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Modification of Carbon Felt With Carbon Nanotubes as Electrodes for Microbial Fuel Cells

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ABSTRACT

Microbial Fuel Cell as an alternative energy device that produces electrical energy. Microbial Fuel Cell is a device that can convert energy in organic matter into electrical energy using microorganisms that carry out metabolism as a catalyst. One of the electrodes, the anode, plays an important role as an electron acceptor in a microbial fuel cell. The anode used can be improved the resulting performance by modifying it using other materials. Therefore, in this study, variations of the anode of Carbon Felt modified by Carbon Nanotubes were used in the Single Chamber Microbial Fuel Cell. From the CF, CF/CNT1, CF/CNT2 and CF/CNT3 anodes used in this study, data on the CF/CNT2 anode showed the most optimal among other anodes with a sheet resistivity value of 14677.5 ± 2538.69 Ω/sq, the average voltage is 0.1228 V, the value of Maximum Power Density (DTM) is 42.45 mW/m2 at an average current density of 246.21 mA/m2. And the activity of microorganisms achieved by the CF/CNT2 anode is that it can oxidize glucose up to 96.13% and produce a biofilm mass of 0.255 ± 0.007 g. Then the concentration of CNT added to the CF anode will affect the electrical energy produced.

KEYWORDS Voltage; Resistivity; Current Density; Energy Density; Biofilm

INTRODUCTION

Global energy demand is increasing, and with the accompanying urgency of reducing carbon emissions, the primary focus is on providing renewable energy sources and improving energy efficiency (Abbassi & Yadav, 2020; Afriani, 2012; Agustining, 2012). To address this challenge, significant efforts are being made to develop clean energy technologies (Yu et al., 2012). Several existing renewable energy generation systems include wind turbines, photovoltaics, and fuel cells (Bhukya et al., 2019; Chen et al., 2011).

One type of fuel cell that has been commercialized is the Microbial Fuel Cell (MFC), which is a device or system that utilizes exoelectrogenic bacteria as biocatalysts to convert chemical energy from a substrate into electrical energy (Ibrahim et al., 2019). MFCs are composed of an anode and a cathode (Roy et al., 2017; Gajda et al., 2020). Microorganisms living at the anode act as biocatalysts that oxidize organic matter in liquid waste to produce protons, electrons, and carbon dioxide gas (Paul et al., 2017; Wang et al., 2019; Liu et al., 2020). The resulting protons move to the cathode through the liquid waste medium, while electrons move from the anode to the cathode through the MFC's external circuit, thereby generating electrical energy. Biofilms formed on the electrodes in MFCs indicate the activity of electricity-producing bacteria (Frank & Nevin, 2010).

MFC technology offers advantages as an alternative source of electrical energy generation, utilizing various substrates such as glucose, wastewater, and sewage sludge (Smith et al., 2015; Minke et al., 2017). However, MFCs have weaknesses, including low electrical energy output and relatively high costs (Manickam et al., 2012). Therefore, efforts to develop MFCs focus on reducing costs while increasing power generation through modifications and testing of various electrodes, membranes, and reactor designs. Electrode modification is one of the most commonly applied approaches. For example, Liu et al. (2020) used a dual-chamber MFC to measure electrical energy and microbial activity, employing several carbon felt anodes modified with amino-Fe_3O_4. The results showed an increase in electrical energy and greater microbial activity on the modified anode. Zhao et al. (2017) also tested carbon felt anodes modified with HNO_3 and H_2O_2, and the results demonstrated an increase in MFC power output. Hidalgo et al. (2016) further modified carbon felt anodes with HNO_3 and PANI in a dual-chamber MFC, and the results indicated an increase in the electrical energy produced.

Carbon Felt (CF) and Carbon Nanotubes (CNTs) are electrode materials of choice. Carbon felt electrodes are generally made from polyacrylonitrile (PAN) fibers (Minke et al., 2017) and are widely used as electrodes due to their good electrical conductivity, large surface area, high porosity, and relatively low cost (Huong Le et al., 2017). CF is commonly used in wastewater treatment applications (Tan et al., 2013). Meanwhile, CNTs are excellent electrical conductors and are commonly used in battery applications.

The urgency of this research is underscored by the need to develop high-performance, cost-effective MFC components. Carbon felt is already a popular electrode material due to its good conductivity, large surface area, high porosity, and relatively low cost (Huong Le et al., 2017). By enhancing its properties with a small amount of highly conductive CNTs, it is possible to achieve a significant improvement in performance without a prohibitive increase in material cost. Understanding the optimal concentration for this modification is crucial for creating efficient and scalable anodes, thereby accelerating the practical deployment of MFC technology for applications such as wastewater treatment and remote power generation.

Therefore, in this study, CF electrodes for yeast MFCs were modified by adding CNTs. Variations in the concentration of CNTs added to CF are expected to affect electrode performance, potentially resulting in maximum electrical energy output.

