Anoxic-Aerobic Microbial Fuel Cell in Treating Spent Caustic Wastewater: Varies in Electrode Pairs

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Abstract—In this study, the performance of a continuous fed anoxic-aerobic Microbial Fuel Cell (MFC) reactor operated with spent caustic wastewater was investigated by varying the electrode pairs of different cathode materials. Three types of electrode pairs tested were carbon-carbon (C-C), carbon-aluminium (C-Al) and carbon-copper (C-Cu). Spent caustic wastewater is the industrial wastewater with high COD concentration influenced by its high sulfur content, high salinity and high alkalinity. Little is known on the capacity of MFC to treat spent caustic wastewater, therefore the present study employed spent caustic wastewater as the feed wastewater. The performance of MFC with different electrode pairs was evaluated in terms of the voltage production, Chemical Oxygen Demand (COD) and sulfide removal efficiency. From the study, C-C electrode pairs was the best electrode pairs for the MFC operation with the highest voltage production of 189.1 mV and highest COD and sulfide removal effiaciency of 89.1% and 99.91% respectively.

Keywords—Microbial Fuel Cell; spent caustic wastewater; electrode; chemical oxygen demand; wastewater treatment; voltage production.

1. INTRODUCTION

Spent caustic wastewater is the industrial wastewater that are mainly discharged from the refineries and petroleum chemical plants [1]. Spent caustic wastewater is named after the wasted or used caustic soda. Caustic soda is the sodium hydroxide solution that contains 5-12 wt% sodium hydroxide, NaOH and 0.1- 4wt % sulphide, S^{2-} and can be characterized according to their origin and composition [2,1]. Caustic soda serves as the scrubbing agent in the desulphurisation process to remove different gases including hydrogen sulfide and carbon dioxide from different hydrocarbon streams [3]. During the caustic scrubbing process, hazardous gaseous react with the sodium hydroxide solutions and the hydrogen sulfide and thiols contaminants are then absorbed producing a waste solution known as the spent caustic [4,1]. There are a few existing treatments available to treat spent caustic wastewater. Most of the methods used in treating spent caustic wastewater were the physicochemical methods that could give incomplete oxidation and are unsustainable along with high chemical consumption, high pressure and temperature used. Therefore, the present study is focusing on the biological process to produce an effective spent caustic wastewater treatment process.

Treating wastewater by using the biological approach can be a great challenge especially in treating spent caustic wastewater. MFC is one of the biological treatment method available to treat spent caustic wastewater. MFC is a bioreactor that uses microorganisms as the biocatalysts which can convert the biomass into bioenergy [5]. The presence of the microorganisms in the MFC reactor degrades the pollutants in the wastewater while generating electrons and protons. The transfer of the generated electrons and protons from the anode to the cathode side of the reactor enables the system to produce electricity. Therefore, both of the wastewater and energy recovery could be achieved simultaneously by using MFC treatment method. MFC treatment method is reported to be still in the development stage in which futher study is required for the treatment method to be implemented for industrial use. MFC has been tested on various source of substrates such as MFC performance has been tested on various types of wastewater such as

domestic wastewater [6,7,8], swine wastewater [9], agro food wastewater [10], artificial wastewater [11], synthetic wastewater [12,13,14], fruit processing wastewater [15], tannery wastewater [16], brewery wastewater [17] and etc. There was a study conducted by Srikanth et al. (2016) on MFC electro-biocatalytic treatment operated with petroleum refinery wastewater and the system is reported to produce power density of 225 mW/m^2 and was also able to achieve 84.4% substrate degradation when being operated in continuous mode and 81% when being operated in batch mode. MFCs study were rarely tested on spent caustic wastewater and although a little is known on the capacity of MFC to treat spent caustic wastewater, the study conducted by Srikanth et al. (2016) has demonstrated MFC as the potential method in treating spent caustic wastewater.

