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Research Paper

Tofu Wastewater (TWW) Treatment and Hydrogen (H_2) Production by Using A Microbial Electrolysis Cell (MEC) System

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Abstract
High organic pollutant in tofu wastewater (TWW) raises a negative impact on environmental sustainability and health. Therefore, the TWW must be treated before it is discharged into the environment. Microbial electrolysis cell (MEC) is one of the green technologies that can be used to treat wastewater and generate hydrogen as well. This work tries to investigate the performance of MEC based on the decrement of organic pollutants in TWW. Some important parameters of organic pollutants in TWW such as chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS), total dissolved solids (TDS), total solid (TS), and pH were evaluated before and after MEC operation. The results showed that the COD and BOD levels decreased around 56% and 35% while pH increased from 7.90 to 7.16. Additionally, the TSS, TDS, and TS decreased by around 35.0%, 45.5%, and 33.2%. In addition, the optimum hydrogen yield (Y_{H2}) and hydrogen production rate (Q_{H2}) were obtained at 114 $\pm$ 0.1 mL H_2/g COD 360 $\pm$ 20 mL H_2/L/d. Overall, the MEC system could be used to reduce the level of organic pollutants in TWW and generated H_2 at the same time.

Keywords
TWW, Organic Pollutants, pH, COD, MEC

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1. INTRODUCTION

Nowadays, environmental sustainability become the main problem that has to solve immediately. There are two environmental issues namely excess of pollutant gases (i.e., CO_2, CO, SO_x, and NO_x) in the atmosphere and high amount of wastewater in the environment (Manisalidis et al., 2020; Igwe et al., 2017). Excess gases and high amounts of wastewater are caused by human activities, population and economic growth (Lin et al., 2022). It is well known that the use of fossil fuels in industry activities and vehicles increases air pollution over time (Lelieveld et al., 2019). In Indonesia (represented by Jakarta city), according to the air quality life index (AQLI) data that the quality of air in Indonesia since 2016 is getting worst (Greenstone and Fan, 2019; Istiqomah and Marleni, 2020). In fact, air quality index (AQI) in Indonesia is ranked 17th worst from 118 countries in the world for the period of 2021. Whereas, approximately 70% of wastewater is generated by households and the rest is produced by other sectors (industries, public facilities, office buildings, etc) (Igwe et al., 2017; Wulan et al., 2022).

As presented above, pollutant gasses and wastewater are two main issues currently focused on by researchers. The number of pollutant gasses can be eliminated by replacing fossil fuel with alternative fuel (i.e., H_2) (Cloete et al., 2022). In addition, the wastewater issue can be solved by performing chemical, physical, and/or biological approaches (Ye and Li, 2023; Gautam et al., 2022). Based on these environmental contexts, the authors suggest choosing the biological method to solve both gasses and wastewater issues. There are two biological methods such as fermentation and microbial electrosynthesis technologies (METs) which generally can be applied to treat wastewater and to generate biogas and/or biohydrogen and electricity (Son et al., 2021). Among these biological approaches, the METs are more interesting compared to the fermentation due to the METs have good efficiency and generate biogas.

One of the METs technologies is microbial electrolysis cells (MEC). Principally, MEC consists of two parts which are the anode and cathode side. The anode side converts the organic substrate to volatile fatty acids (VFAs) and electrons (e^-) (see Equation 1), while the cathode side generates hydrogen (H_2) from protons (H^+) and electrons (see Equation 2). Due to the hydrogen production being unspontaneous reactions so the additional energy must be

supplied into the MEC system. Generally, the amount of additional energy in the MEC systems is required in the range of 0.20 - 0.80 V (Call and Logan, 2008).

