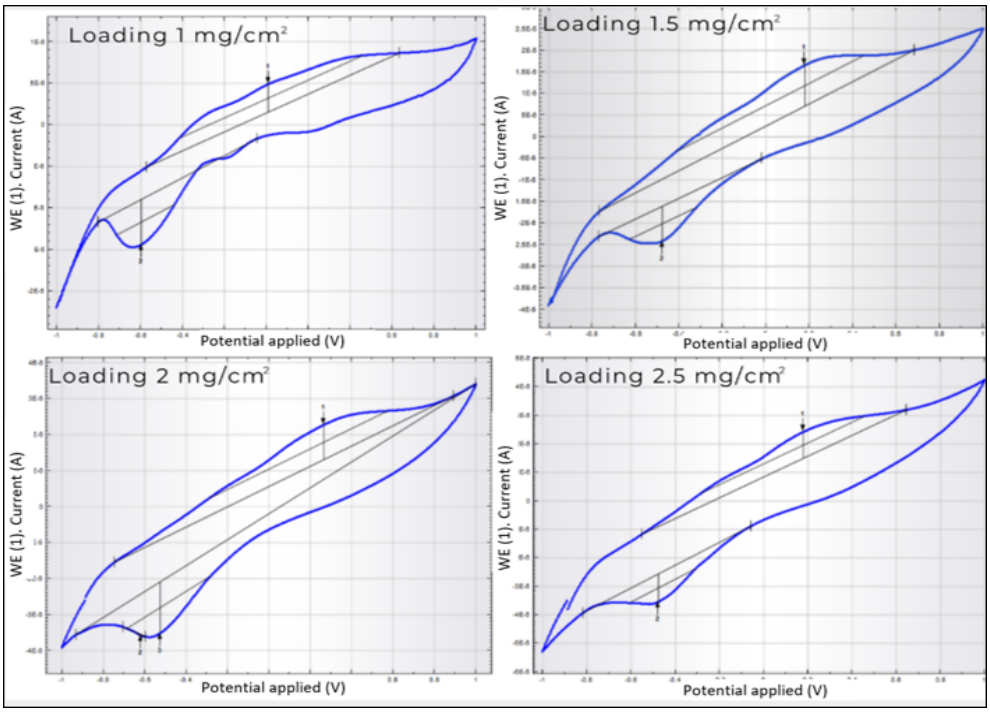

Figure 2. Voltammogram of Cu_2O/C Electrode with Various Catalyst Loading

Table 2. Data from Fitting the Nyquist Curve and Conductivity Values for Electrodes with Cu_2O/C Catalyst

Catalyst Loading (mg/cm^2)	Impedance Parameters Rs (Ω)	Rp (Ω)	Electrical Conductivity (S/cm)
1	101.290	3406.8	1.910^{-5}
1.5	68.724	2855.6	2.2810^{-6}
2	83.072	2108.5	3.0410^{-6}
2.5	80.916	2208.1	2.9110^{-6}

by flowing current to dissociate H_2O molecules into H_2 and O_2 (Lopata et al., 2020). The catalyst loading and resistance for charge transfer (RCT) are inversely related (Saadi et al., 2022). At a catalyst loading of 2.5 mg/cm^2, the EIS measurement value decreases because high loading and a thick catalyst layer will increase the HFR (high-frequency resistance) due to the electrical resistance of the layer seen in the SEM test in Figure 4 shows that the electrode with a loading of 2.5 mg/cm^2 has a defective structure and cracks on the surface this will encourage nucleation (not free bubble growth) resulting in easier removal of oxygen bubbles from the catalyst layer (Smolinka, 2009).

3.3 Determination of Electrocatalyst Surface by Scanning Electron Microscope and Energy Dispersive X-Ray (SEM-EDX)

The result of SEM-EDX characterization shows the morphology and elemental analysis of the electrocatalyst loading 2.5 mg/cm^2. This characterization was carried out after carrying out the EIS measurement because at a loading of 2.5 mg/cm^2, the conductivity value decreased so it was decided to carry out SEM characterization, and the results were obtained in Figure 4.

