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Cr(VI) Removal Using A Dual Chamber Microbial Fuel Cell

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Abstract

Chromium ions is notably a hazardous heavy metal due to its toxic and carcinogenic nature, particularly in its hexavalent form, Cr(VI). One of the major Cr(VI) pollution source is from electroplating industry effluent, which may contain high concentrations that pose a risk of contamination of aquatic and soil ecosystems if not treated carefully. One of the alternative method known to be able to treat Cr(VI) wastewater is by using microbial fuel cell (MFC). This research focused on on the removal of Cr(VI) from synthetic electroplating wastewater using a 4L dual-chamber MFC under fed-batch condition, as well as investigating the impact of mixed liquor suspended solids (MLSS) and chemical oxygen demand (COD) concentrations to its performance. Observed parameters include the efficiency of Cr(VI) removal and power density. Septage sludge and acetate were both used as biomass and substrate source respectively. Based on this research, it can be concluded that the highest Cr(VI) removal efficiency and power density were achieved using specific MLSS and COD concentration that resulting in F/M ratio of 0,459 to 0,489 gCOD/gMLSS. From initial Cr(VI) concentration of 50 mg/L, the highest removal was obtained by MFC running with initial MLSS and COD concentrations of 3.500 and 1.500 mg/L respectively, achieving 62,17% over 312 hours. This setup also produced the highest power density of 48,22 mW/m².

Keywords : Dual Chamber; Microbial Fuel Cell; Power Density; Septage Sludge

INTRODUCTION

Cr(VI) pollution in the environment is a serious issue that needs to be addressed. Even in small concentrations, Cr(VI) can have harmful effects on the environment, especially on living organisms. According to the Ministry of Health Regulation No. 2 of 2023, the permissible level of dissolved Cr(VI) in drinking water and sanitation is a maximum of 0,01 mg/L. The occurrence of Cr(VI) pollution has been increasing along with the development of human activities, particularly in industrial operations that using chromium on their production process. Chromium is widely used as a raw material in metal plating, leather tanning, painting, steel manufacturing, and wood processing industries. Each of these industrial activities potentially produces untreated Cr(VI) waste. The electroplating industry is one of the largest sources of Cr(VI) pollution because it frequently uses Cr(VI) bath to coat metals (Saha et al., 2022). The electroplating process includes several steps, i.e pretreatment and preparation, plating, rinsing, passivating, and drying. The cleaning and pretreatment stages utilize various solvents and surface stripping agents, such as caustic soda and various strong acids, depending on the type of metal surface being plated (Singh & Ram, 2016). Due to differences in process efficiency, the composition of the electroplating baths, and the effectiveness of wastewater treatment practices, the wastewater from metal plating can contain chromium concentrations ranging from 0,19 to 25.176 mg/L (Rajoria et al., 2022). This makes metal plating industrial waste highly dangerous even in small amounts.

Chromium generally exists in nature in two oxide forms: trivalent chromium (Cr(III)) and hexavalent chromium (Cr(VI)). The tendency of chromium to form trivalent or hexavalent ions depends on the pH and oxidative properties of the surrounding environment. In acidic conditions, chromium tends to form hexavalent ions, specifically chromate ions (CrO_4^{2-}) (Masduqi & Assomadi, 2016). These two valence forms of chromium have different chemical properties. Cr(III) is considered

harmless because it is poorly absorbed by the digestive system and is needed in small amounts for glucose tolerance factor formation and insulin metabolism in humans. Conversely, Cr(VI) is a strong oxidizer that is toxic, carcinogenic, mutagenic, causes liver and kidney necrosis, and induces allergic contact dermatitis in living organisms (Barceloux & Barceloux, 1999). Cr(VI) is also known for its high solubility and mobility in the environment, especially in water bodies.

Given the significant negative impact of Cr(VI) on the environment, it is necessary to treat electroplating industrial waste to meet the government's environmental quality standards before disposal. Currently, electroplating wastewater treatment typically involves technologies such as electrocoagulation, electrodialysis, electrodeionization, ion exchange, adsorption with activated carbon, and microbial degradation (GracePavithra et al., 2019). Most of these technologies have drawbacks, including high energy requirements, the need for chemicals, and high operational costs. Therefore, there is a need for a cost-effective yet efficient alternative technology for treating Cr(VI) waste (Khairani & Azam, 2007). With the increasing growth of industries producing Cr(VI) waste, stringent regulations on wastewater quality, and the demand for environmentally sustainable development, an effective and efficient treatment technology is essential (Agustina et al., 2018).

MFCs are an alternative technology for treating Cr(VI) wastewater. They are favored in contemporary applications because of their high efficiency and minimal emissions, especially when compared to traditional combustion systems that often emit greenhouse gases. MFCs operate on the principle of using the metabolism of anaerobic microbes to catalyze the oxidation of organic substrates, which generates electrons for electricity production. Typically, an MFC comprises two chambers: the anode and cathode, which are separated by a proton exchange membrane (PEM) that facilitates proton transfer.

Within the anode chamber, anaerobic microbes and organic substrates co-exist. The microbes break down organic matter to extract energy and propagate. This decomposition yields CO_2, protons, and electrons as byproducts. The electrons are transferred to the anode via a cellular respiration chain (Rismani-Yazdi et al., 2008). MFCs can utilize a variety of organic substrates, including glucose, acetate, and complex polymers like starch or cellulose. Additionally, they can process domestic, industrial, and slaughterhouse waste as substrates (Das, 2018). The anaerobic breakdown of glucose in the anode chamber can be illustrated by the following chemical reaction:

$$C_6H_{12}O_6 + 6H_2O \xrightarrow{microorganism} 6CO_2 + 24H^+ + 24e^- \qquad (E^o = 0{,}14V \text{ vs SHE}) \qquad (1)$$

The electrons concentrated at the anode are transferred to the cathode through an external circuit with a specific electrical resistance. Upon reaching the cathode, the electrons are reduced by a terminal electron acceptor (TEA) such as oxygen (Logan and Rabaey, 2012).

