PENGARUH PENAMBAHAN LUMPUR TINJA DAN EM4 DALAM DUAL CHAMBER MICROBIAL FUEL CELL UNTUK MENGOLAH AIR LIMBAH INDUSTRI BATIK

THE EFFECT OF SEPTAGE SLUDGE AND EM4 ADDITION IN A DUAL-CHAMBER MICROBIAL FUEL CELL TREATING BATIK WASTEWATER

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Abstrak

Kementerian Perindustrian Indonesia menyatakan bahwa produksi kain batik pada tahun 2017 mencapai rata-rata 500 juta meter/tahun, yang setara dengan 25 juta m^3/tahun air. Dengan kebutuhan air yang besar dalam proses produksi, industri batik dapat menyebabkan pencemaran air pada badan air. Sistem Dual-Chamber Microbial Fuel Cell (DCMFC) dapat menurunkan kadar polutan organik dalam air limbah dan menghasilkan energi listrik secara bersamaan. Tujuan dari penelitian ini adalah untuk mempelajari pengaruh penambahan lumpur tinja dan konsorsium EM4 terhadap densitas daya yang dihasilkan oleh sistem DCMFC dan mempelajari pengaruh perbedaan pH pada sistem DCMFC terhadap penyisihan COD. Pengaruh variasi penambahan lumpur IPAL dan EM4 terhadap produksi energi listrik diperoleh hasil yang terlalu besar perbedaannya. Hasil ini disebabkan oleh beberapa faktor, antara lain arus listrik, tegangan listrik, pertumbuhan mikroorganisme, dan pH. Densitas daya maksimum pada variasi EM4 sebesar 297,61 mW/m^2, power density pada variasi lumpur tinja sebesar 287,26 mW/m^2, dan densitas daya reaktor kontrol sebesar 185,99 mW/m^2. pH optimal untuk lumpur tinja dan EM4 untuk mendegradasi COD adalah sekitar 7. Pada sistem DCMFC dengan EM4, nilai MLSS meningkat secara stabil dari 1500 mg/L hingga 1800 mg/L tanpa memperhatikan pH. Dalam sistem dengan lumpur tinja menunjukkan efek tergantung pH pada MLSS (dalam kisaran 1250-2400 mg / L), dengan pH 7 yang merugikan. Pembentukan biofilm pada anoda meningkat dari waktu ke waktu dalam sistem EM4 di semua kondisi pH, dengan pertumbuhan penting dalam sistem lumpur tinja yang diamati pada pH 6. Penyisihan COD lumpur tinja dan EM4 adalah yang tertinggi, masing-masing sebesar 33,83 dan 40,76%.

Keywords: Dual-chamber microbial fuel cell, EM4, Energi Listrik, Limbah batik, Lumpur tinja

Abstract

The Indonesian Ministry of Industry stated that the production of batik cloth in 2017 reached an average of 500 million meters/year, which is equivalent to 25 million m^3/year of water. With a large water demand in the production process, the batik industry can cause water pollution in waterbodies. The Dual-Chamber Microbial Fuel Cell (DCMFC) system can reduce organic pollutant levels in wastewater and produce electrical energy simultaneously. The purpose of this research was to study the effect of the addition of septage sludge and EM4 consortium on the power density produced by the DCMFC system and to study the effect of pH differences in the DCMFC system on the removal of COD. The effect of variations in the addition of sewage treatment plant sludge and EM4 on the production of electrical energy obtained results that are too large a difference. This result was attributed to several factors, including electric current, electric voltage, microorganism growth, and pH. The maximum power density in the EM4 variation was 297.61 mW/m^2, the power density in the septage sludge variation was 287,26 mW/m^2, and the control reactor power density was 185.99 mW/m^2. The optimal pH for septage sludge and EM4 to degrade COD is about 7. In the DCMFC systems with EM4, MLSS values increased steadily from 1500 mg/L to 1800 mg/L regardless of pH. In the systems with septage sludge showed pH-dependent effects on MLSS (in the range of 1250-2400 *mg/L), with pH 7 being detrimental. Biofilm formation on the anode increased over time in EM4 systems across all pH conditions, with notable growth in septage sludge systems observed at pH 6. The COD removal of the septage sludge and EM4 was highest at 33.83 and 40.76%, respectively.*

Keywords: *Dual-chamber microbial fuel cell, Batik wastewater,Eelectrical energy, EM4, Septage sludge*

1. INTRODUCTION

Data from the Ministry of Industry in 2017 show that batik production in Indonesia averages 500 million meters per year, with water use in the production process reaching 25 million m^3/year (Indrayani, 2018). However, the progress of the batik industry has also been accompanied by environmental problems. Current environmental problems come from the activities of the batik manufacturing process, which produces liquid waste. Liquid waste in the form of dyes produced by the rest of the dyeing material, the process of washing and rinsing the batik cloth. In addition, generally, batik industrial waste consists of the remaining mori, wax splashes, remaining dyeing water, wax residue, and rinse water (Apriyani, 2018).