In the morphology of the Cu_2O/C electrode surface at magnifications of 5000× and 10000×, white granules, which represent Cu atoms, are visible, while at a magnification of 500×, cracks are visible, marked by lines. This is caused by the catalytic electrode manufacturing process, which involves distribution on the GDL and sintering using a furnace at a temperature of 350°C for 3 hours. The heating affects the electrode surface due to significant differences in thermal expansion between various parts of the electrode, resulting in stress within the distributed catalyst structure, ultimately leading to cracks on the electrode surface (Zhang et al., 2020). Additionally, extreme operational conditions in the furnace, such as rapid heating and cooling cycles, can also increase the risk of crack formation on the electrode (Li et al., 2023).

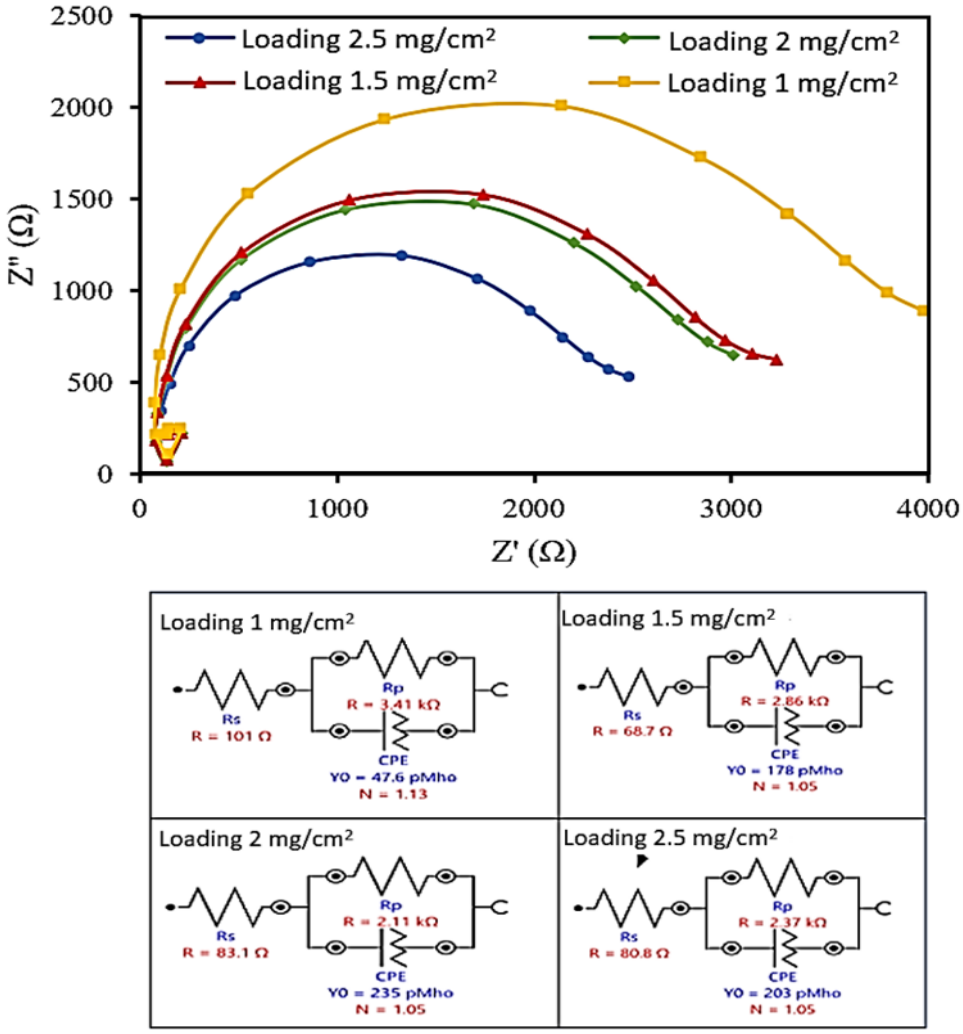

Figure 3. Nyquist Plot and Circuit Model of Electrodes with Cu_2O/C Catalyst at Various Catalyst Loadings

3.4 Hydrogen Production Using the PEM Water Electrolysis Method at Various Current and Catalyst Loading

The production of hydrogen by water electrolysis occurs throu -gh a water-splitting process that involves two half-reactions, the hydrogen evolution reaction (HER) at the cathode, and the oxygen evolution reaction (OER) at the anode. Furthermore, both reactions are very important for the overall water-splitting efficiency (Tang et al., 2020). The HER mechanism begins with the adsorption of hydrogen ions on the surface of the catalyst, known as the Volmer reaction. The continuous adsorption reaction of hydrogen ions from the electrolyte forms hydrogen molecules (Yuan et al., 2023; Singh and Ahuja, 2021). In this study, the current variation was implemented to facilitate charge transfer. The flow of electrons through the electrode results in electron transfer to other electrodes, thus facilitating redox reactions (Zahoor et al., 2023). The oxidation of H_2O at the anode produces H^+ ions, which migrate to the cathode and form H_2 gas, as described in Equation (1). The measurements with varying catalyst loadings were conducted starting from 1, 1.5, 2, and 2.5 mg/cm^2. The obtained data regarding the hydrogen production rate using Cu_2O/C catalyst at different loadings are presented in Figure 5.