$$6O_2 + 24H^+ + 24e^- \rightarrow 12H_2O \qquad (E^o = 1{,}23V \text{ vs SHE}) \qquad (2)$$

In the treatment of electroplating industrial waste, Cr(VI) acts as an oxidizing agent that replaces oxygen in the cathode compartment. Cr(VI) undergoes reduction to Cr(III), which has a lower toxicity, thereby reducing the potential negative impact on the environment. The half-cell reduction reaction of Cr(VI) to Cr(III) in the cathode compartment can be represented by the following equation:

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow Cr^{3+} + 7H_2O \quad (E^o = 1{,}33\ V) \qquad (3)$$

Under standard conditions (pH = 0), the above reaction has a reduction potential of 1,33 V compared to the standard hydrogen electrode (SHE). This value is higher than the E^o of O_2/H_2O, which is 1,23 V. This indicates that Cr(VI) is more readily reduced than oxygen, making it suitable for treating MFCs.

As shown in Eq.1, MFCs use microorganisms as catalysts to oxidize organic matter, transferring electrons produced from this oxidation to the anode, thus generating bioelectricity. The amount of bioelectricity generated is proportional to the number of electrons produced, which is directly linked to the ability of microorganisms to degrade a substrate (Hidayat et al., 2022; Jung & Pandit, 2019). Thus, investigating COD and MLSS in anode compartment is crucial because they significantly influence the performance of MFCs (Fazli et al., 2018; Kim et al., 2016; Tamilarasan et al., 2017). MLSS

represents the concentration of active biomass, which is crucial for sustaining microbial activity and facilitating continuous oxidation processes. Similarly, COD measures the amount of organic matter available for oxidation, which is directly correlated with the potential for electron generation and power output. Generally, higher substrate concentrations yield higher power generation over a wide concentration range (Gurung et al., 2012). However, there is specific range of COD concentrations that is optimal for achieving both maximum COD removal efficiency and power output, which depends on the configuration of the MFC used and the characteristics of the treated wastewater (Scott & Yu, 2016).

In this study, the Cr(VI) concentration in synthetic electroplating industrial waste was removed using a dual chamber MFC. The main objective of the present study was to examine the effect of MLSS and COD concentrations on Cr(VI) removal in the MFC system by using septage sludge as biomass source. The performance was evaluated based on Cr(VI) removal efficiency and power density. The experimental variables in this study included MLSS concentrations of (1.200, 2.000, and 3.500 mg/L) and COD (500, 1.000, and 1.500 mg/L) in the anode compartment, while Cr(VI) concentration was initially set to 50 mg/L in the cathode compartment. Observation is conducted within 13 days for each MFCs setup.

METHODS

Seeding and Acclimatization

Septage sludge obtained from the inlet of Solid Separation Chamber (SSC) at the Septage Sludge Treatment Plant (IPLT) in Keputih Village, Sukolilo District, Surabaya. The septage sludge collection followed the SNI 8990-2021 standard for "Methods of Sampling Wastewater for Physical and Chemical Testing." During seeding and acclimatization, the sludge was combined with a substrate solution in a sealed plastic container to a total volume of 20 L.

The substrate solution (pH 7,0) was prepared by dissolving 0,31 g of NH_4Cl, 2,75 g of $Na_2HPO_4 \cdot 12H_2O$, 4,97 g of $NaH_2PO_4 \cdot 2H_2O$, and 0,13 g of KCl (Merck, p.a.) in distilled water. Sodium acetate was added to obtain a mixed COD concentration of 2.500 mg/L. The septage sludge and substrate solution were mixed through trial-and-error process to achieve an MLSS concentration of 3.500 mg/L. The mixture was then flushed with N_2 gas at 1 L/min for 30 min to lower the dissolved oxygen content and to establish anaerobic conditions.

During the acclimatization period, the system pH was maintained between 6,5-7,5 using 0,1 M HCl and NaOH solutions. Samples were collected every 24 h to monitor COD levels. Acclimatization was deemed complete when the COD reduction consistently showed stable results with less than 10% fluctuation (Amelia Fitriani et al., 2015).

MFC Reactor

In this study, 4 sets of dual chamber MFC reactor was constructed in a lab-scale size. The reactor vessels are cylindrical plastic containers with an effective volume of 4 L each. One side of each vessel is perforated and connected to a 10 cm PVC pipe containing a salt bridge. This salt bridge is made from a 1 M KCl solution dissolved in 5% agar. The opposite side of each vessel is also perforated and equipped with a sampling valve. The electrodes used are graphite electrodes with an effective surface area of 29,532 cm^2 each. These electrodes underwent activation by immersion in a 1 M HCl solution for 24 hours, followed by immersion in a 1 M NaOH solution for another 24 hours. This activation process aims to remove foreign metal and organic contaminants adhering to the electrodes (Wu et al., 2018). Each electrode is connected to copper electrical cables and secured with wax at the ends. Both electrodes are connected to a digital multimeter (DT-830B) to measure the voltage and electric current generated. The schematic diagram of the MFC reactor setup is depicted in Figure 1.