MFC has gained the interests of many researchers due to its significant advantages such as a non-pollution wastewater treatment technology, energy benefited and mild operating conditions [18]. However, despites its significant advantages, MFC also has major drawback which hindered MFC from the industrial implementation. Most often MFC was reported to produce low energy production that could not meet the energy-neutral operation at practical scale [19,20]. MFC technology is still in the developing stage whereby its application is limited to lab scale and its transfer to industrial operational scale would involve high capital cost [21]. With the aim to overcome MFC limitations, some researchers attempted to optimize MFC in terms of its configuration such as MFC was commonly stacked together in series or in parallel [22,23,19,24,25]. Oh and Logan (2007) reported that stacking multiple MFCs together in series would create problems such as voltage reversal, contact voltage losses and erratic operation [26]. Other feasible alternatives used to optimize MFC electrode materials and configurations whereby electrodes were also commonly stacked for higher voltage output and MFC scaling-up purpose [27,28,29].Continuous investigations on MFC optimization should be conducted to contribute towards MFC development.

Generally, an MFC configuration is made up of two separated chambers known as anode and cathode chamber. Both of these chambers are connected by a proton exchange membrane (PEM) or salt bridge to allow the transfer of electrons [30]. Anode is the place for oxidation of organic material occur whereas cathode serves as the place for oxygen reduction to form water [31] . Tamilarasan et al. (2016) reported that the MFC performance is influenced by four processes namely microbial catabolism, electron transfer from the microbes to the anode, reduction of the electron acceptors at the cathode and proton transfer from the anode to the cathode [32]. The factors mentioned are associated with the type of electrode material used. According to Sangeeta and Muthukumar. (2012), the MFC performance is dependent on the electrode material used, as it must be conductive, biocompatible and chemically stable in the reactor solution [33]. In the present study, the single chamber anoxic-aerobic MFC reactor was investigated by varying the electrode pairs of different cathode materials in order to obtain the optimum MFC operation in treating spent caustic wastewater and energy production.

2. MATERIALS AND METHOD

A. Wastewater preparation

Spent caustic wastewater was collected from a petrochemical industry located in Gebeng, Kuantan. Firstly, any solid particles or any unwanted waste will be filtered and removed from the wastewater obtained. Then, the wastewater is neutralized with diluted sulphuric acid to pH 7.0. It is reported that microorganisms can generally adapt wide range of ambient pH, however most of bacteria favor neutral pH conditions for their optimal growth [34]. The COD and sulfide concentration of the wastewater was then adjusted to the range of 400-500 mg/L and 80- 100 µg/L respectively by wastewater dilution. The adjustment was made in order to create influent that have characteristic for biological treatment. According to Sipma et al. (2004), the waste to be used for biological purpose need to be applied with dilution factors up to three in order to reduce the pH and sodium level down to an acceptable concentration for neutrophilic sulfide- oxidizing bacteria [35]. Sodium acetate was added as the source of additional nutrients for bacteria [36]. Table 1 summarizes the charactherization of the prepared wastewater.

Table 1: Characterization of prepared wastewater

Parameter	**Values**
pH (mg/L)	7
Nitrate (mg/L)	14.6
Phosphate (mg/L)	0.4

Parameter	Values
Sulphate (mg/L)	66
Sulfide (mg/L)	8.0
Chemical Oxygen Demand (mg/L)	400

B. Acclimatization

Acclimatization is a pre-treatment process which is required in order to allow adaption of bacteria to the new environment [37]. The acclimatization process were conducted by diluting the biomass with water to new mixed liquor suspended solid (MLSS) concentration (1.5 to 3.0 g/L). The observation on the survival of the microorganisms to the new environment was indicated based on the COD removal and mixed liquor volatile suspended liquid (MLVSS) reaching the constant value [38]. In this study, the acclimatization period ended when the MLVSS/MLSS ratio of the sludge is more than 60% which indicates the sludge is able to perform well in the new environment. The sludge was ready to be fed to the reactor when it achieves 80% of COD removal and shows a constant MLVSS value.

C. Electrode preparation

For the study on the effects of type of electrode material used on MFC, there were three sets of electrode materials prepared. Carbon electrode was employed as the anode material in all three sets of electrode combination due to its surface morphology. It is reported that carbon electrode surface structure could provide more microbial attachment than the other type of electrode materials. The microbial attachment at anode is crucial as according to Scott et al. (2007), special microorganisms enriched on the anode electrode play an important role in consuming organics and producing electrons and protons [39]. Therefore, adopting carbon material as the anode of the MFC reactor could encourage the presence of high population of special microorganisms to degrade more pollutants which would be resulting in a higher efficiency of wastewater treatment and energy recovery. Table 2 shows the sets of electrode combinations prepared for the study.