The research questions in this study are: (1) How does the concentration of CNT added to the CF electrode affect the electrical energy generated in the Single-Chamber Microbial Fuel Cell? (2) How does the concentration of CNT affect the biofilm formed on the anode surface?

The objectives of this research are: (1) To determine the effect of CNT concentration added to the CF electrode on the electrical energy generated in the Single-Chamber Microbial Fuel Cell. (2) To determine the effect of CNT concentration on biofilm formation on the anode surface.

METHOD

Tools and Materials

Experimental Toolkit

The following is a table of tools and their specifications in this study:

Table 1. Table of Tools and Their Specifications

Tool Name	Specification
Reaktor Single Chamber Microbial Fuel Cell	*Custom*, acrylic (28 ml volume) Silicone gasket

Membrane	Nafion 117 Dupon
Resistor	Resistor Cast RS-50
Multimeter	Multimeter UNI-T UT61-E
Magnetic Stirer	Magnetic Bar Merk vitlab
Oven	Denpoo Deos 820 38 liter
GlucoDr	allmedicus

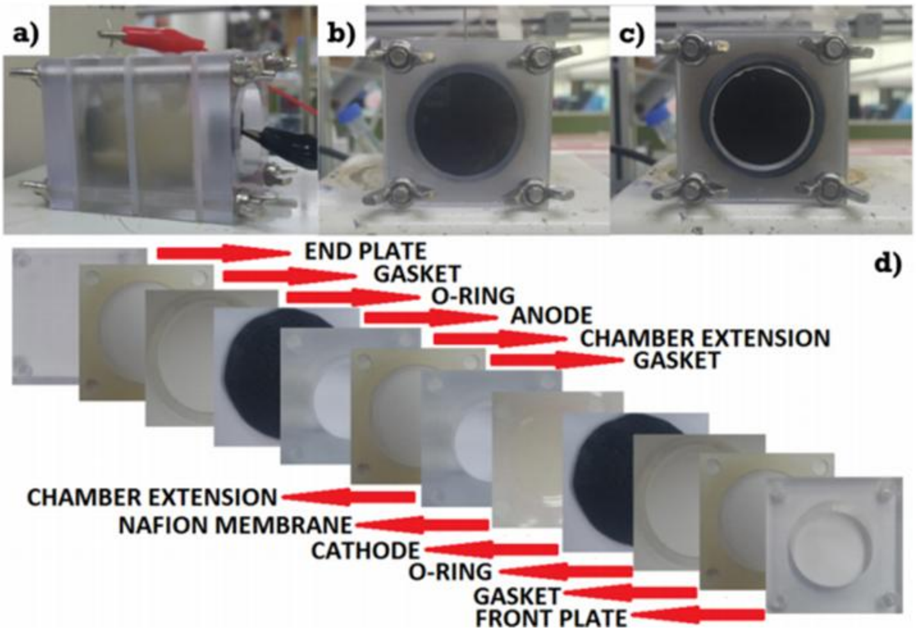

Figure 1. (a) Side view (b) anode side (c) cathode side (d) whole part view (Christwardana, et al., 2019)

Ingredients

The list of materials used in this study can be seen in Table 3.2.

Table 2. List of Ingredients

Ingredients	Specification
D-Glucose	KGaA, Serial CAS-No: 127-09-3.
Carbon Nanotube	Maxlab, Serial No: GE308068566
Carbon Felt	KWK Steel Co.Ltd., China
Aquadest	Aquadest laboratory of the Indonesian Institute of Technology.
Yeast Extract Granulated	KGaA, CAS-No: 8013-01-2, Darmstadt, Germany.
Saccharomyces cerevisiae	S.I.L France
Peptone	HiMedia Laboratories Pvt. Ltd, Nashik, India.
NaOH	MERCK 1.06498. 1000

Research Variables

Independent Variables

1. CNT Concentration : 0% wt solution, 2% wt solution, 4% wt solution, 6% wt solution.

Bound Variables

The variables bound in this study are *resistivity* values, voltage, maximum power density, glucose levels, and biofilm weight.

Fixed Variables

1. Room temperature 24 – 30 °C
2. Incubation time of 48 hours for 1 cycle, total 3 cycles or 144 hours
3. Electrolyte volume at anode 28 mL
4. Media concentration (→ medium YPD substrate)

Experimental Design

This research includes the incubation stage of MFC, MFC blank test on various concentrations of Carbon Nanotubes in the anode. Here is a flowchart from the research:

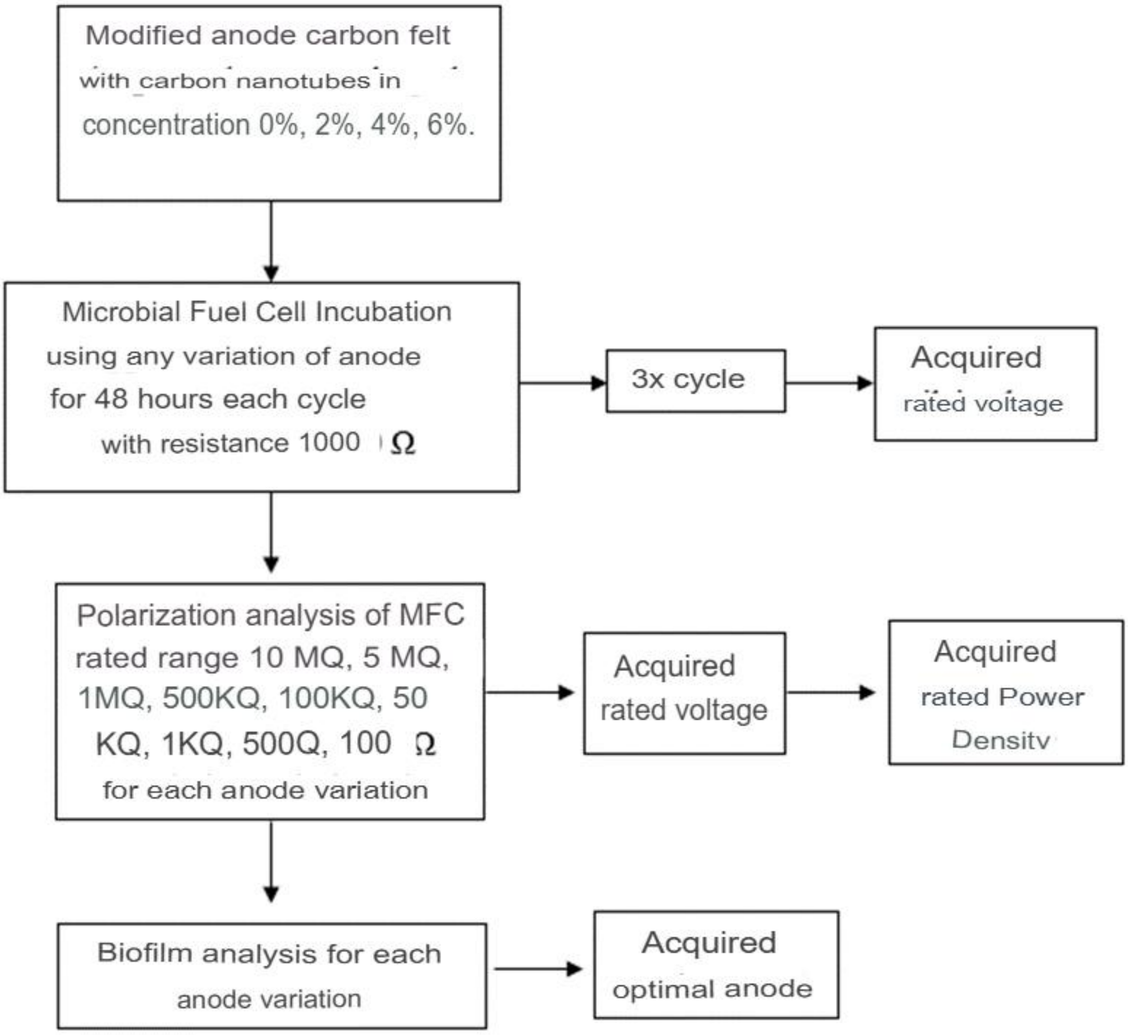

Figure 2. Research Work Diagram

Modifikasi

Carbon Felt is cut to a diameter of 3 cm Before entering the electrode incubation in *Treatment* first soaked in 500 ml of NaOH 1M solution with a temperature of 80 oC for 1 hour after which the electrodes are dried with an oven for ± 2 hours at 80oC. After drying, the anode is modified with *Carbon Nanotube*. The method is to put the cut CF into a beaker glass with a concentration *Carbon Nanotube* which are different are 0%, 2%, 4%, 6%. Soaking for 30 minutes. After that the CF is dried with the help of an oven for 3 hours at a temperature of 70 ^{oC} (Huong Le, et al., 2017).

MFC Incubation

This MFC incubation method uses a set of Single Chamber Microbial Fuel Cell tools made of archipelago with two electrodes with a diameter of 3 cm. Then a medium for microbes, namely YPD medium, is included. The ingredients are in the form of granulated yeast extract used as much as 5 mg/ml, D-Glucose used as much as 14 mg/ml, peptone as microbial nutrition as much as 2.5 mg/ml. The microbes used are *Saccharomyces cereviseae* from bread yeast as much as 14 mg/ml. Basis for microbial selection *Saccharomyces cerevisiae* is used because it is easy to obtain and breed. In addition, *Saccharomyces cerevisiae* is aerobic. The ingredients are dissolved with aquadest as much as 28 ml and put into the MFC. Then the MFC is incubated for approximately 1 week until the microbes enter the stationary phase which is characterized by a stable voltage. (Christwardana, et al., 2019)

Analysis

Voltage

The MFC incubation phase is carried out by connecting the MFC reactor with a multimeter as a recording device for the resulting voltage that was previously passed on the *external resistance* 1K Ω, the voltage will continue to be recorded every 15 minutes for up to 144 hours (48 hours per cycle). The voltage data generated after passing the resistance will be analyzed on each anode variation used, from the series of voltage data it is determined which anode sample can produce the highest voltage.