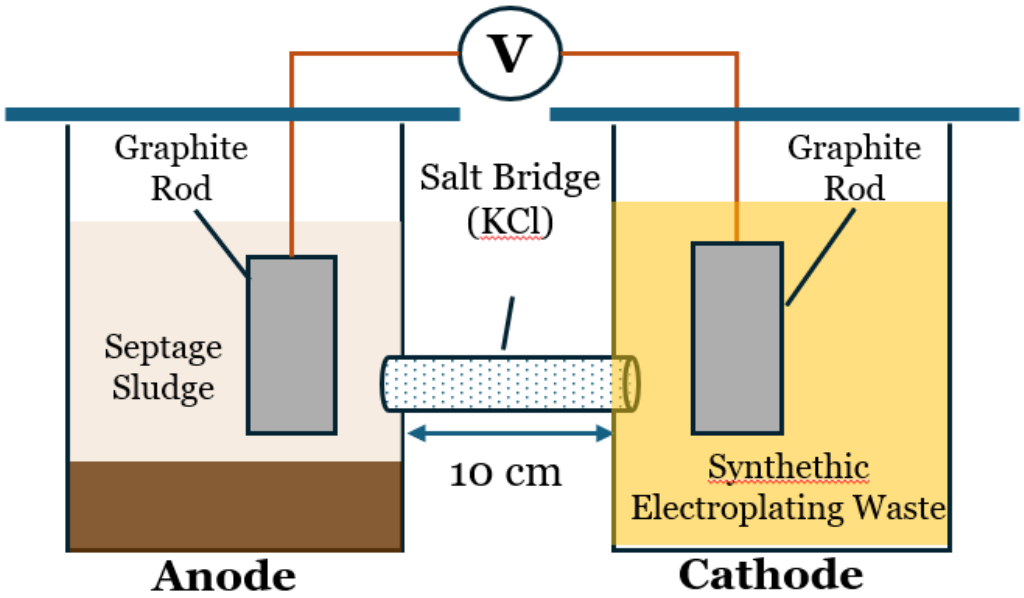

Figure 1. Microbial Fuel Cell Setup

Cr(VI) Removal Stage

Upon completion of the acclimatization phase, the septage sludge was moved to the anode chamber and combined with a substrate solution with a specific initial COD concentration according to the experimental step. Technical grade $NaCH_3COO$ (sodium acetate) was gradually added until a final volume of 4 L was reached. In the cathode compartment, synthetic electroplating wastewater was prepared by dissolving $K_2Cr_2O_7$ crystals (Merck, p.a.) in distilled water to create a 4 L solution with a Cr(VI) concentration of 50 mg/L. The pH of the cathode compartment was adjusted to 2 using 0.1 M H_2SO_4.

Both compartments were then purged with N_2 at a rate of 1 L/min for 15 min to eliminate dissolved oxygen, thereby establishing anaerobic conditions in the anode and preventing any competition between oxygen and Cr(VI) removal at the cathode. Parameters such as COD, MLSS, electric current, and voltage at the anode, along with Cr(VI) concentration at the cathode, were monitored every 24 h.

Analysis and Calculations

This research was conducted experimentally on a laboratory scale at the Workshop dan Laboratorium Teknologi Pengolahan Air, Departemen Teknik Lingkungan, Institut Teknologi Sepuluh Nopember, from February to June 2024. The procedures for analyzing the test parameters in this study are detailed in Table 1. Water sampling from the MFC reactor followed methods compliant with SNI 6989.59:2008.

Table 1. Analytical Method

Parameter	Methods	Interval
COD	Closed Reflux by Titrimetry (SNI 6989.73:2019)	Every 24 h
MLSS	Gravimetric (SNI 06-6989.3-2004)	
Cr(VI)	Colorimetry (SNI 6989.71:2009)	
Voltage	Manually using Multimeter Digital (DT-830B)	
Current		

The concentration of Cr(VI) ions was measured using the colorimetric method, which detects the red-violet complex formed from the specific reaction between Cr(VI) and diphenylcarbazide at low pH (APHA, 2003). The measurements were conducted using a 721G UV-Vis Spectrophotometer at a wavelength of 540 nm.

Power density can be calculated by using following equation :

$$Power\ Density\ \left(\frac{mW}{m^2}\right) = \frac{I\ (mA) \times V\ (volt)}{A\ (m^2)} \quad (4)$$

With I = current (mA), V = voltage (volt), dan A = anode surface area (m^2). *Removal efficiency* Cr(VI) and COD can be calculated by using following equation :

$$\%Removal\ Efficiency = \frac{Co-Ct}{Co} \times 100\% \quad (5)$$

With Co = Initial concentration of Cr(VI) or COD, Ct = final concentration Cr(VI) or COD at time = t.

RESULTS AND DISCUSSION

Acclimatization of Sewadge Sludge using Sodium Acetate

The acclimatization process helps the microbial consortium in septage sludge adapt to a new substrate, sodium acetate. The acetate group is often used as a substrate in MFC applications because it is inert to fermentation conversion by anaerobic microbes at room temperature (Khater et al., 2017). In an MFC system, this is advantageous because acetate can promote the growth of specific microbes that release electrons during metabolism. These microbes are called electrochemically active bacteria (EAB). Additionally, acetate is a simple carbon compound, making it easy for EAB to degrade (Sonawane et al., 2022). In this phase, septage sludge and substrate solution are mixed in a sealed plastic container and then purged with N2 gas at a flow rate of 3 L/min for 15 min. This N2 gas flow reduced the dissolved oxygen (DO) content in the system, creating an anaerobic environment. Under anaerobic conditions, acetate degradation during acclimatization is represented by the following reaction equation (Li et al., 2008).