Table 2: Set of electrodes

Set	Anode	Cathode	Area (cm^2)
1	Carbon	Carbon	900
2	Carbon	Copper	900
3	Carbon	Aluminium	900

D. MFC operation

An anoxic aerobic MFC reactor was used with an effective volume of 4 liter. The MFC reactor was inoculated with aerobic digestor sludge and was operated in a continuous mode. Two pumps were set at the anode and cathode chamber to pump the wastewater into the cell and transfer the treated wastewater for settlement before undergoing analysis. Both anode and cathode chambers were separated by using a baffle. The anode and cathode electrodes were connected by using copper wire to form a circuit. Magnetic stirrer has been used to ensure no sludge sediment at the bottom of the reactor. Figure 1 shows the MFC setup. A multimeter was used to measure the voltage generated by this system. The experiment conducted were maintained at solid retention time (SRT) and hydraulic retention time (HRT) of 20 days.

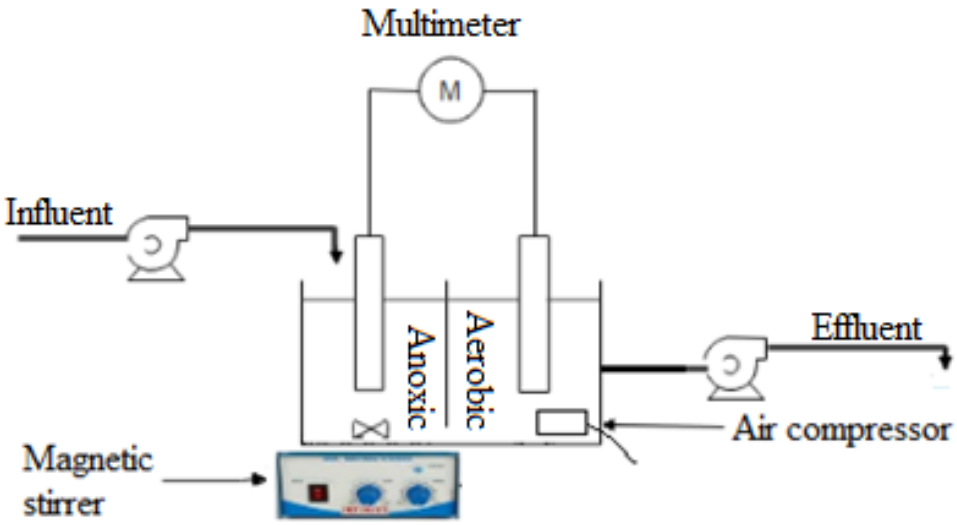

Figure 1: MFC Setup

3. RESULTS AND DISCUSSION

A. COD and Sulfide Removal Efficiency with different type of Electrode Pairs

The anoxic-aerobic MFC reactor in treating spent caustic wastewater was operated by varying the electrode pairs of different cathode materials. The electrode pairs tested were carbon-carbon (C-C), carbon-copper (C-Cu) electrode and carbon-aluminium (C-Al) electrode. The wastewater treatment efficiency was evaluated in terms of its COD and sulfide removal efficiency. Figure 2 shows the COD and sulfide removal of the anoxic-aerobic MFC reactor operated with different electrode pairs. The COD and sulfide concentration of the influent wastewater were 402 mg/L and 8 mg/L respectively.