Microbial fuel cells are a cheap and effective waste treatment method before wastewater is discharged into the water bodies. The Dual-Chamber Microbial Fuel Cell (DCMFC) is a system capable of forming or producing electrical energy from substrate oxidation reactions with the help of microorganisms and bacteria. The components in the DCMFC system consist of two chambers, namely the cathode and anode chambers, where the component on the anode is microorganisms/bacteria. Microorganisms perform metabolism under anaerobic conditions by breaking down glucose into protons (H^+), electrons (e^-) and carbon dioxide (CO_2). The electrons produced can be used as sources of electric current. Electrons flow to the cathode through the outer circuit, while protons diffuse through the salt bridge to the cathode (Sari et al., 2016). DCMFC has been used to treat batik industry waste by adding inoculum in the form of sludge, resulting in an organic (as chemically oxygen demand, COD) removal rate of 92% (Rezky, 2023). Furthermore, the addition of yeast to wastewater in Microalgae Microbial Fuel Cell (MMFC) systems has been used to degrade COD concentration. The yeast in the MMFC system is used as a catalyst to oxidize waste, which increases the rate of electron transfer. The voltage of 0.322 V was produced while COD decreased by 48.5% (Polontalo et al., 2021). In the other study, the addition of microbes from fecal sludge is proven to boost the production of power density (Prayogo et al., 2017). In addition to generating power density, the use of fecal sludge in Microbial Fuel Cell (MFC) can reduce BOD, COD, and TSS by 92.7%, 93.9%, and 98.6%, respectively (Akatah et al., 2019). The addition of EM4l consortium can produce a potential difference and current strength of 0.623 V and 0.325 mA (Kurniati et al., 2020).

This study analyzed how different amounts of sewage sludge and EM4, as well as different pH levels, affected the performance of a batch DCMFC reactor treating batik wastewater. The study focused on observing the growth of microorganisms, the degradation rate of organic matters (measured as COD), and the production of electric current, including the power density.

2. METHODS

DCMFC batch reactors have been used in this study. The reactor was equipped with a 10 cm single graphite carbon electrode in the anode chamber, as well as a copper electrode in the cathode chamber, while a KCl-based salt bridge was used to connect both chambers. 4 liters of batik industrial wastewater, as substrate, was placed in the anode chamber and 4 liters of $KMnO_4$ 0.2 M was placed in the cathode chamber.

The first stage of experiment started with the preliminary research to characterize batik wastewater and reactor system preparation.
The second stage of the experiment was carried out with the variation of septage sludge and EM4 added in the anode chamber. Then the third stage was carried out with pH setup differences in the anode chamber until the power density value tends to stabilize or decrease.

2.1. Preliminary Research

Before conducting the main research, preliminary research was done to determine the types of pollutants found in batik industry wastewater and prepare reactor needs as follows:

a. Sampling
The batik wastewater was obtained through grab sampling from a wastewater basin of a batik industry located near Surabaya. Initial characteristic analysist was carried out to determine the organic concentrations and pH, as mentioned in the regulation.

b. Biomass Preparation
At this stage, the seeding and acclimatization process of septage sludge and EM4 were carried out by introducing these with available anaerobic bacterial microorganisms in the batik wastewater. During these processes, COD measurements were taken until stationary COD depletion was obtained (a fluctuation rate of no more than 10%).

c. Salt Bridge Preparation
The reactor equipped with a salt bridge made by boiling 100 ml of distilled water, adding KCl and nutrient agar, stirring until homogeneous and thickened, and then placing it into a clear pipe with a diameter of 1 inch and length of 10 cm, connecting the two chambers of the DCMFC reactor (Rezky, 2023).

d. Electrode Preparation
Before using, the carbon electrodes were cleaned and activated by soaking in 1 M HCl followed with 1 M NaOH solution for 1 day. The copper electrode was cleaned of dirt or biofilm formed on the surface using a fine paper sanding. The electrodes were then stored in distilled water. Right before used, the electrodes were dried thoroughly and weighed.