$$CH_3COO^- + 4H_2O \xrightarrow{EAB} 2HCO_3^- + 9H^+ + 8e^-$$

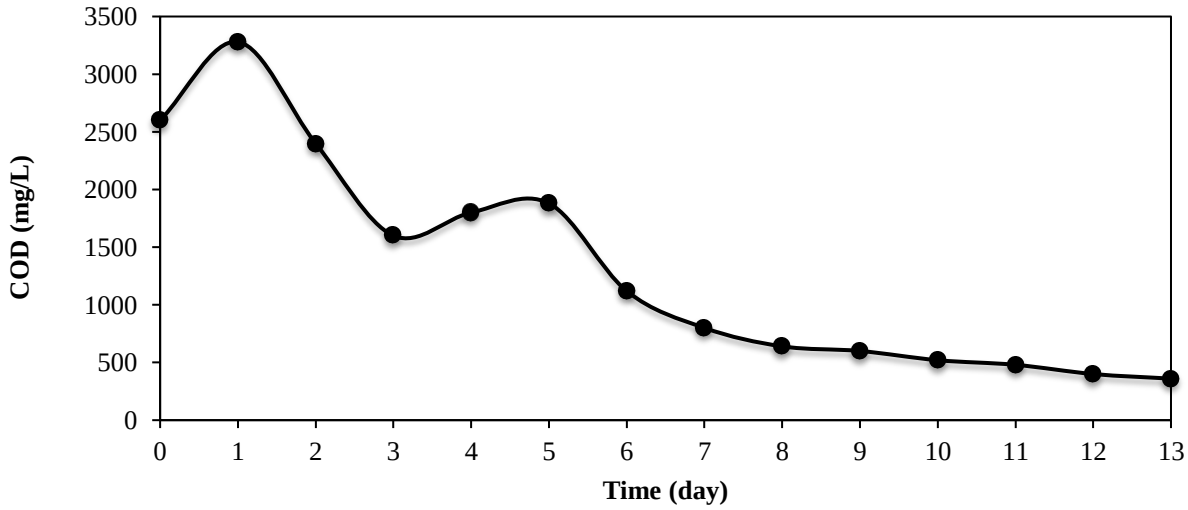

Figure 2. COD Reduction during Acclimatization Stage

In this study, the acclimatization process began with initial COD and MLSS concentrations of 2,600 and 3,500 mg/L, respectively, in the pH range of 6.5 - 7.5. As shown in Figure 2, the COD concentration decreased over time during the acclimatization process. This reduction in the COD concentration indicates acetate degradation by the microbial consortium in the septage sludge. The COD concentration decreased by 86.15% from 2,600 to 360 mg/L. The total COD reduction in this study is similar to the 86% reduction reported by Karlikanovaite-Balıkçı & Özgün (2020) over 30 days using domestic waste and acetate as microbial and substrate sources in MFC experiments. Regarding the acclimatization period, the microbial consortium in septage sludge adapted faster, requiring only 312 hours (13 days) to stably degrade the substrate. This may be due to the presence of trace elements in septage sludge, such as Ni, Co, Cu, and Fe, which are essential for microbial growth (Colón et al.,

2015; Rose et al., 2015). According to Lee et al. (2022), using septage sludge can enhance the anaerobic degradation of organic materials. Additionally, Figure 4.3 shows fluctuations in COD concentration, especially between 0 and 120 h. From 0 to 24 h, COD increased from 2,600 mg/L to 3,280 mg/L, possibly due to shock loading (Rohim et al., 2015). When septage sludge is added to the substrate solution, the organic load on the microbes suddenly increased. This can cause microbes to enter a lag phase, hindering substrate degradation. From 24 to 72 h, the COD concentration sharply decreased by 51.2%, from 3,280 mg/L to 1,600 mg/L, indicating microbial adaptation to the substrate and the start of degradation. However, at 96 and 120 h, COD increased from 1,600 to 1,880 mg/L. This could be due to the formation of intermediate compounds via hydrolysis, acidogenesis, or acetogenesis of organic substrates originated in septage sludge (Hunter et al., 2021; Schievano et al., 2012; Yuan and Zhu, 2016). These intermediate compounds were further degraded, as evidenced by the relatively stable COD reduction from 1,120 to 480 mg/L from 144 to 312 h. This COD reduction indicates that the microbial consortium in the sludge has been fully adapted, allowing stable substrate degradation. At the end of this phase, the septage sludge is transferred to the anode compartment of the MFC system.

Effects of Initial MLSS and COD Concentration on Cr(VI) Removal

The successful reduction of Cr(VI) concentration in a microbial fuel cell system relies on effective proton and electron transfer from the anode to the cathode. Sodium acetate acts as a carbon source (substrate), which is decomposed by microbes at the anode to produce protons and electrons. These protons and electrons then travel through the salt bridge and copper cables to the cathode chamber, proceed to reduce Cr(VI) ions to Cr(III) based on following Eq :

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow Cr^{3+} + 7H_2O \quad (E^\circ = 1{,}33\ V)$$

This result highlights that the efficiency of Cr(VI) removal is directly linked to the metabolic activity of the microorganisms in the anode chamber.

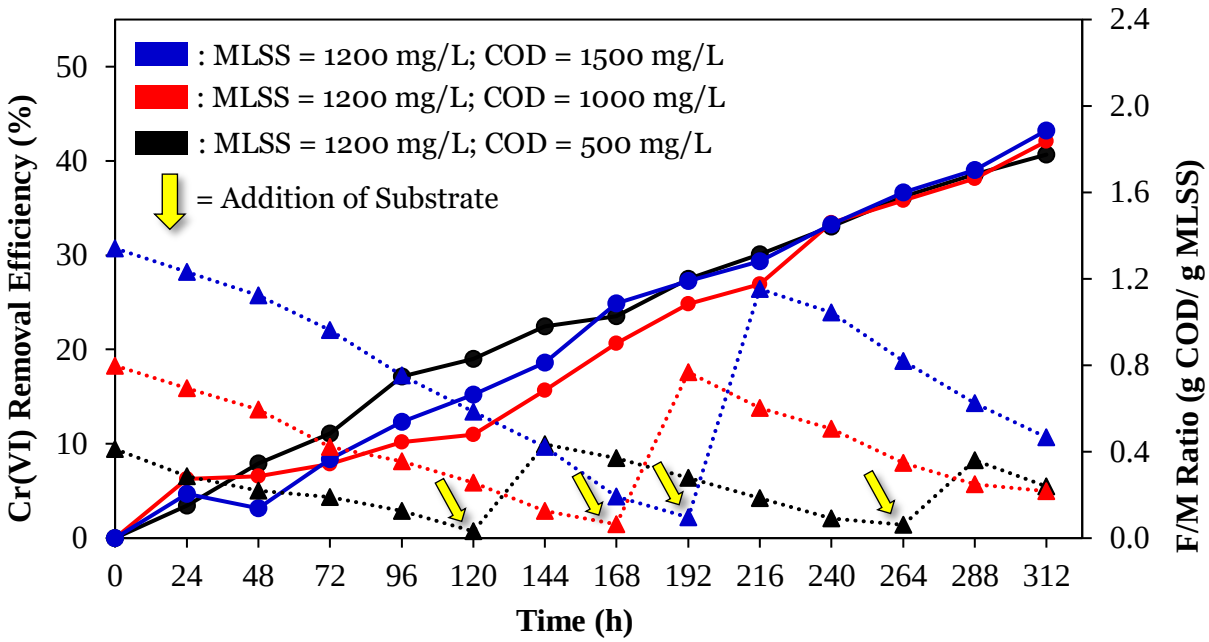

Figure 3(a). Cr(VI) Removal Efficiency (—●—) and F/M Ratio (···▲···) at Initial MLSS Concentration = 1.200 mg/L