Polarization

The polarization curve is used to characterize current as a function of voltage. Changing the *external resistance* (load) of the circuit will obtain a new voltage and a new current at that resistance. Therefore, to obtain a polarization curve a different set of resistors on the circuit is used to measure the voltage at each resistance. MFCs that have passed the incubation period for 144 hours (48 hours per cycle) are then polarized tested without changing the MFC reactor composition, testing using *external resistance* starting from 10M Ω for 60 minutes, 5M Ω for 60 minutes, 1M Ω for 30 minutes, 500K Ω for 30 minutes, 100K Ω for minutes, 50K Ω for 30 minutes, 1K Ω for 15 minutes, 500 Ω for 15 minutes, and 100 Ω for 15 minutes.

Because the voltage is obtained at each *external resistance* that is changed, the power density can be obtained with the calculation below.

Calculating power density (P)

$$I = \frac{V}{R}$$

$$V = I \times R$$

$$P = \frac{V^2}{R}$$

Resisitivity

Resistivity analysis on the anode was carried out using the four probe method. The four probe method is a simple tool for measuring the resistivity of semiconductor samples. By passing current through two probes inserted into the sample to measure the voltage through the probes, it allows for the measurement of the substrate resistivity. The four probe method is carried out by assembling a simple tool as in Figure 3.3, the tool circuit consists of two 1.5 V batteries connected in series and then connected to the CF anode using a cable. After that, the two ends of the multimeter are attached to the top of the anode with a distance between the two sides of 1.1 cm (Hidalgo, et al., 2016). Testing is carried out every 5 minutes on each anode sample to obtain a voltage value that can be seen on the multimeter after which the voltage value obtained is calculated with the Four Point Probe Resistivity Calculator to obtain the resistivity value on each anode sample, the lower the resistivity value, the higher the conductivity of the anode sample (Wang, et al., 2019).

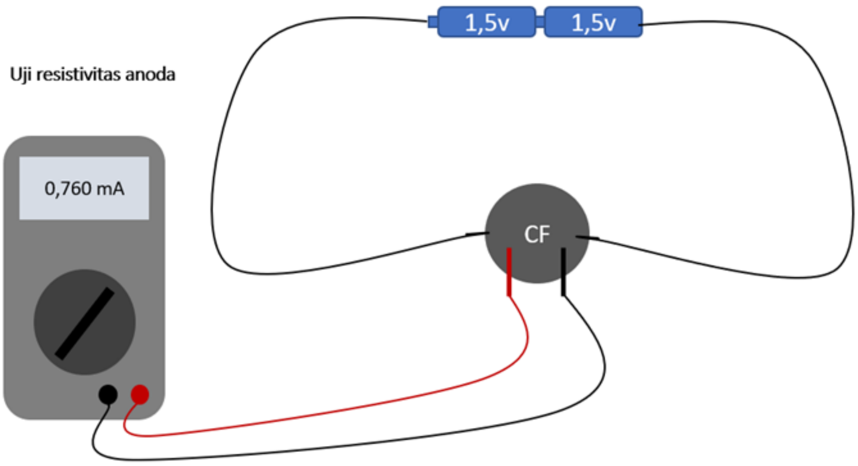

Figure 3. Anode resistivity testing

Biofilm

Biofilm measurements were conducted at room temperature for 144 hours. The initial mass was the anode mass before incubation, and the final mass was the anode mass after 144 hours of incubation. The difference between the two indicated the biofilm weight. The purpose of this test was to determine the amount of Saccharomyces cerevisiae microbial activity on each anode variation. The higher the microbial activity, the higher the voltage.

Glucose Levels

Glucose levels were measured at room temperature using a glucose tester. Each cycle, for 48 hours, measured the initial and final glucose levels. The difference between the two indicated a decrease in glucose levels. The purpose of this test was to determine the growth of Saccharomyces cerevisiae microbes, which is indicated by a decrease in glucose levels in the media.

RESULT AND DISCUSSION

Effect of Anode Variation on MFC Electrical Energy

Anode Resistance

This experiment was conducted after the CF was modified or treated with NaOH and partially with CNTs. The purpose of this test was to determine the resistivity values produced by each variation of the CF sample with CNTs.