The load of catalyst used has an impact on the rate of hydrogen production (Nouri-Khorasani et al., 2017). The data indicates that the optimal catalyst loading for the Cu_2O/C catalyst on the electrode is 2.5 mg/cm^2, resulting in a hydrogen production rate of 3.75 mL/s. The lowest hydrogen production rate was observed at a catalyst loading of 1 mg/cm^2, which yielded only 0.72 mL/s.

Catalyst loading is directly proportional to the surface area. When the surface area is large, the loading of the catalyst required also increases and therefore there are more active sites on the catalyst and the ability of the electrode to split water into protons and oxygen is also getting better (Lopata et al., 2020). However, when the catalyst loading is too high on a small surface area this can cause agglomeration (clumping),

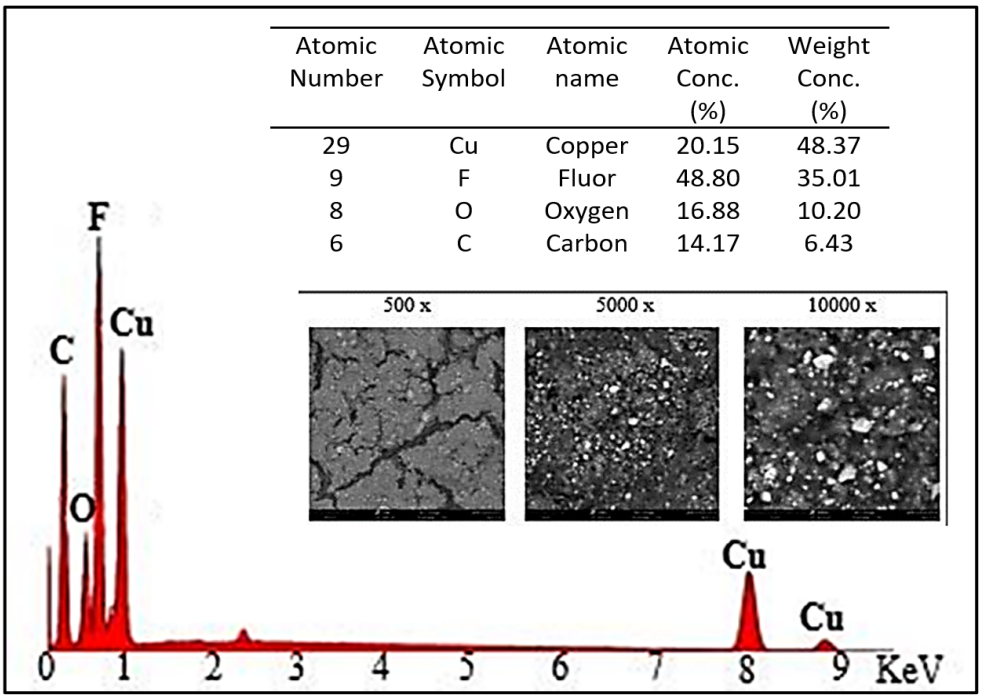

Figure 4. SEM-EDX Analysis of Electrode with Cu_2O/C Catalyst

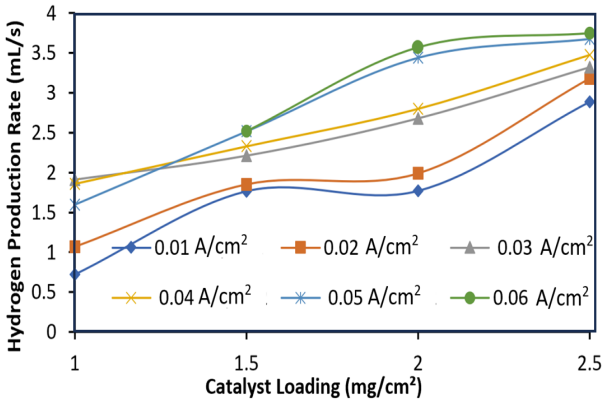

Figure 5. The Hydrogen Production Rate by Catalyst Loading Varies on the Electrode with a Cu_2O/C Catalyst