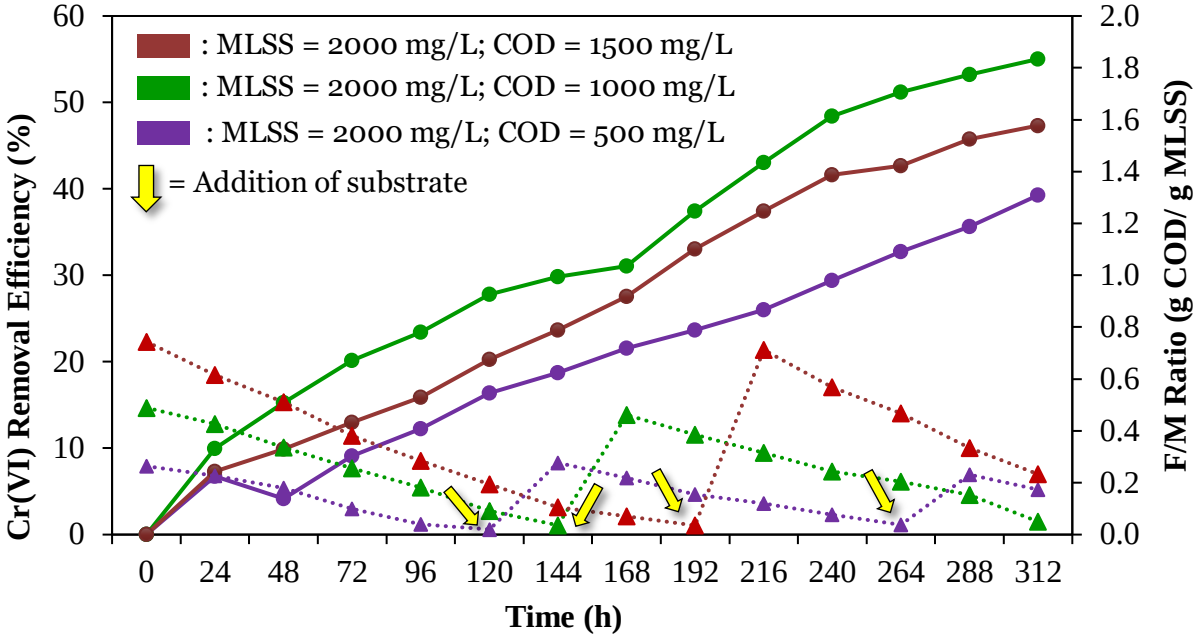

Figure 3(b). Cr(VI) Removal Efficiency (—●—) and F/M Ratio (···▲···) at Initial MLSS Concentration = 2.000 mg/L

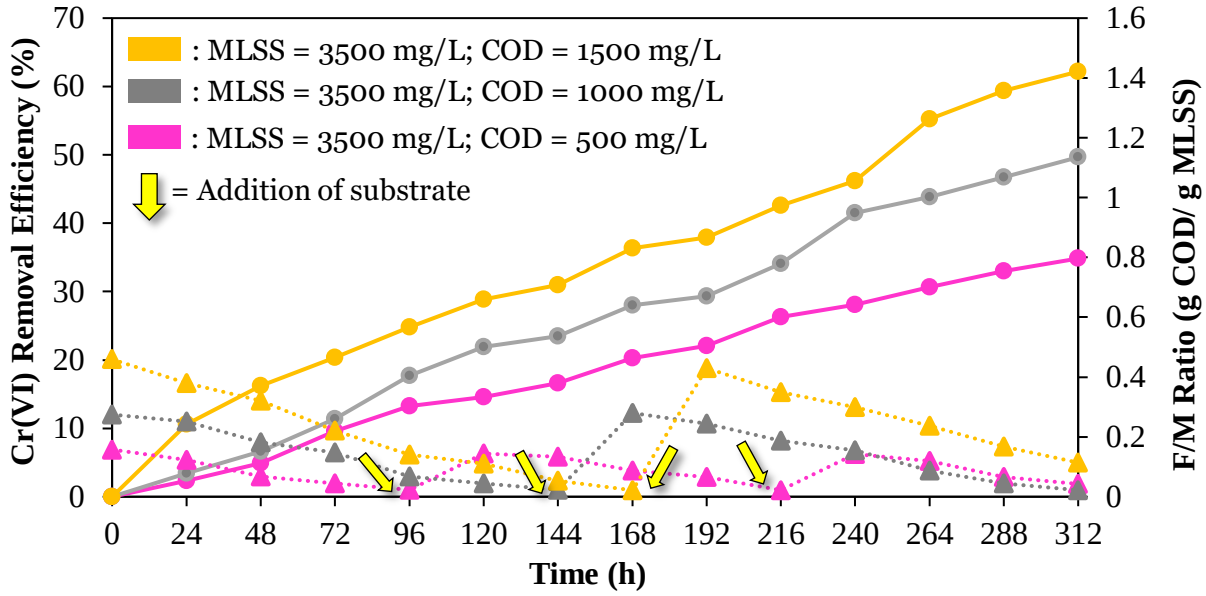

Figure 3(c). Cr(VI) Removal Efficiency (—●—) and F/M Ratio (···▲···) at Initial MLSS Concentration = 3.500 mg/L