Table 3. Anode resistivity test

Anode	Average		
	***Sheet resistivity* (Ω/sq)**	***Wafer resistivity* (Ω-cm)**	***Resistivity corrected for thickness* (Ω-cm)**
CF	30213,33 ± 9250,91	15106,67 ± 4625,45	14569,62 ± 4461,02
CF/CNT1	16995 ± 0	8497,5 ± 0	8195,41 ± 0
CF/CNT2	14677,5 ± 2538,69	7338,75 ± 1269,35	7077,86 ± 1224,22
CF/CNT3	12842,83 ± 5850,82	6421,39 ± 2925,34	6193,11 ± 2821,34

Table 1 shows that the addition of CNT will decrease the resistivity and increase the conductivity of the anode, seen from the sheet resistivity value of CF is 30213.33 ± 9250.91 Ω/sq, CF/CNT1 is 16995 ± 0 Ω/sq, CF/CNT2 is 14677.5 ± 2538.69 Ω/sq, and CF/CNT3 is 12842.83 ± 5850.82 Ω/sq. So the trend of resistivity decreases because the addition of CNT makes the CNT that coats the CF anode more and more will affect the performance of the MFC (Huong Le, et al., 2017). The characteristics of the electrode (anode) are the main factors in the effectiveness of the reaction, such as choosing an anode that has good conductivity and a large surface area, other things such as the anode as a material that must have good biocompatibility for yeast immobility on its surface are very necessary (Wang, et al., 2019). The data in Table 4.1 above shows that the higher the concentration of CNTs in CF, the smaller the resistivity value, which indicates that the resistance (resistance) of the anode will be smaller. Current density is the movement of electrons through a conductor in a circuit caused by voltage or tension to flow in the circuit (Patrick & Fardo, 2020), so with a smaller resistance makes it easier for the current density path so that the electrode will be easier to conduct electricity because the electrons are easier to move (Wang, et al., 2019). Related to Ohm's law, a small resistance value makes the electric current greater or more electricity flows at the anode. Haeney, (2004) said that the electrical resistivity of a material is a number that describes how much the material resists the flow of electricity. Resistivity is measured in ohm meters (Ω m). If electricity can flow easily through a material, the material has low resistivity. Meanwhile, if electricity has difficulty flowing through a material, the material has high resistivity. This means that high resistivity is the same as low conductivity, and low resistivity is the same as high conductivity.

Voltage

This experiment was conducted on an MFC incubation mass (3 cycles) using a fixed external resistance of 1kΩ. The voltage of each anode variable was measured every 15 minutes for 144 hours, as shown in Figure 4.1. This test aimed to determine the magnitude of the voltage generated by the various anodes used.

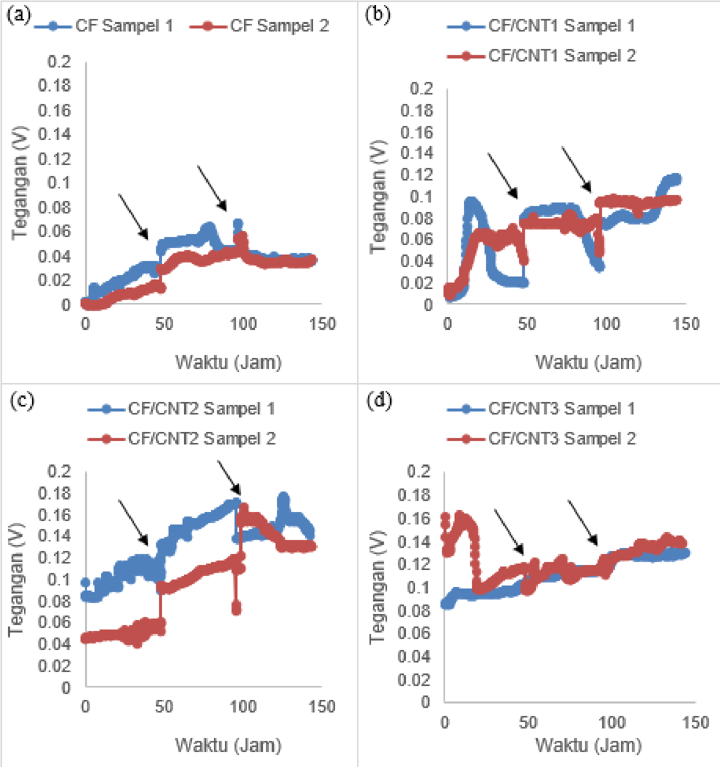

Figure 4. MFC voltage graph with anodes (a) CF (b) CF/CNT1 (c) CF/CNT2 (d) CF/CNT3 operated for 144 hours, the arrow indicates the media change time

Figure 4 above shows the electrical energy produced by MFC as proof that anodes with higher CNT content will produce higher voltages because the electric current produced is also greater (Patrick & Fardo, 2020). The voltage produced by the MFC reactor is closely related to the activity of Saccharomyces cerevisiae as a microbe that experiences a cycle of lag phase, growth phase, stationary phase, and death phase. A stable voltage graph indicates that the microbes in the media are experiencing a stationary-growth phase (Christwardana, et al., 2019). Looking at the average voltage in the stationary-growth phase or a stable graph, the voltage produced by the CF anode is 0.0340 V, CF/CNT1 is 0.0820 V, CF/CNT2 is 0.1228 V, and CF/CNT3 is 0.1250 V which are compared in Figure 4.2.