2.2. Primary Research

The main research used 7 pairs of reactors made of plastic cubes with a volume of 4 liters for each chamber. The study was conducted for a total of 240 h. Measurement of electrical energy produced was carried out every 24 h to find the optimal time needed to achieve maximum power density. Measurements of COD were done every 48 h to determine the COD removal. Measurements of MLSS were carried out every 24 h to determine the biomass growth in the batik wastewater.

3. RESULTS AND DISCUSSION

3.1. Preliminary Research

Sampling of batik wastewater and septage sludge using the grab sampling method. After sampling wastewater from the batik industry, the characteristics of the wastewater were analyzed. From the results of the analysis of the initial characteristics of batik wastewater, the organic parameters analyzed still do not meet the quality standards that refer to Appendix XLII (textile industry wastewater) Regulation Number 5/2014 of the Minister of Environment Republic Indonesia. The result was presented in Table 1.

Table 1. Characteristics of Batik Wastewater

Parameters	Test Results	Quality Standards	Units
COD	10471.42	150	mg/L
BOD	2091.87	60	mg/L
TSS	704	50	mg/L
pH	8.46	6–9	

From the data listed in Table 1, the batik wastewater characteristics have a BOD/COD ratio of 0,2. The BOD/COD ratio can be used for biological processes because it is in the biodegradable range of 0.2–0.5 (Samudro & Mangkoedihardjo, 2010). However, the ability of batik wastewater for biological processes is weak compared to the biodegradable range. The septage sludge and EM4 used in this study were seeded and acclimatized first with the batik wastewater substrate. Seeding and acclimatization were carried out simultaneously under anaerobic conditions.

Table 2. Seeding and Acclimatization Composition

Description	Volume (ml)	Substrate Volume (L)	Co-substrate Volume (ml)
Septage Sludge	144	12	75
EM4	144	12	75

At the beginning of the seeding and

acclimatization stages, co-substrate in the form of molasses solution is dissolved to add the nutritional needs of microorganisms. During this process, COD was collected every day and gas checked as a sign that the microorganisms are active. The research proceeded to the main research after COD has remained constant over time. The result of COD measurement was shown in Figure 1.

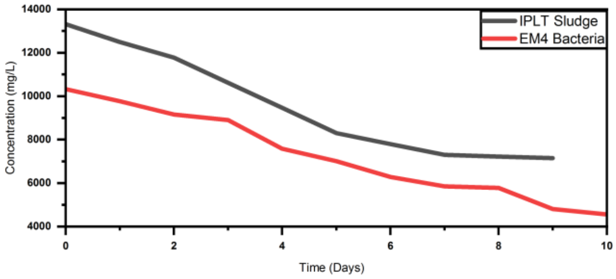

Figure 1. COD Measurement Results

Based on the measurement data of COD levels during the seeding and acclimatization stages of EM4 in batik wastewater, it was found that stationary conditions began to occur on day 9 and 10. Meanwhile, the decrease in COD levels during this stage of septage sludge with batik wastewater shows that stationary conditions begin to occur in the time after day 7. The stationary COD level indicates that the microbes in the sludge were growing and ready for use.

3.2. Effect on Electrical Energy Generation

The ability of a DCMFC system to produce strong currents and voltages is depended to the microorganism activity. Because microorganisms degrade organic levels, they produce electrons and protons. The produced electrons flow through the external circuit to the electron acceptor in the cathode chamber, and protons cross through the salt bridge to the cathode chamber. The difference in the reduction potential between electron donors and acceptors determines the potential energy availability of microorganisms for anabolism. Meanwhile, in the cathode chamber, KMnO4 was used to capture electrons from the anode chamber, acting as an electron acceptor. An oxidation reaction takes place in the cathode chamber because KMnO4 is reduced by gaining electrons and protons (Utami et al., 2020). The reactions occurring in the anode and cathode chambers were as follows.