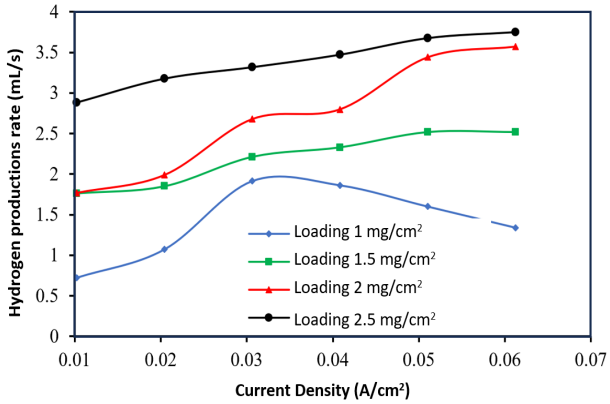

Figure 6. The Hydrogen Production Rate with Varying Current Densities at Electrodes with Cu_2O/C Catalyst

which can reduce the active site of the catalyst particles. This can reduce the performance of the catalyst and slow down the rate of hydrogen production (Maillard et al., 2005).

The hydrogen production rate was measured at various currents (in the form of current density), ranging from 0.5 A to 3 A (0.01, 0.02, 0.03, 0.04, and 0.05 A/cm^2). The choice to convert current (A) to current density (A/cm^2) is to determine the rate of hydrogen production accurately. The data obtained from the measurements of the rate of hydrogen production using a Cu_2O/C catalyst at various current densities are presented in Figure 6.

Various streams of hydrogen production have been carried out and the results obtained the optimum current for the hydrogen production rate is obtained with a current density of 0.06 A/cm^2. at low current density, the formation rate of H_2 is also low because it diffuses into the bulk electrolyte before reaching the concentration threshold for bubble nucleation (Porciúncula et al., 2012). The current use follows or is almost the same as the research conducted by previous research (Bhandari et al., 2014; Acar and Dincer, 2018). Based on Faraday's Law, the rate of hydrogen production from water electrolysis is proportional to the charge transferred, that is the greater the current used, the faster the rate of hydrogen production (Maric and Yu, 2019; Buelvas et al., 2014).

At a catalyst loading of 1 mg/cm^2, optimum hydrogen production occurs at a current density of 0.03 A/cm^2 with a hydrogen production rate of 1.91 mL/s and decreases at a current density of 0.04 - 0.06 A/cm^2. This is because, at a loading of 1 mg/cm^2, it has the lowest ECSA value so it is unable to facilitate a higher current density. The higher current density will result in a decrease in cell resistance in the electrolysis process (Buelvas et al., 2014). This decrease in the cell causes the conductivity properties of the electrode to increase where the conductivity properties are directly proportional to the size of the H_2 bubbles formed. This does not have a good impact on the rate of hydrogen production because, at a high current density, the reaction for the formation of H_2 bubbles runs faster than the transfer of bubbles from the surface of the catalyst to the bulk electrolyte which causes gas accumulation. If the H_2 bubble size increases, the H_2 gas bubble evolves from the electrode surface to the bulk electrolyte more slowly and will reach a saturated concentration for bubble formation H_2 (Porciúncula et al., 2012; Majlan et al., 2018).

4. CONCLUSIONS

The hydrogen production using various catalyst loadings has indicated that the highest production rate was achieved with a catalyst loading of 2.5 mg/cm^2. The rate of production of hydrogen increased as more load of catalyst but was found strongly influenced by the surface area utilized. The catalyst loading is directly proportional to the surface area, and with a large surface area, the required catalyst loading also increases. Therefore, there are more active sites on the catalyst, which improves the electrode's ability to split water into protons and oxygen furthermore, the effect of current on hydrogen production, at a higher current density can also cause a decrease in the resistance of the cell in the electrolysis process, and therefore the performance efficiency of an electrode for hydrogen production decreases. Meanwhile, the results of ECSA and EIS measurements showed a higher catalyst loading of 2 mg/cm^2. At a catalyst loading of 2.5 mg/cm^2 the EIS measurement value decreases because at high loading, a thick catalyst layer will increase the HFR (high-frequency resistance) due to the electrical resistance of the layer.

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