Anode: Organic substrate $CH_3COO^- + 4H_2O \rightarrow 2HCO_3^- + 9H^+$

Cathode: $8H^+ + 8e^- \rightarrow 4H_2$

Overall: $CH_3COO^- + 4H_2O \rightarrow 2HCO_3^- + H^+ + 4H_2$

Theoretically, the performance of MEC depends on some parameters such as anode and cathode performance, type and characteristics of substrates, and amount of additional energy input into the system (Apostolopoulos et al., 2021). The substrate with a high simple organic acid, rich nutrient, and neutral pH are very appropriate for generating H_2. In general, the additional energy at the optimum condition is obtained at approximately 0.80 V (Meshram and Jadhav, 2017). This work tries to investigate the performance of f MEC system based on the TWW quality and hydrogen production when using TWW as substrate. Some parameters such as the change of pH, COD, BOD, TSS, H_2 yield, and H_2 production rate are evaluated.

2. EXPERIMENTAL SECTION

2.1 Materials

Materials such as acrylic blocks and cation exchange membrane (CEM, CMI 7000s) were obtained from Fuel Cell Institute Laboratory (FCIL) University Kebangsaan Malaysia (UKM). Whereas, all chemicals were purchased from online shops. Some chemicals needed in this work were hydrochloric acid (HCl, analytical grade), sodium acetate (CH_3COO-Na, 99%), potassium chloride (KCl, analytical grade), ammonium chloride (NH_4Cl, analytical grade), potassium dihydrogen phosphate (KH_2PO_4, analytical grade) and di-potassium hydrogen phosphate (K_2HPO_4, analytical grade), ethanol (C_2H_5OH, 99.5%) and deionized water (DW).

The spectrophotometer (Thermo-scientific) was used to determine the chemical oxygen demand (COD). The pH tester (H198103, Hanna Instruments) was used to measure the initial and final pH of TWW. The power supply (YIHUA 1502 DD, DC power supply) was used to supply the additional voltage to the MEC system. The scanning electron microscope and electron dispersive X-ray (SEM-EDX, JEOL JSM 5800) was used to characterize the electrode surfaces before and after the MEC system run.

2.2 Methods

2.2.1 Tofu Wastewater (TWW) Preparation and Identification

A total of 10 liters of TWW sample was collected from the tofu home industry nearby Imogiri Barat. Some physicochemical parameters such as pH, chemical oxygen demand (COD), biological oxygen demand (BOD) and total suspended solid (TSS), total dissolved solids (TDS), and total solid (TS) were measured by using APHA, Winkler, and Spectroscopy methods, respectively. The characterizations of fresh TWW were presented in Table 1. Initially, the

Table 1. Characteristic of TWW Sample

Parameters	Average Values	Unit
pH	7.90	-
TDS	4546.67	mg/L
TS	7920.00	mg/L
TSS	1903.33	mg/L
BOD	1400.10	mg/L
COD	1066.67	mg/L

TWW sample was filtered by using a filter cloth to separate the solution and active sludge before it was used in the MEC system. Then, a filtered solution of TWW was kept and used as a substrate for further work. Meanwhile, the active sludge of TWW was used as a mixed-culture of electroactive bacteria (EAB) source in the anode before the MEC system was operated. It was well known that the EAB must previously be attached and enriched at the anode to enhance the system performance (Satar et al., 2021).

2.2.2 Preparation of Electrodes

The preparation of the electrode was addressed to remove the physical impurities on the material surface. The electrode preparation was performed in acid and alkali solutions as described by Satar et al. (2021). The Electrodes (both anode and cathode) were fabricated from the graphite foam (GF) materials with a configuration of 2.5 cm × 2.5 cm (Figure 1). Before it was used in the MEC system, the electrodes were immersed in the 250 mL HCl (1 M) for 1 hour and washed using deionized water (DW) three times. Then, the electrodes were immersed in 250 mL KOH (1 M) for 1 hour and washed using DW three times. Lastly, the electrode materials were dried in a vacuum oven at 80 ^oC overnight (Satar et al., 2018).