Generally, the experimental results showed that overall efficiency of Cr(VI) removal is influenced by the initial concentrations of MLSS and COD in the anode chamber, with results varied from 34,84% to 62,17% during observation period of 13 days. At an initial MLSS concentration of 1.200 mg/L, the Cr(VI) concentration decreased by approximately 40,68% to 42,08% across various COD concentrations in the anode compartment. Figure 3(a) illustrates that higher initial COD concentrations lead to greater Cr(VI) removal efficiency. This phenomenon occurs because higher substrate availability enhances microbial degradation processes, resulting in increased release and transfer of electrons and protons to the cathode (Safitri et al., 2023). A further increase of MLSS concentration to 2.000 mg/L, the Cr(VI) concentration was reduced by 39,26% to 55,02%. Based on Figure 3(b), it can be seen that Cr(VI) removal increased as the initial COD concentration increased from 500 to 1.000 mg/L and then decreased at 1.500 mg/L. This fluctuation is likely due to excessive

substrate availability, which causes instability during acetate degradation by microbes within the system. Anaerobic degradation of organic materials can be disrupted when the food-to-microorganism ratio (F/M ratio) deviates from the optimal value. Wagiman (2001), noted that an excessively low F/M ratio can hinder metabolic processes due to an insufficient food supply for microbes. Conversely, excessively high F/M ratios can lead to metabolic imbalances among microbes. Moreover, excess substrate can induce self-inhibition during microbial growth (Zhao et al., 2016). At an MLSS concentration of 2.000 mg/L, the highest Cr(VI) removal efficiency of 55,02% was achieved by using initial COD concentration 1.000 mg/L, resulting in F/M ratio of 0,489 gCOD/gMLSS. These findings agree with those of Banerjee et al. (2023), indicating that the optimum F/M ratio generally ranges from 0,4 to 0,5 gCOD/gVSS in MFC systems. A similar trend was observed at an initial MLSS concentration of 3.500 mg/L, where Cr(VI) removal efficiency increased from 34,84% to 62,17% with an initial COD supply ranging from 500 to 1.500 mg/L. The highest Cr(VI) removal efficiency was attained at an initial COD concentration 1.500 mg/L with an F/M ratio of 0,459 gCOD/gMLSS. In conclusion, the optimal F/M ratio for MLSS concentrations between 2.000 and 3.500 mg/L ranged from 0,459 to 0,489 g COD/gMLSS. However, the opposite trend is observed at MLSS = 1200 mg/L, where the highest Cr(VI) removal efficiency occurred at an F/M ratio of 1,341. This phenomenon suggests limitations in the microbial capacity to degrade organic substrates, as indicated by the minor difference in Cr(VI) removal efficiency across various conditions, only differing by 1.4%.

Effects of Initial MLSS and COD Concentration on Electricity Production

The electrical power output was measured daily by measuring the voltage and current in each MFC reactor. Electrical power generation in MFCs occurs simultaneously with the degradation of organic compounds by microbes. Based on these observations, the voltage started to increase 24 h after the reactors were operated. Over the 312-hour observation period, the generated voltage exhibited fluctuating patterns, forming 2-3 peaks as shown in Figures 5 (a,b,c). These patterns indicate that during this period, there were 2-3 cycles of electrical voltage production.

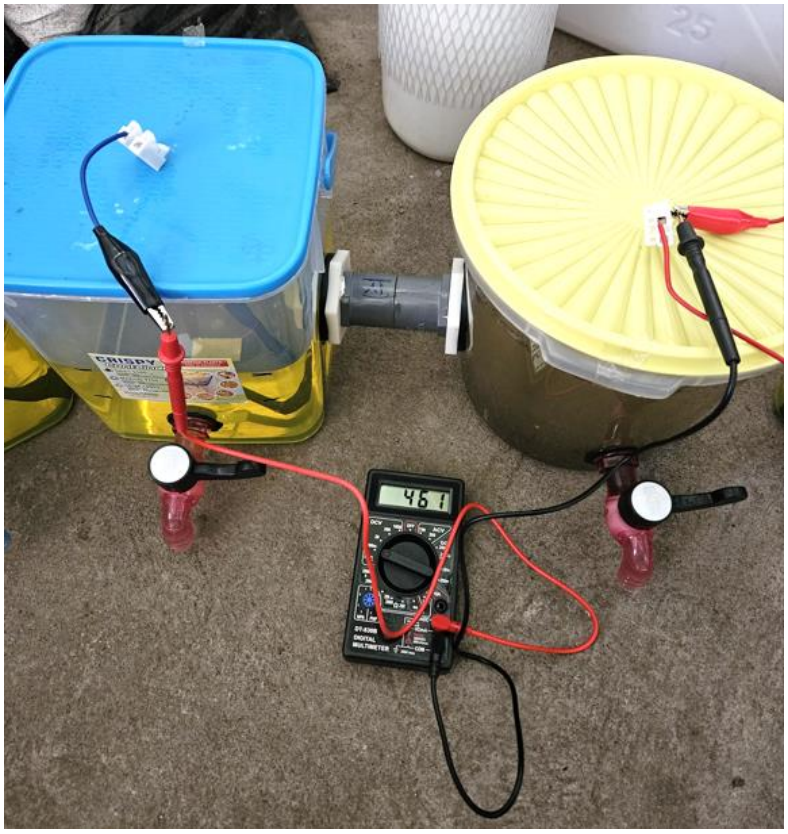

Figure 4. Voltage Measurement

Based on Figure 5(a), at an initial MLSS concentration of 1.200 mg/L, the peak electrical

voltages generated at various COD concentrations ranged from 611-670 mV. At an initial COD concentration of 500 mg/L, there were 3 peaks in electrical voltage production observed at 48, 168, and 288 h ranging from 596, 632, and 601 mV, respectively. This indicates that the electrical voltage production tends to increase with the addition of substrate in the anode compartment, which is correlated with the COD removal process. Over time, substrate is continuously consumed until the COD concentration drops to 40-80 mg/L and couldn't be reduced further due to the limited biodegradability of sodium acetate. During this period, substrate was added into the anode chamber at a certain amount to finally reach the initial COD concentration i.e 500, 1.000, and 1.500 mg/L.