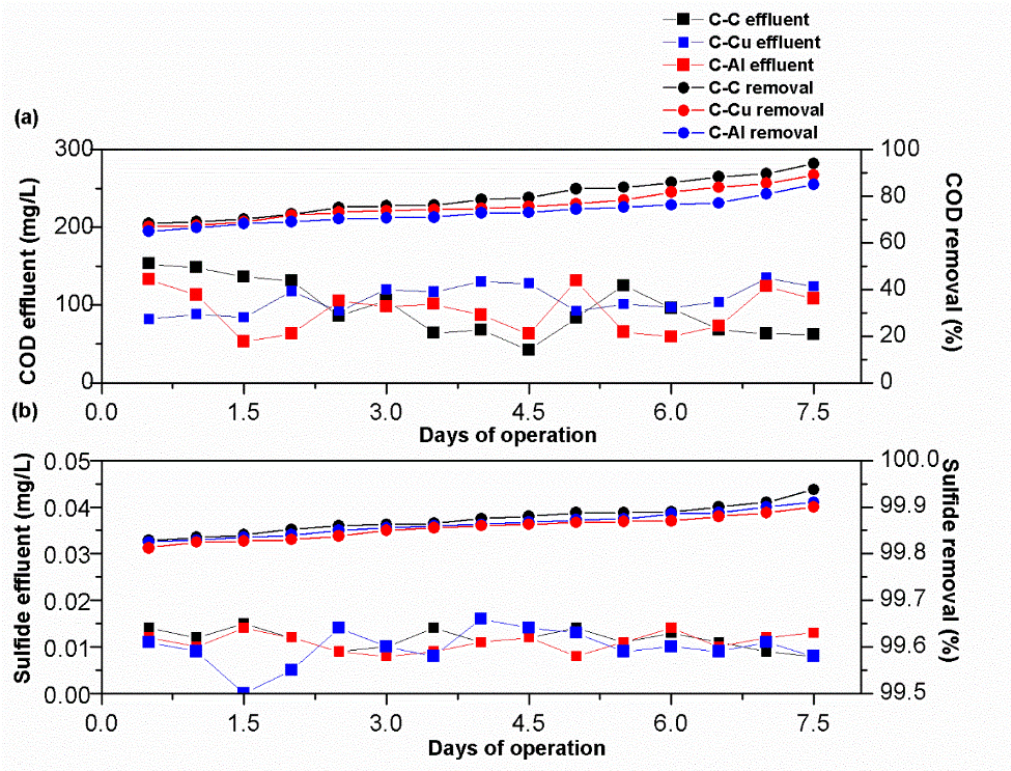

Figure 2: a) Concentration of COD and COD removal efficiency versus days of operation. b) Sulfide concentration and sulfide removal efficiency versus days of operation.

Based on Figure 2, it is shown that the C-C electrode gives the highest COD and sulfide removal efficiency of 94.1% and 99.93% respectively making C-C electrode as the best electrode material in the MFC operation, followed by C-Cu electrode and C-Al electrode. The highest COD and sulfide removal efficiency achieved by C-Cu electrode was 89.1% and 99.91% respectively. Whereas, the highest achievable COD and sulfide removal efficiency for C-Al electrode was 85% and 99.90% respectively. Significant reduction of COD and sulfide concentration was observed in all three sets of electrode pairs of the MFCs operation when all of the MFC operations achieved the removal efficiency of higher than 80%. The good wastewater treatment efficiency achieved in all MFCs operation might be due to the employment of the carbon electrode as the anode in all three electrode pairs. Scanning Electron Microscopy (SEM) analysis were conducted to observe the surface morphology of the three types of the electrode materials used. As observed in Figure 3, carbon material apparently shows higher surface roughness compared to other types of electrode materials. There were studies carried out to investigate the relationship of microbial attachment and surface roughness in which it is reported that higher surface roughness led to a better bacteria adhesion in which resulted in more microbial attachment bacterial growth [40,41]. The findings could explain the formation biofilm at the anode electrode of all three MFCs operation.

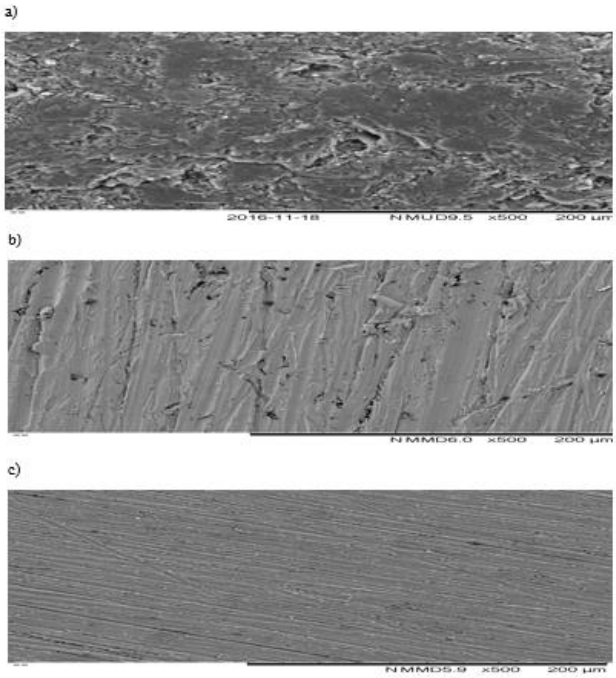

Figure 3: a) SEM analysis of carbon electrode. b) SEM analysis of copper electrode. c) SEM analysis of aluminium electrode.