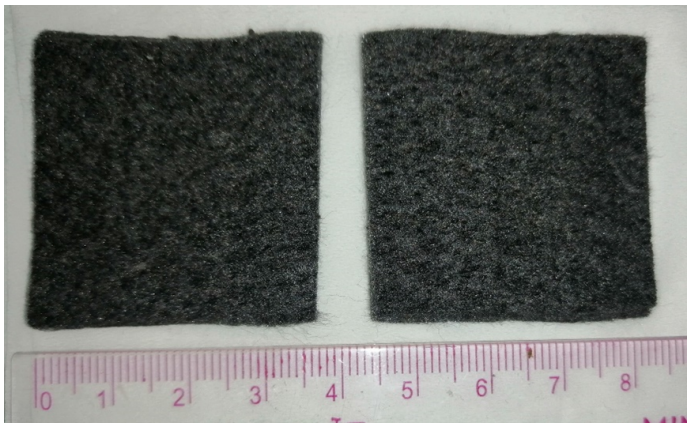

Figure 1. Graphite Foam Electrodes; Anode (Left) and Cathode (Right) Materials

2.2.3 Dual Chamber MEC Set-up and Operation

The MEC system was designed in the dual chamber types which were fabricated from acrylic block materials. The MEC system was divided into the anode and cathode chambers with an active volume of 50 mL, respectively (Satar et al., 2020a). Both anode and cathode chambers were separated by CEM (CMI, 7000s) to prevent a short circuit in the system. The anode and cathode materials were installed with the iron sheet to facilitate the current transfer from a power supply to the system. The schematic and photograph of the MEC system were presented in Figure 2.

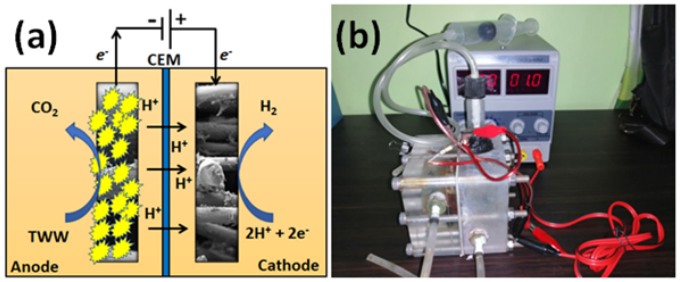

Figure 2. The Design of the MEC System; (a) Schematic and (b) Photograph of Dual Chamber MEC System

The MEC system was operated at room temperature and fully maintained in anaerobic condition to avoid contamination on EAB at the anode and hydrogen gas (H_2) release from the cathode (Bora et al., 2022). Before the MEC was operated, the anode was initially enriched with EAB using active sludge of TWW while the cathode was without chemical catalysts. During the enrichment process, the 1.0 g/L CH_3COONa solution was used as substrate at the anode while 1 M KCl as anolyte at the cathode. A direct current power supply (YIHUA 1502 DD) was needed to supply 1.0 V of additional energy into the system because hydrogen production is an unspontaneous reaction. An external resistor (R = 1.0 ohm) was installed at the MEC system to convert a voltage (V) from the power supply to current (I). Once the H_2 gas was produced from the system, the CH_3COONa substrate in the anode was replaced with TWW solution. The operation of the MEC system was stopped when the H_2 was stably produced in three consecutive runs.

2.2.4 Gas Analysis and Calculation

The generated H_2 gas was collected using a 25 mL syringe for three consecutive cycles. The amount of generated H_2 was calculated based on the purity of H_2 production by using dual chamber MEC (69–77%). The n_{H2} is a mole of H_2 that is calculated based on the ideal gas formula at standard conditions (Equation 1). The M_{H2} is the molecular mass of H_2 (2 g/mol). The COD removal (ΔCOD) is calculated based on the total COD value at the beginning (COD_0) minus the COD value at the final (CODt). The H_2 yield (Y_{H2}) and H_2 production rate (Q_{H2}) were calculated by using Equation 2 and Equation 3, respectively (Satar et al., 2020b).