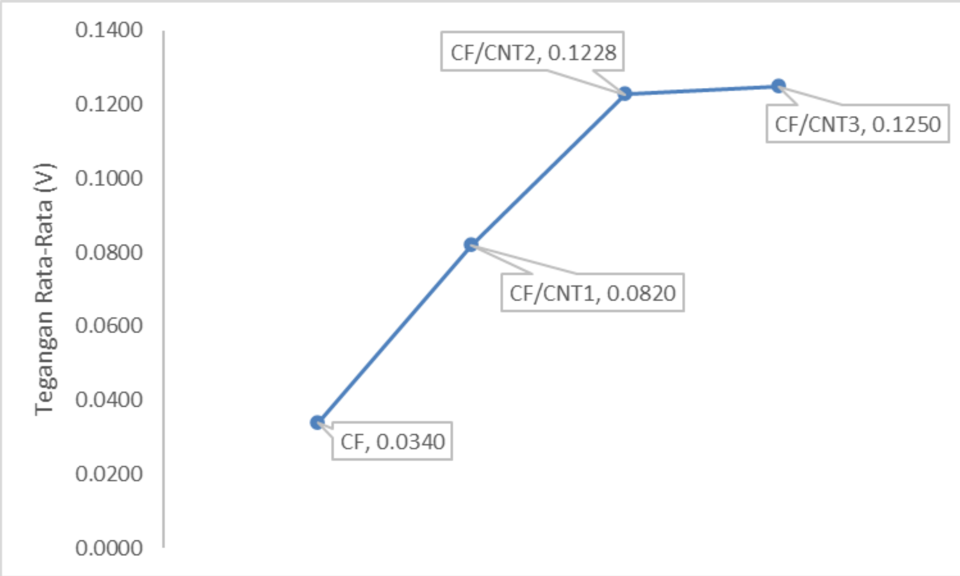

Figure 5. Comparison of the average voltage generated by each anode

Looking at the comparison of the average voltage of each anode in Figure 4.2, there is a significant increase in value from the CF anode to CF/CNT2, but from the CF/CNT2 anode to the CF/CNT3 anode, the increase is not significant. This can also be seen in the average voltage difference of CF:CF/CNT1 of 0.0481 V, CF/CNT1:CF/CNT2 of 0.0408 V, and CF/CNT2:CF/CNT3 of 0.0022 V. Analysis of the results obtained shows the effectiveness of adding CNTs to the CF anode only up to CF/CNT2, while the CF/CNT3 anode sample has already reached saturation of the CNT composition that can be added. This saturation is visible when many of the CNT particles fall off or refuse to adhere to the CF anode.

Polarization

The MFCs, which had undergone a 144-hour incubation period, were then subjected to polarization testing. This test was conducted to obtain the maximum voltage value by changing the external resistance, as shown in Figure 6