Anode : $C_6H_{12}O_6 + H_2O \rightarrow 6CO_2 + 24e^- + 24H^+$ (1)

Cathode: $MnO_4^- + 4H^+ + 3e^= \rightarrow MnO_2 + 2H_2O$ (2)

In addition, the growth of microorganisms also affects the production of electrical energy. The growth phases of microorganisms are divided into 4, namely: 1) The lag phase is a phase of adaptation or the ability of bacteria to adjust to new environmental conditions. 2) The exponential phase is the second phase of growth. This phase is evidenced by the occurrence of a very fast growth period. 3) The stationary phase is the phase when the growth rate is equal to the microbial death rate, resulting in a decrease in the overall number of microbes. 4) The death phase is a phase that can be observed by an increase in the number of death rates that exceed the number of growth rates (Risna et al., 2022).

3.2.1. Production of Electric Current and Electric Voltage

In the DCMFC system, electrical energy is generated as electrons and protons produced by microbial metabolism move from the anode chamber to the cathode chamber via external circuit and salt bridges, respectfully. The power density can be calculated by multiplying the obtained current and voltage values and dividing with the total area of electrode used.
The measurement of strong current and voltage was performed every 24 hours using an avo-multimeter. The results of the electric current measurements for the variations in septage sludge and EM4 are shown in Figure 2.

The results showed that the strong electric current produced for each variation lasted for 240 h and tended to decrease. The lowest electric current strength was observed in the control reactor, with peak production at 48 h reaching a maximum of 0.84 mA. Furthermore, for the variation with the addition of EM4, the highest current strength was produced, with peak production of 1.36 mA at 168 h. The variation with the addition of EM4, after reaching its peak production, decreased slowly, but the strong electric current produced remained higher compared to the other variations. The variation with the addition of sewage treatment plant sludge reached a peak electric current strength of 1.24 mA at 192 h. To determine which variation was more optimal for producing a strong electric current, a comparison was made as shown in Figure 3.

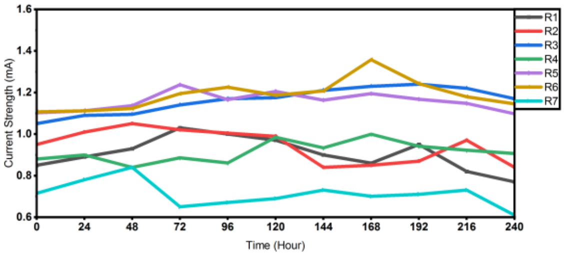

Figure 2. Electric current strength of different septage sludge and EM4

Description:
R1: Anode chamber with septage sludge and pH 6
R2: Anode chamber with septage sludge and pH 7
R3: Anode chamber with septage sludge and pH 8
R4: Anode chamber with EM4 and pH 6
R5: Anode chamber with EM4 and pH 7
R6: Anode chamber with EM4 and pH 6
R7: Anode chamber without any addition and pH setup
*reactor code used in each graph

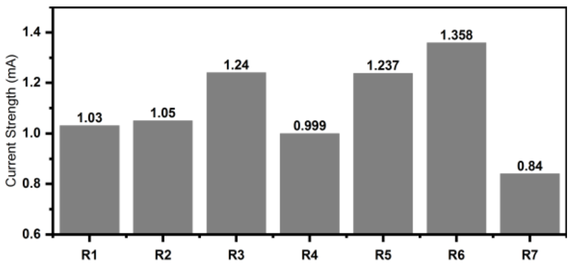

Figure 3. Comparison of Electric Current Strength under Different Variations in septage Sludge and EM4

After comparing each variation to find out which variation was more optimal for producing the electric current strength, the maximum electric current strength value was obtained in R6 with variations in the addition of EM4 conditioned at pH 7. After obtaining the current strength, voltage measurements were made for variations in the sewage sludge and EM4, the results of which can be seen in Figure 4.

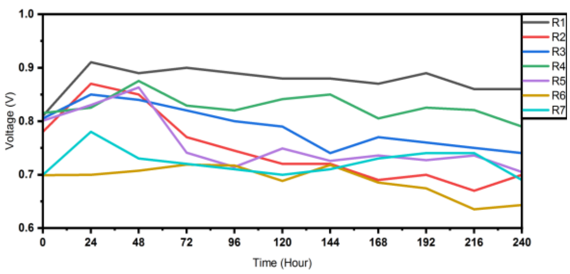

Figure 4. Electrical voltage variations in the septage sludge and EM4 Bacteria

The lowest voltage production occurred in the control reactor, where the maximum voltage was only 0.78 V at 24 h. In the variation with the addition of EM4, the highest voltage production occurred at 48 h under pH 6, producing a maximum voltage of 0.87 V. The variation with the addition of sewage treatment plant sludge reached its peak voltage production at 24 h under pH 6 conditions, producing a maximum voltage of 0.91 V. Similarly, the variation with the addition of septage sludge reached its peak voltage production at 24 h under pH 6 conditions, producing a maximum voltage of 0.91 V. To determine which variation was more optimal in producing electric voltage, a comparison of the maximum electric voltage for each variation was shown in Figure 5.