Table 2. Summary of TWW Characterization at the Final of MEC Run

Parameters	Average Values	Standard (Peraturan Daerah DIY, 2016)
pH*	7.16 ± 0.1	6.0 – 9.0
TDS	2476.67 mg/L	-
TS	5293.33 mg/L	-
TSS	1236.67 mg/L	200 mg/L
BOD	490.04 mg/L	150 mg/L
COD	469.33 mg/L	300 mg/L

Note: * = the pH of TWW after the MEC system run

$$n_{H2} = \frac{PV}{RT} = \frac{V_{H2}}{22.4L} \quad (1)$$

$$Y_{H2} = \frac{n_{H2} \times M_{H2}}{V_l \times \Delta COD} \times 100\% \quad (2)$$

$$Q_{H2} = \frac{V_{H2}}{V_A \times t} \quad (3)$$

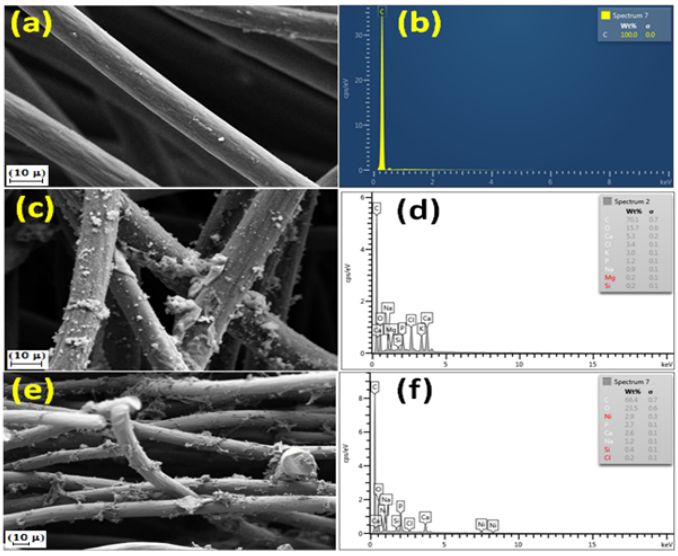

Figure 3. Images of Surface Morphologies of Electrode and Element Compositions: (a) Electrode Image, (b) EDX Spectra Data Before the MEC System Run, (c) Anode and (e) Cathode Morphologies, (d) EDX Spectra of the Anode and (f) EDX Spectra of Cathode After the MEC System Run

3. RESULTS AND DISCUSSION

3.1 Electrode Images Before and After the Experimental Run

The surface morphologies and element composition of electrodes before and after the MEC system run were presented in Figure 3. Either anode or cathode surfaces before the MEC system run was observed clean as shown in the typical image of GF surface (Figure 3(a)). Based on the SEM-EDX results, the main element on the electrode surface was carbon (C) with 100% of relative composition (Figure 3(b)). Meanwhile, the morphologies of anode and cathode surfaces after the experiment run were presented in Figure 3(b) and (d) respectively. The C compositions at the anode and cathode surfaces were observed to be reduced to 70.1% (Figure 3(d)) and 66.4% (Figure 3(f)), respectively. These facts might due to the presence of other chemical components or impurities at the anode and cathode surfaces as a result of the oxidation and reduction reactions at their surfaces. Furthermore, the MEC systems were continuously supplied with the additional voltage from a power supply, thus the oxidation and reduction reactions occur during the MEC system operation.

According to some references, the presence of impurities on the electrode surface inhibits the bio-electrochemical reaction due to the current flow being slow (Apostolopoulos et al., 2021; Satar et al., 2020b; Carmona-Martínez et al., 2015). Consequently, the MEC performance gradually dropped after three months of the system operation (Carmona-Martínez et al., 2015). Therefore, the anode and or cathode of the MEC system is generally replaced with a new one before it is used again.