The presence of the microorganisms attached at the anode electrode can be observed in Figure 4. The microorganisms attached at the anode electrode might consists of electrogenic and non- electrogenic bacteria, and both types of bacteria involved in the COD consumption. The presence of these bacteria have led to a higher COD consumption with higher pollutants degradations. Thus, less COD and sulfide concentration of the effluent of the MFC operations were observed and resulted in achieving COD and sulfide removal of higher than 80% efficiency in all MFC operations. Apparently, the electrode at the anode serve not only for the exchange of electrons to take place but also as the supporting material for the culture of the microorganisms [42]. It is reported that carbonaceous material as the anode material is an excellent alternative to improve MFC wastewater treatment efficiency as this material possess good conductivity and well suited for the bacterial growth [8,43].

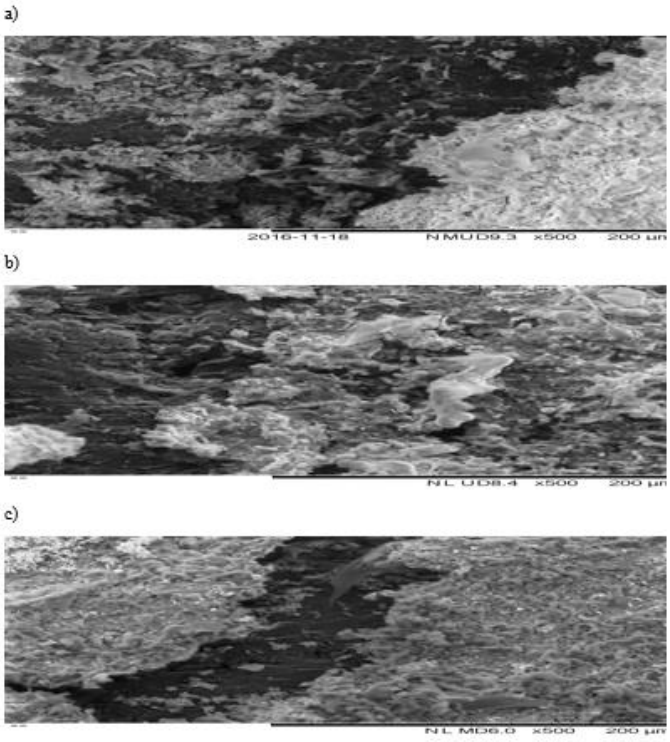

Figure 4: a) SEM analysis of anode (carbon) after MFC operation by using C-C electrode pair. b) SEM analysis of anode (carbon) after MFC operation by using C-Cu electrode pair. c) SEM analysis of anode (carbon) after C-Al electrode pair.

In the present study, it is demonstrated that C-C electrode was the best electrode pairs among the three pairs of electrode combinations as it produced the highest COD and sulfide removal efficiency. This shows that the cathode material does influence the performance of MFC. The cathode compartment of an MFC operation is the place where the reduction of oxygen to water takes place. The oxygen reduction reaction could be catalyzed by the cathodic biofilms. In a research conducted by Martin et al. [44], it is assumed that the oxygen reduction reaction takes place at the material surfaces. Thus, in this study, the excellence performance of C-C electrode pairs was due to higher biofilm attachment at the cathode in which the cathodic reduction reaction is affected by it. Higher cathodic biofilm led to a better biocatalytic activity which helps to improve the cathodic reaction [44]. The result is compatible with previous research conducted by Sangeeta and Muthukumar [33] which reported that carbon electrode is the best electrode material among stainless steel, aluminium and iron electrode in wastewater treatment [33]. A study on electrochemical process using different types of electrode material also reported that although graphite is limited by its conductive quality, graphite electrode is highly effective for COD, SS and nutrients removal for dischargeable wastewater with enough reaction time [31].