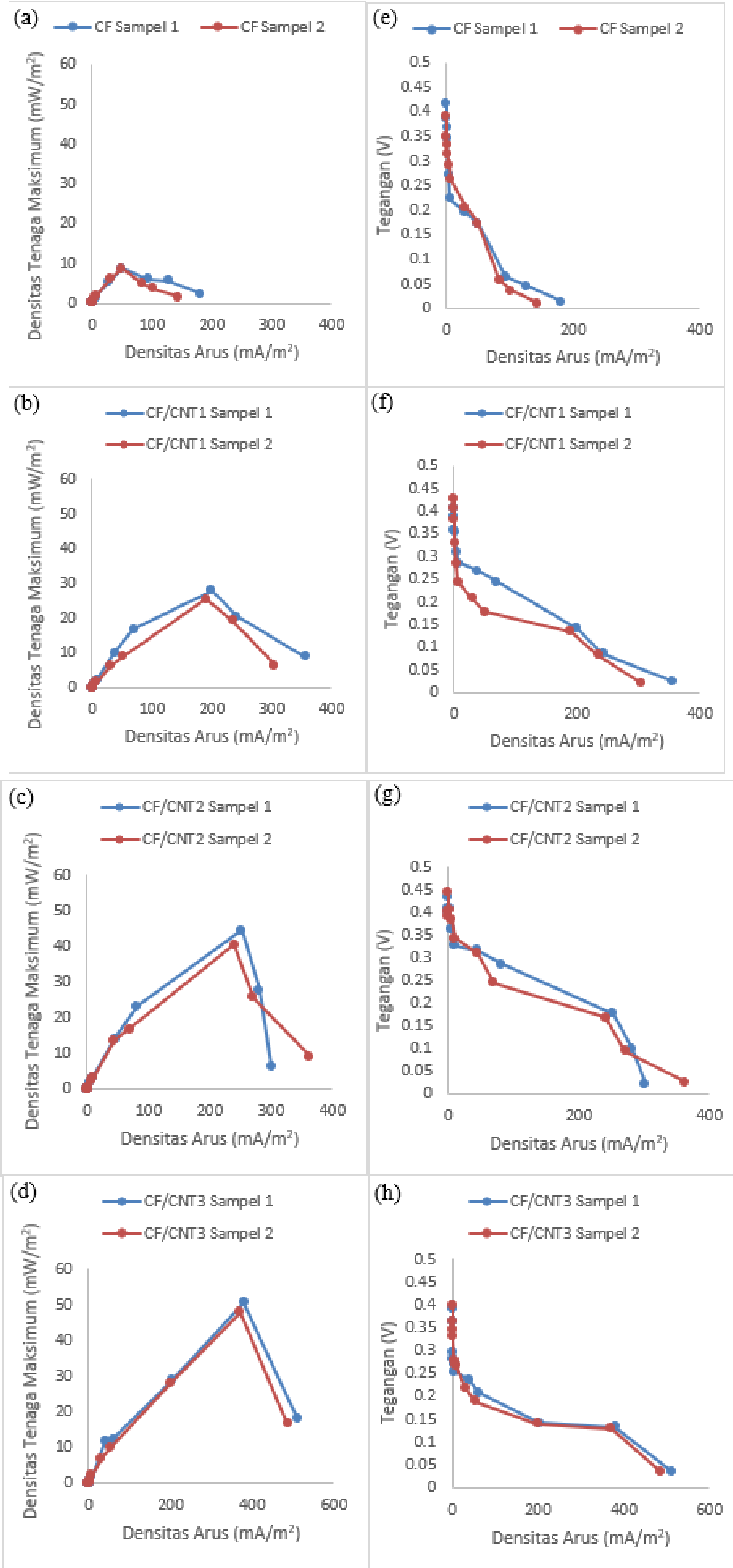

Figure 6. Polarization Curves of MFC (a) CF (b) CF/CNT1 (c) CF/CNT2 (d) CF/CNT3, while e) – (h) are their maximum power density curves

The voltage will decrease further if the resistance is lowered. Open Circuit Voltage (OCV) can be achieved under these conditions, and in Figure 4.3 (a) – (d) the highest average OCV value is obtained at the CF anode which is 0.4032 V, CF/CNT1 is 0.4162, CF/CNT2 is 0.4406, and CF/CNT3 is 0.3956 V. MFCs that use various power sources have the objective of maximizing output power or obtaining high current density. So it is seen from the possibility of a voltage drop that makes the current density increase to maximize the power density in that range.

Figure 4.3 (a) – (d) is used to calculate the data in Figure 4.3 (e) – (h). In the data obtained, the highest value on the power curve is used as the Maximum Power Density (MPD). At the CF anode, the MPD value is 8.60 mW/m2, CF/CNT1 is 26.71 mW/m2, CF/CNT2 is 42.45 mW/m2, and CF/CNT3 is 49.15 mW/m2; while the average current density value at the MPD value at the CF anode is 49.57 mA/m2, CF/CNT1 is 195.29 mA/m2, CF/CNT2 is 246.21 mA/m2, and CF/CNT3 is 374.71 mA/m2. So the analysis seen from the resistance value of the CF/CNT3 anode which is lower than the CF, CF/CNT1, and CF/CNT2 anodes makes the DTM value and current density achieved higher. The resulting DTM of CF/CNT3 is higher because it has a greater resistivity than other anodes, the smaller resistance of the anode opens the way for the current density to flow larger electrons so that the resulting DTM will be larger (Wang, et al., 2019).

Effect of CNT Concentration on Biofilms

Glucose Reduction

This experiment was conducted at room temperature, with each cycle lasting 48 hours. The purpose of this test was to determine the growth activity of the microbe Saccharomyces cerevisiae, as indicated by a decrease in glucose levels in the media, as shown in Table 4.2 and Figure 4.