3.2 TWW Characterization

The quality of TWW before and after being treated with the MEC system was evaluated to ensure whether the characteristics of TWW meet the national standard of Indonesia or not. The national standard of Indonesia concerning the TWW quality regulated by Minister of Environment of the Republic of Indonesia No. 5, 2014 was referred to in this work. Some parameters of fresh TWW such as pH, COD, BOD, TSS, TDS, and TS were detailly measured (Table 1). Based on Table 1 the pH of fresh TWW was observed around 7.90. According to the Regulation of Government of Yogyakarta Special Region, No. 7, 2016, concerning the wastewater standard (Peraturan Daerah DIY, 2016), the pH of wastewater must be around 6.0 – 9.0 before discharge to the environment. Based on this pH condition indicates the fresh TWW can be discharged into the environment. It is well known the low pH of wastewater cause a negative impact on the pH of the soil, consequently, the nutrition absorption by plants is inhibited (Rachmawati and Wardiyati, 2018). Furthermore, the low-pH wastewater may also interfere the marine life and aquatic biota (Xu et al., 2023). Hence, the fresh TWW must be primarily treated before it discharges to the environment. In this work, the pH of fresh TWW was treated by using the MEC system. The final pH of TWW was quite decreased to 7.16. This fact indicates the TWW still be safely discharged into the environment. Overall, the characterization of TWW at the final MEC run was presented in Table 2.

In addition to pH, the TSS, TDS, and TS were evaluated to ensure the physicochemical properties of TWW meet with the Regulation of Government of Yogyakarta Special Region No 7 2016. The physical properties of TSS, TDS, and TS involve the presence of solid impurities in TWW. The high number of solid impurities in TWW inhibits sunlight and oxygen entry into the water, consequently, marine life and or aquatic biota are disrupted. In this work, the TSS, TDS, and TS of the treated TWW decreased around 35.0%, 45.5%, and 33.2% respectively. Due to the TSS, TDS and TS were higher than the TS standard (200 mg/L), so further treatment must be conducted. For example, the filtration process can be used to reduce these solid impurities.

Furthermore, the COD value is principally indicated the number of organic matters that exist in a sample. In addition, the high BOD value indicates a low number of dissolved oxygen (DO) exists in the sample. Based on two conditions which are the high COD and BOD values harm the environment. This work shows the BOD and COD values of TWW were changed after it was treated with the MEC system. The BOD was decreased from 1400.10 mg/L to 490.0 mg/L (decrement of 35.0%), while the COD value was decreased from 1066.67 mg/L to 469.3 mg/L (decrement of 56.0%). The presence of EAB at the anode of MEC consumes the organic matter of TWW during the MEC operation. However, the final BOD and COD values were quite high than the regulation, hence, the TWW must be re-treated more either chemical, biological or physical method. The high COD and BOD values might be due to the low performance of the anode. The performance anode is very associated with the ability of EAB to consume the organic pollutants. In this work, the MEC system was only operated under normal conditions, consequently, the EABs might be poisoned by oxygen so their abilities were low. Therefore, the MEC system must fully be maintained at anaerobic conditions.

3.3 Biohydrogen Production and MEC Performance

The phenomenon of the current trend at the enrichment and steady-state stages of the MEC system run was shown in Figure 4. At the enrichment stage in the MEC anode, the generated current was observed very low (0.02 - 0.03 mA) for more than 20 days. There were very low gases produced from the system at the enrichment stage. Whereas, the current production at the steady state was observed higher (0.4 ± 0.1 mA) for 10 days. However, the current generation was low due to there were no catalysts used at the cathode of MEC, so the Y_{H2} and Q_{H2} were quite low. Therefore, the chemical and or biological catalysts must be applied onto the cathode surface to enhance the H_2 evolution reaction (HER).