After comparing each variation to find out which variation is more optimal for producing electrical voltage, the maximum electrical voltage value was obtained in reactor 6 with variations in the addition of septage sludge conditioned at pH 8.

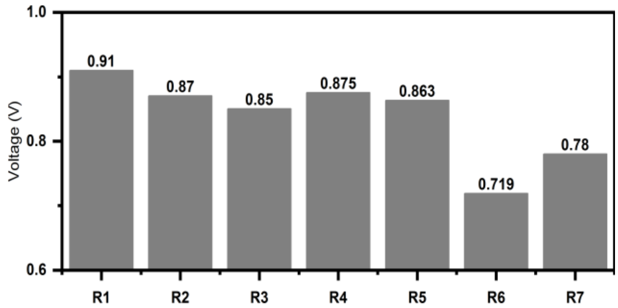

Figure 5. Comparison of Electrical Voltage at Different septage Sludge and EM4

3.2.2. Power Density Production

The power density is a measure of the power density calculated by multiplying the results of the current and voltage, and then the results are divided by the electrode surface area. The electrode surface area was 0.003297 m^2. Power density in MFC systems is influenced by the type of microorganism and its growth, organic substrate, environmental conditions, electrode design, anode and cathode conditions, current and voltage, and the use of electrolyte solution. Optimizing the combination of these factors is important for improving the performance and efficiency of MFC systems. In accordance with the research objectives, power density is a parameter used to assess the production of electrical energy generated by variations in the addition of septage sludge and EM4. Then, the production of power density for variations in the septage sludge and EM4 is shown in Figure 6.

The results indicated that the lowest power density was produced in the control reactor, with a maximum power density of 185.99 mW/m² at 48 h. Despite the absence of added microorganisms, the control reactor still generated power density due to microorganisms present in the batik wastewater substrate. For the EM4, the highest maximum power density was produced at pH 7, reaching 297.61 mW/m² after 48 h. The peak power density production in the EM4 variation occurred during the exponential phase, which tends to rise, indicating rapid and constant microbial cell division. An increase in the number of bacterial cells allows more protons and electrons to be generated through metabolic processes.

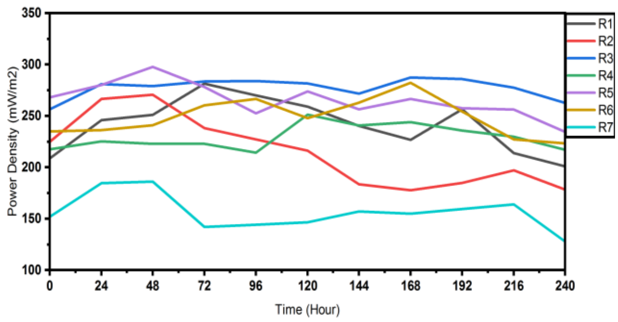

Figure 6. Power Density Variations in septage Sludge and EM4

For the septage sludge variation, the maximum power density reached 287.26 mW/m² at pH 8. This occurred because the bacteria were in the stationary phase, where slow bacterial growth was accompanied by the presence of dead bacteria. A comparison of the maximum power density for each variation is shown in Figure 7.

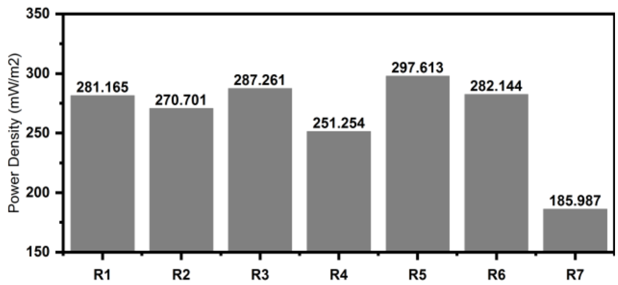

Figure 7. Comparison of power density among septage sludge dan EM4

Research aimed at examining the effect of adding septage sludge and EM4 on the power density of DCMFCs found that the highest power density was achieved with the addition of EM4, reaching a maximum value of 297.613 mW/m² at the 48 h. After peaking at 48 h, the power density production decreased due to the growth of microorganisms. To determine the growth of microorganisms in the DCMFC system, MLSS analysis can be performed, and to identify the biofilm that inhibits electron transfer in the DCMFC system, biofilm analysis can be conducted.