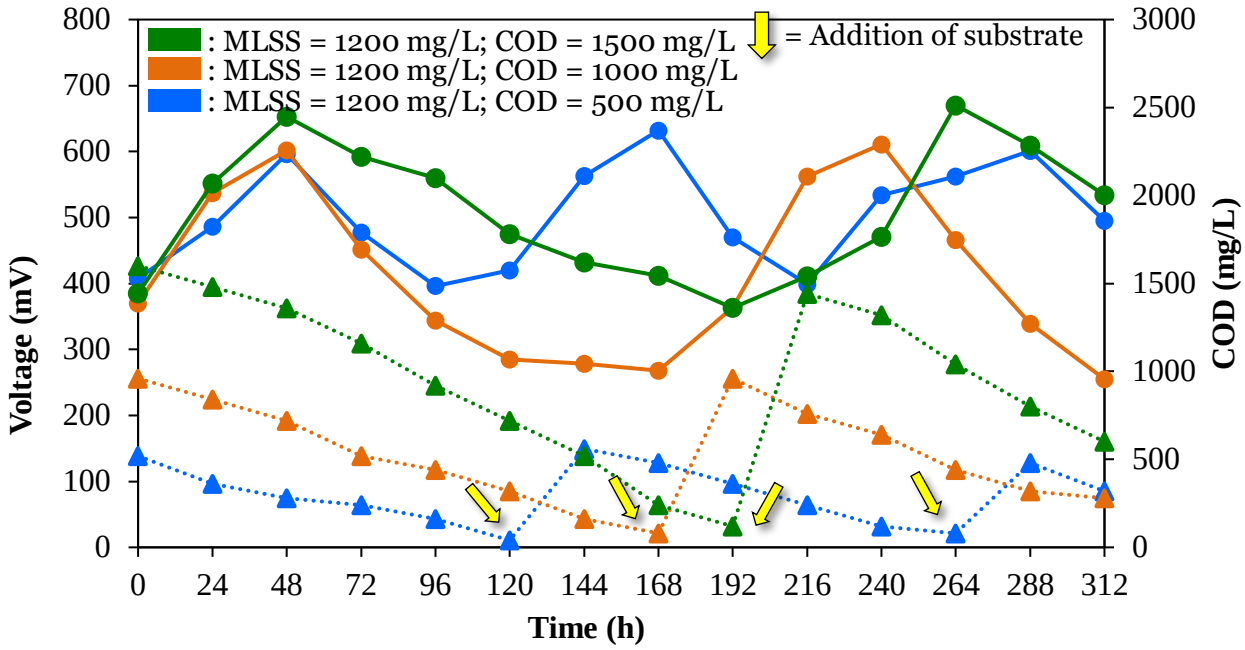

Figure 5(a). Voltage Production (—●—) and COD Concentration (···▲···) at Initial MLSS Concentration = 1.200 mg/L

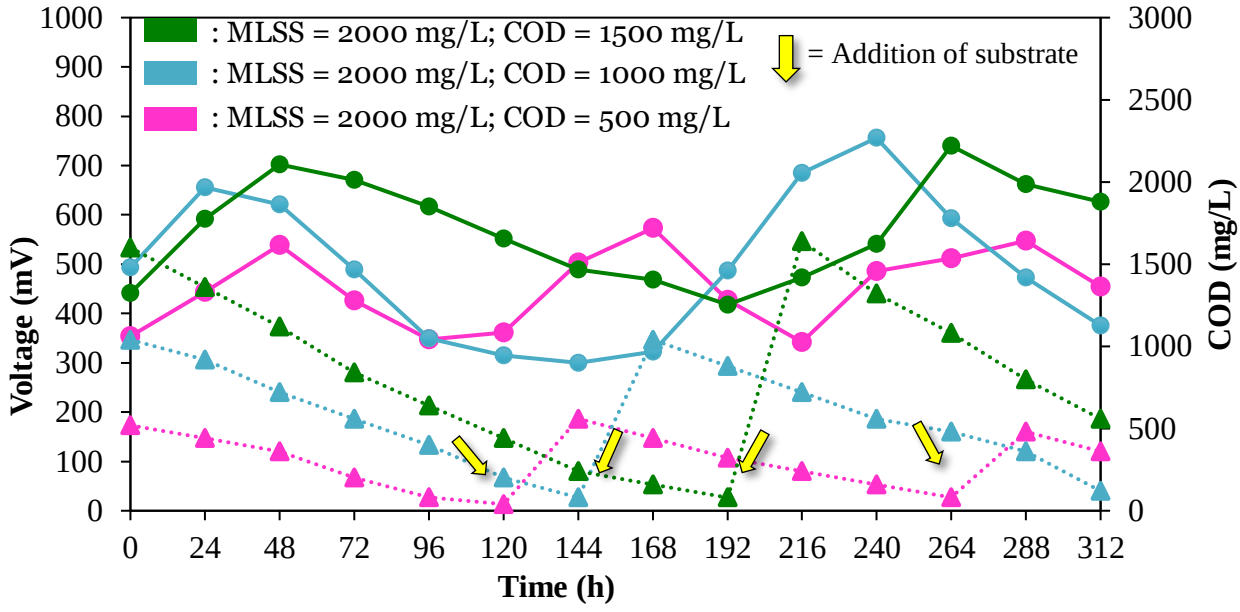

Figure 5(b). Voltage Production (—●—) and COD Concentration (···▲···) at Initial MLSS Concentration = 2.000 mg/L

Following Eq. 6, the reduction in the substrate concentration leads to a decrease in the mass of protons and electrons transferred to the cathode. This is also demonstrated in Fig. 5(a,b,c), where there

is a decrease in the generated voltage as the COD concentration decreases due to microbial metabolism. In this study, the highest electrical voltage was achieved at MLSS and initial COD concentrations of 3.500 and 1.500 mg/L, reaching 828 mV at 240 h. In this condition, the F/M ratio is at an optimal value, allowing for more electrons to be released and transferred to the cathode, resulting in higher voltage produced.