Table 3. Summary of H_2 Production from Dual Chamber MEC Using TWW Substrate and Selected References

Parameters	MEC Design			
	Dual Chamber	Dual Chamber	Dual Chamber	Dual Chamber
Anode-cathode	GF-GF	GF-GF/Pt	GF-CC/SSW	GF-Ni 201
Substrate	TWW	EGF	EGF	Acetate
E_{ap} (V)	1.0	1.0	1.0	0.9
Y_{H2} (mL H_2/gCOD)	114.1 ± 0.1	553.6 ± 53.5	330 ± 30	-
Q_{H2} (mL/L/d)	360 ± 20	590 ± 10	520	380 ± 40
References	This work	(Satar et al., 2020b)	(Ribot-Llobet et al., 2013)	(Selembo et al., 2009a)

Note: CC/SSW = carbon cloth combined with stainless wool; GF = graphite foam; GF/Pt = graphite coated with Pt catalyst; Ni 201 = nickel allow 201; EGF = effluent of glucose fermentation.

Principally, the overpotential at the cathode is high, hence, the catalysts are required to increase the total amount of H_2 production (Park et al., 2022).

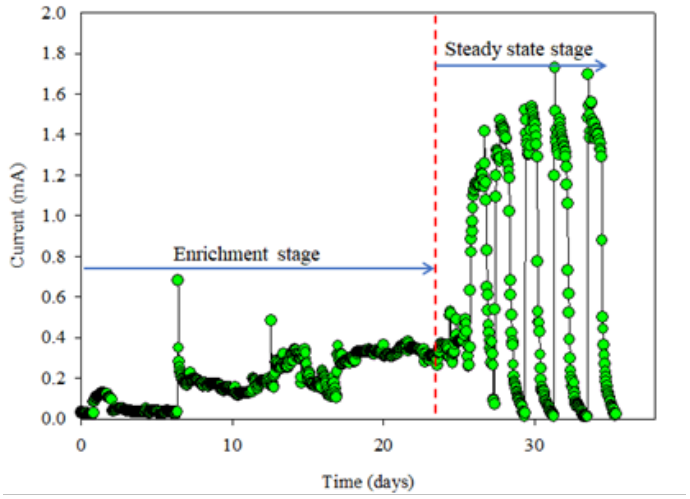

Figure 4. Typical Current Generation During H_2 Generation for 36 Days of the Experiment Run

Hydrogen (H_2) produced from the MEC system was measured by using a syringe (50 mL) without further GC analysis. Based on some literature the purity of H_2 produced from the dual-chamber MEC generally reached around 69-77% (Satar et al., 2020b; Chae et al., 2010; Selembo et al., 2009b; Kadier et al., 2018). Based on these references, hence, the purity of H_2 produced in this work can also be assumed around 70%. After three consecutive cycles of biogas production, the total amount of gas volume was obtained at 68.1 ± 2.0 mL (or 48.1 mL of assumed H_2) for 24 hours. The COD removal (ΔCOD) along with the system operation was obtained at 597 ± 10 mg/L. According to Equation 1, the Y_{H2} was calculated at around 8.87% (or 114.1 mL H_2/g COD). Meanwhile, the Q_{H2} was calculated at around 360 ± 20 mL/L/d. This work demonstrated that the H_2 can be generated by a dual chamber MEC system using TWW as substrate. However, the performance of MEC (without cathode catalyst) using TWW as substrate was quite lower than that of the selected references in Table 3. As mentioned above this issue might be due to the rate of H^+ reduction reaction to H_2 at the cathode being quite slow.

4. CONCLUSIONS

The dual chamber MEC system was successfully applied to treat TWW and generated H_2 at the same time. The TSS, TDS, TS, COD, and BOD values of the final TWW were reduced by approximately 45.5%, 35.0%, 33.2%, 56%, and 35%, respectively, lower than that of fresh TWW. Meanwhile, the pH decreased from 7.90 to 7.16. In addition, the Y_{H2} and Q_{H2} were obtained at approximately 8.87% (99.5 ± 0.1 mL H_2/g COD) and 360 ± 20 mL/L/d, respectively. Due to there being no catalyst on the cathode so the performance of dual chamber MEC was lower than that of the selected reference. In the next research, the cathode of the dual chamber MEC system will be coated with biological and or chemical catalysts to improve the Y_{H2} and Q_{H2}.

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