MLSS analysis measures the number of dissolved and suspended solids present in the activated sludge mixture in wastewater treatment systems. The quantity of MLSS can provide an idea of the biological conditions in the DCMFC system. However, it cannot be used to determine the specific number of active microorganisms. MLSS analysis is carried out by the gravimetric method every 48 h. The results of MLSS measurement can be seen in Figure 8.

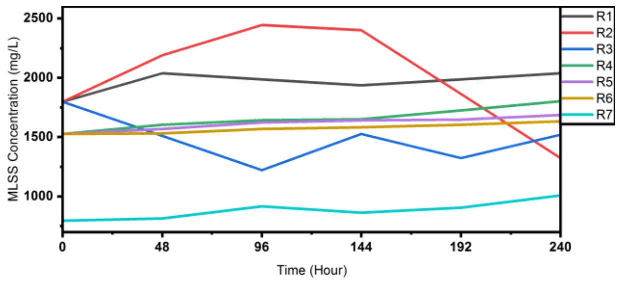

Figure 8. MLSS measurement

The results of the MLSS analysis showed data on the variation in EM4; the MLSS value increased until the end of the study, ranging from 1500 to 1800 mg/L. The increase in MLSS in the EM4 variation indicated that microorganisms were in a stationary phase, suggesting that the growth rate of microorganisms was less than their death rate. In contrast, the MLSS analysis for the variation in septage sludge microorganisms revealed they were in a different phase. At pH 7, the septage sludge microorganisms experienced a death phase, meaning the number of dead microorganisms was greater than the number of living ones. This death phase occurred because the living microbes found it difficult to adapt, having been initially conditioned in an aerobic condition on oxidation ditch in STP. However, at pH levels of 6 and 8, the microorganisms were in a stationary phase, indicating that the ratio of living to dead microorganisms was equal.

As the organic levels were reduced, microorganisms in the form of biofilms adhered to the surface of the electrode, including the extracellular polymeric substances (EPS). As the microorganism's population grew, both living and dead cells formed a layer on the anode. When the biofilm covered the anode surface, the number of electrons transferred to the electrode was reduced, resulting in a decrease in the electric current. Biofilm measurements were performed by weighing the electrodes every 48 hours. The results of the biofilm measurements are shown in Figure 9.

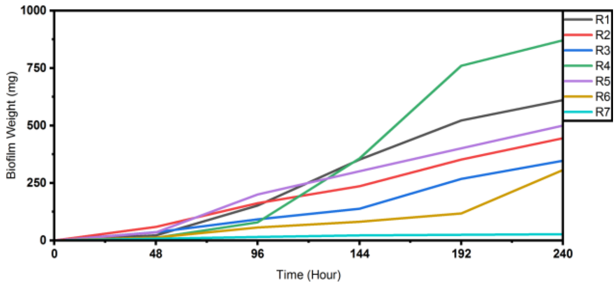

Figure 9. Biofilm weights on the anode

Biofilm production increased for each variation. In the variation with the addition of EM4, the largest increase in biofilm weight was observed under pH 6, with an approximate weight increase of 1064 mg. At pH 7 and pH 8, the weight gains were 499 mg and 307 mg, respectively. For the variation with the addition of septage sludge, the largest increase in biofilm weight was also observed under pH 6 conditions, with an additional weight of approximately 610.7 mg. At pH 7 and pH 8, the weight gains were 443.99 mg and 346 mg, respectively. The effect of biofilm weight on the maximum power density value in the variation with EM4 can be seen in Figure 10.

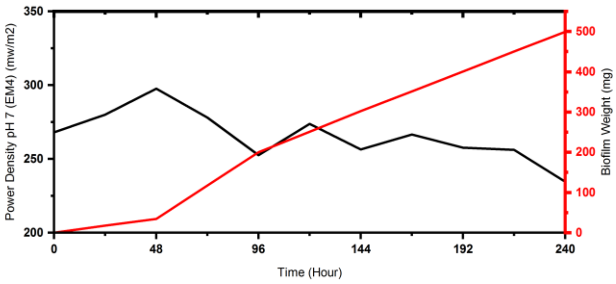

Figure 10. Comparison of Power Density and Biofilm Weight

Based on Figure 10, observations of power density parameters and biofilm weight began with the variation of adding EM4 conditioned at pH 7. The biofilm in this variation increased from the beginning to 48 h, at which time the maximum power density was reached. Subsequently, the biofilm weight increased, inhibiting the electron transfer process, and resulting in a decrease in the power density value.