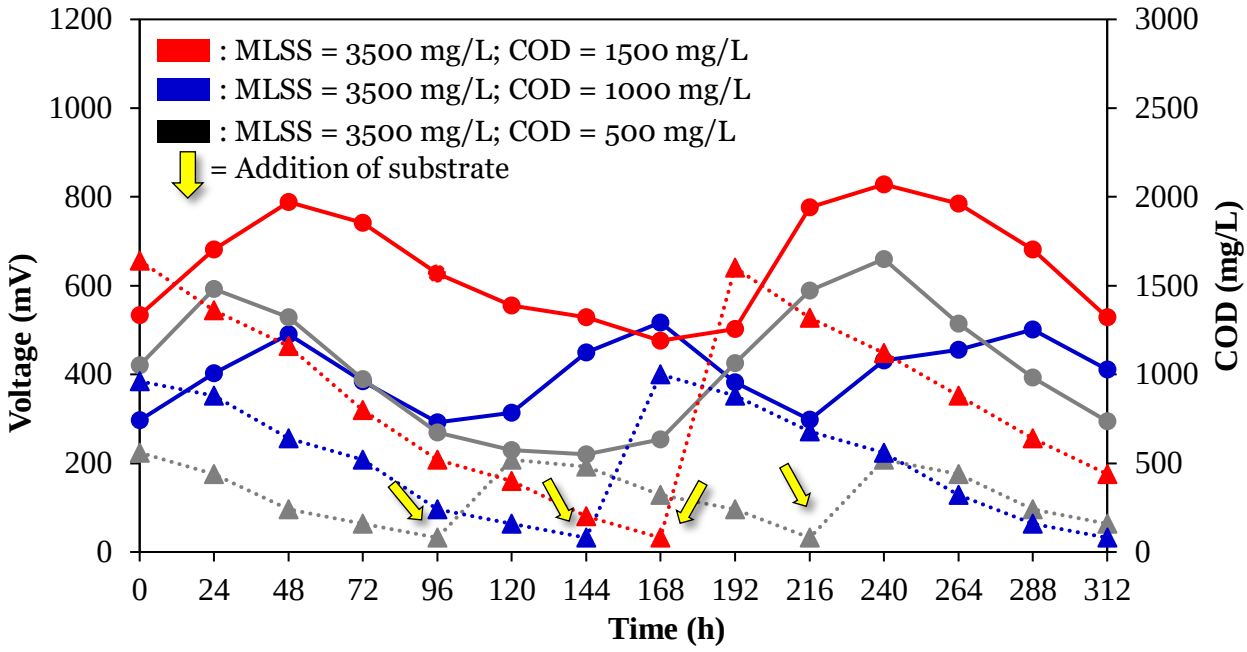

Figure 5(c). Voltage Production (—●—) and COD Concentration (···▲···) at Initial MLSS Concentration = 3.500 mg/L

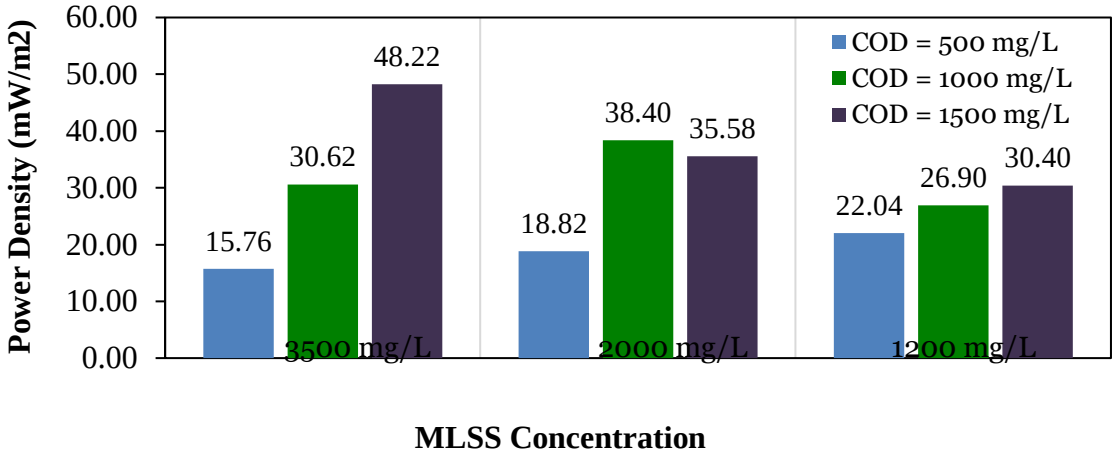

Figure 6. *Power Density* on Various COD and MLSS Concentration

Based on Figure 6, it can be observed that the highest peak power density was achieved at MLSS and initial COD concentrations of 3.500 and 1.500 mg/L, reaching 48,22 mW/m2. It was found that an increase of COD level generated higher power density especially at MLSS 1.200 and 3.500 mg/L. This trend was aligned with the Cr(VI) removal efficiency and voltage production, indicating that the MFC reactor operated at an optimal initial F/M ratio. On the other hand, the lowest peak power density occurred at MLSS and initial COD concentrations of 3.500 and 500 mg/L, respectively, reaching 18,69 mW/m^2. This might be resulted from high MLSS concentration which led to microbial population competition due to limited substrate availability, ultimately hindering overall electricity production processes (Alattabi et al., 2019).

CONCLUSION

Based on the results of this research, it can be concluded that the initial concentrations of MLSS and COD affect Cr(VI) removal efficiency and the power density produced. Higher initial concentrations of MLSS and COD generally resulted a higher Cr(VI) removal efficiency and power density. However, this condition tends to be effective when the F/M ratio is also at an optimal value. The highest Cr(VI) removal efficiency and power density achieved in this study were at initial MLSS and COD concentrations of 3.500 and 1.500 mg/L, respectively, which were 62,17% and 48,22 mW/m^2 over 312 hours. The optimal F/M ratio range was found to be 0,459-0,489 gCOD/gMLSS.

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