Besides that, it can also be observed that the performance of C-Cu and C-Al electrode pairs were slightly lower than C-C electrode pairs. Copper and aluminium might be good in conductivity however, both electrodes are metals in which making them not to be applicable in MFCs since metal material could possessed toxicity of trace material ions to the bacteria. According to Tekle and Demeke. [45], metal electrode consists of non-corrosive stainless steel mesh can be utilized, however copper is not suggested due to its toxicity to bacteria [45]. The SEM analysis of the cathode materials is shown in Figure 5. The deficiency of copper material as cathode material is due to its lower population of active bacteria at the cathodic biofilms causing the system to encounter lower biocatalytic activity compared to carbon cathode material. Based on this experiment, carbon material has been found as the best cathode material in MFC operation. Akarsu et al. [31] has also reported that although graphite has a limited conductive property, graphite electrodes are still highly effective for COD, SS and nutrients removal with enough reaction time [31]. C-C electrode material might be the best electrode material in the COD and nutrients removal efficiency due to high oxidizing power of catalytic species at its electrode surface, suitability for microbial attachment and its non-corrosive property.

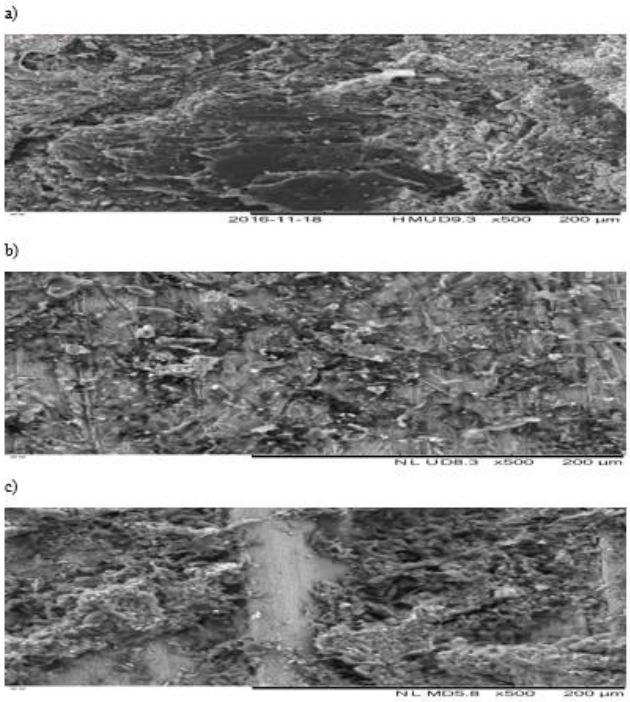

Figure 5: a) SEM analysis of cathode (carbon) after MFC operation by using C-C electrode pair. b) SEM analysis of cathode (copper) after MFC operation by using C-Cu electrode pair. c) SEM analysis of cathode (aluminium) after MFC operation by using C-Al electrode pair.

3.2. Effects of Electrodes on Energy Production

The output voltage of the MFC operation were measured. The data was recorded and the performance of MFC in terms of the energy production had been assessed. Figure 6 shows the output voltage of the MFC operation of different electrode materials.

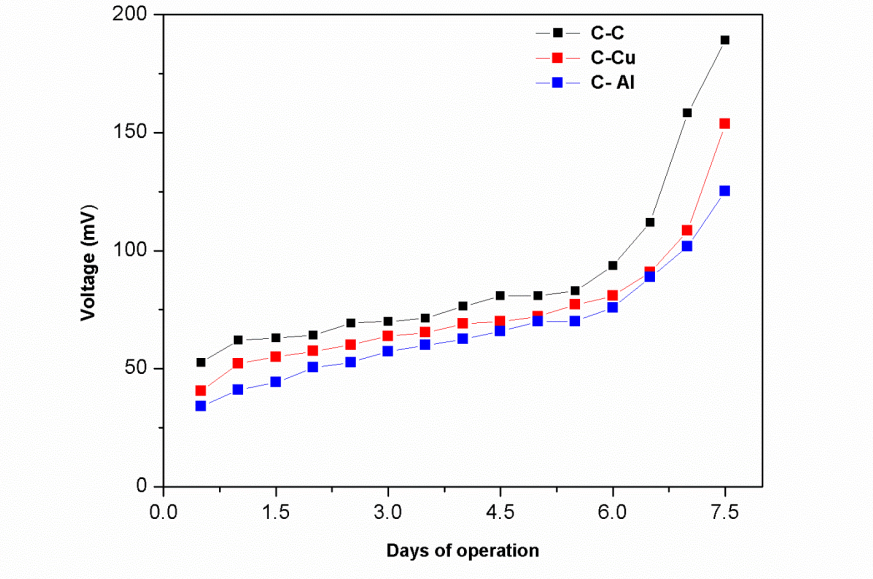

Figure 6: Voltage generated from MFC operation using different electrode pairs versus days of operations.