3.3. Effect of pH on the reduction of organic content

Microbial growth influenced the degradation of organic content in DCMFCs. Microbial growth in all reactors generally decreased during the initial processing phase, followed by a not-so-significant increase in the following days. This decrease might have been due to microbial adaptation to the substrate load and wastewater conditions (Wardhan & Effendi, 2019). One of the factors that influenced wastewater conditions was pH. The pH of wastewater affected anaerobic microorganisms. Appropriate pH conditions were necessary to maintain enzyme activity and microorganism balance. Because the microbes used included more than one type, pH variations were carried out to determine the optimum pH for each microbe. To determine the effectiveness of pH variation, COD levels were measured. Good microbial growth was supported by changes in pH, which were considered a factor affecting bacterial growth. Most microbes grew well at approximately neutral pH, with pH 6.0-8.0 being the optimum condition for bacterial growth (Sjarif & Rosmaeni, 2019).

3.4. Effect on COD removal

Initially, COD in batik wastewater ranged from 7800 to 8300 mg/L. The measurement results and efficiency of COD removal are shown in Figure 11.

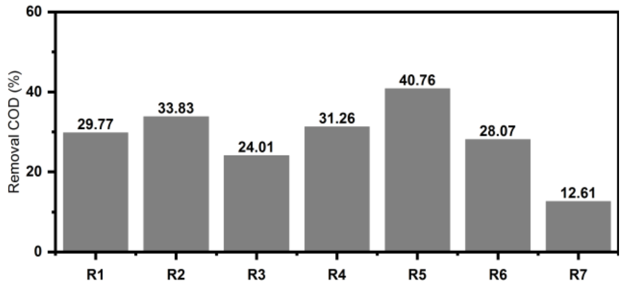

Figure 11. COD removal efficiency

Based on the results of this study, it was observed that the lowest reduction efficiency occurred in the control reactor (12.61%) because no additional microorganisms were added. The EM4 exhibited the highest reduction efficiency at pH 7 (40.76%). The pH 6 had a reduction efficiency of 31.26%, while the lowest efficiency was 28.0% for the pH 8. This result was consistent with the expectation that the optimum pH range for bacterial growth is 6.5–8 [15]. Furthermore, the septage sludge exhibited the highest reduction efficiency at pH 7 (33.83%). For the pH 6, the reduction efficiency was 29.77%, and the lowest efficiency was 24.01% for the pH 8.

The observed decrease in efficiency was consistent because, as the MFC operation time increased, the organic content in the wastewater or substrate decreased. This reduction was due to the activity of microorganisms in the wastewater, which lowered the organic levels in the anode chamber.

4. CONCLUSION

Based on the results of this study, variations in the addition of EM4 produced a higher power density than the addition of septage sludge. In the DCMFC system that uses a wastewater substrate with the addition of EM4 and is conditioned at pH 7, the highest power density was reached 297.61 mW/m^2 at 48 h. The septage sludge was conditioned at pH 8 to produce a maximum power density of 287.26 mW/m^2 over 168 h. Meanwhile, the control reactor generated a power density of 185.99 mW/m^2 over 48 h. The MLSS values in the DCMFC systems with EM4 increased steadily from 1500 mg/L to 1800 mg/L throughout the study period, regardless of the pH conditions. In the systems with the addition of septage sludge, the pH level influenced the metabolic activity and survival of MLSS, with pH 7 being detrimental in this case. Biofilm attached in the anode in the system with EM4 increased over time for all pH conditions, as well as in the systems with septage sludge significantly on pH 6.

Based on the results of research related to differences in the pH conditions in the DCMFC systems with a pH range of 6-8. It was found that the optimum pH for the septage sludge and EM4 to degrade COD was 7. The ability of the septage sludge to remove COD was 33.83%. The ability of EM4 to remove organic content was thus higher than that of the septage sludge. With the ability to remove COD levels of 49.76%. It is recommended for other studies to use variations addition of EM4 to reduce organic content and enhance power density.

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