Table 4. Measurement of Glucose Amount

Electrode	Siklus	Initial Glucose (mg/L)	Final Glucose (mg/L)	Decreased glucose levels %
1. CF	1	14000	1228.07	91.23%
	2	14049.12	1277.19	90.91%
	3	14049.12	1277.19	90.91%
2. CF	1	13950.88	1228.07	91.20%
	2	14000.00	1203.51	91.40%
	3	13950.88	1178.95	91.55%
1.CF/CNT1	1	13877.19	1203.51	91.33%
	2	13950.88	1228.07	91.20%
	3	14049.12	1203.51	91.43%
2. CF/CNT1	1	14049.12	1277.19	90.91%

	2	14000	1178.95	91.58%
	3	13877.19	687.72	95.04%
1. CF/CNT2	1	13950.88	564.91	95.95%
	2	13926.32	540.35	96.12%
	3	13950.88	564.91	95.95%
2. CF/CNT2	1	13877.19	589.47	95.75%
	2	13950.88	540.35	96.13%
	3	13926.32	564.91	95.94%
1. CF/CNT3	1	14049.12	515.79	96.33%
	2	14049.12	491.23	96.50%
	3	14049.12	491.23	96.50%
2. CF/CNT3	1	14000	491.23	96.49%
	2	14024.56	491.23	96.50%
	3	14049.12	491.23	96.50%

The decrease in glucose levels is due to the activity of the microbe Saccharomyces cerevisiea consuming glucose, resulting in glucose depletion because the fermentation-causing microorganism reacts with the organic substrate (glucose) that is suitable for its growth. Data on the decrease in glucose levels can be seen in table 4.2 above, glucose levels are low in each cycle indicating that the yeast is in the stationary phase. The stationary phase occurs when the yeast growth rate is equal to the death rate. So the total number of microbes will remain and begin to lack nutrients in the media, therefore it will be directly proportional to the decrease in tension because the number of living microbes begins to decrease (Riadi, 2016). CF as the main foundation of the anode only has a surface area of 0.0583 m2/g while CNT has a surface area of 900 m2/g, which when CNT as a material added to the CF anode causes the surface area to increase to 150 m2/g which is possible to be a place for better electron transfer than the CF anode alone (Huong Le, et al., 2017). The decrease in glucose levels in CF ranges from 90% to 91%, then a significant decrease in glucose levels begins to occur in CF/CNT1 by 91% to 95%, and CF/CNT2 by 95% to 96%, while in CF/CNT3 the decrease in glucose levels begins to be insignificant, which is stable at 96%. So CF/CNT2 is the optimal point because the surface area is larger than CF and CF/CNT1 so that yeast metabolism runs well, a decrease in glucose levels of 95% to 96%. In the CF/CNT3 table, the decrease in glucose levels began to stabilize at 96% and there was no significant increase, this indicates that the CF/CNT3 anode began to experience saturation point because the attached CNTs had begun to fill the entire surface area of the CF.

Biofilm Weight

This experiment was conducted at room temperature for 144 hours. The initial mass was the mass of the anode before incubation, and the final mass was the mass of the anode after 144 hours of incubation. The difference between the two indicated the weight of the biofilm. The purpose of this test was to determine the amount of Saccharomyces cerevisiae microbial activity on each anode variation.

Table 5. Biofilm Measurements on Anodes

Electrode	Initial Mass (g)	Final Mass (g)	Biofilm Mass (g)
CF	0,760 0,014±	0,965 0,007±	0,205 0,021±
CF/CNT 1	0,825 0,035±	1,055 0,021±	0,230 0,056±
CF/CNT 2	0,755 0,007±	1,010 0,000±	0,255 0,007±
CF/CNT 3	0,775 0,007±	1,040 0,014±	0,265 0,007±

The biofilm analysis data in the table that has been carried out on each anode sample, obtained the weight of the biofilm on the CF anode of 0.205g, CF / CNT1 of 0.23g, CF / CNT2 of 0.255g and CF / CNT3 of 0.265g which can be seen in table 4.3 above. The weight of the biofilm on the CF anode is lower than the CF / CNT anode because the CF anode is only composed of carbon felt so that the surface area is smaller than the CF / CNT anode which is composed of carbon felt that has been treated with a CNT solution, this CNT solution functions to make the surface area of the anode larger. The larger surface area on the CF / CNT has an impact on the number of biofilms becoming more and the resulting voltage will be greater. The heaviest biofilm on the CF / CNT3 indicates that the activity and metabolism of the Saccharomyces cerevisiae microbe are running well. The large amount of biofilm attached to the anode indicates that the number of Saccharomyces cerevisiae microbes is growing well, resulting in more electrons being produced, thus producing higher or greater electrical energy (Frank & Nevin, 2010). The large amount of biofilm also indicates that the CNT substance added to CF/CNT3 increases the surface area of the anode, making it a good place for the growth of Saccharomyces cerevisiae microbes.

CONCLUSION

On *Single Chamber Microbial Fuel Cell* with the large concentration of CNT added to the CF anode will affect the electrical energy produced. It is shown that the higher the CNT level will cause an increase in the voltage values, current density and power density generated by MFC. And the CF/CNT2 anode is the most optimal in this study. There is an increase in microbial activity that forms *a heavier biofilm* on the CF anode when the concentration of CNT in the CF anode is greater because the *surface area* or surface area is getting larger.

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