As shown in Figure 6, the output voltage for the three electrode pairs tested shows significant increment. However, it is observed that C-C electrode material gives the highest voltage value of 189.1 mV, followed by C-Cu electrode which give the voltage value of 153.7 mV. The highest voltage value achieved by C-Al electrode is 125.1 mV. The result shows that C-C electrode is the best electrode material in the power production of MFCs. Basically, the electricity generated by the MFCs operation is influenced by the rate of electron transfer from anode to cathode. In the present study, it is assumed that the difference in voltage production among the electrode pairs was affected by the cathode electrode materials since all three electrode pairs employed carbon electrode as the anode. Thus, the MFCs operation of all three electrode pairs had its highest microbial attachment capacity encouraging better oxidation by the bacteria at the anode. Therefore, microbial oxidation reaction is maximized in the anode chamber which could contribute to the optimization of the cathode reaction [46]. By using carbon electrode as anode, the bacteria could attached itself firmly on the carbon surface and can simultaneously provide electron transfer paths to anode [47]. Also, the rough surface anode can stimulates bacteria to produce their nanowires which help them to form bond between each other and provide an electron transfer bridges [48]. This explains the significant voltage increase that occurs in MFCs operation of all three electrode pairs.

However, among the three electrode pairs, C-C electrode pair has shown the highest voltage production than the other two types of electrode pairs. This is also mainly due to the enrichment of biofilm at the carbon cathode. As discussed before, carbon material possessed high surface roughness that led to higher biofilm formation at the cathode. The bacteria attached at the cathode act as the biocatalysts to accept electrons from cathode electrode assisting the transfer of electrons and contribute to an efficient oxygen reduction reaction. Apparently, higher bacterial formation and attachment at the carbon electrode could lead to a higher oxidation rate thus improving the rate of oxygen reduction [49]. Similar observation was observed by Dumas et al. [50] who conducted comparative study between graphite and stainless steel electrode and found that graphite electrode resulted in better energy production [50]. Carbon electrodes are highly porous among other electrode material and has less polishing level than copper and aluminium. Increasing in polishing level decreased the surface roughness value and amount of bacterial adhesion. The surface roughness has promoted the bacterial adhesion and colonization. Higher surface roughness value can help in providing a more suitable structural heterogeneity of the biofilm. This property will improve the biofilm activities, mass transfer dynamics and the open circuit potential in MFCs [40,41]. These findings explain the factor contributing to higher biofilm formation at cathode. Higher biofilm formation is encouraged at the cathode MFCs as according to Bergel et al. [51], the presence of the biofilm on the MFCs' cathode surface led to efficient electron density [51].

Besides that, another factor that could contribute to better energy production of MFC is the conductive properties of the electrode material. Higher conductivity of the electrodes are favorable as it improves the electron's transport from the anode to cathode. This is because the electrical conductivity of the material has reduced the internal activation resistance of the MFC in a short time, improving the MFC performance [52]. Carbon electrode has good conductivity as according to Zhou et al. [49] reported that graphite rod also has excellent electrical conductivity and chemical stability which therefore it is commonly used as the electrodes in MFCs [49]. Copper material has high conductivity value as well, however, this material cannot generate the highest electricity since copper has high degree of antimicrobial activity which limits its ability to collect the current [14]. Above all, it is agreed that C-C electrode is the best electrode pair compared to C-Al and C-Cu electrode materials due to its highly porous electrode surface, high corrosion resistance, do not possessed toxicity to bacteria, and has excellent conductivity and chemical stability.

4. CONCLUSION

The study conducted demonstrates the capacity of an anoxic-aerobic MFC reactor in providing effective spent caustic wastewater treatment and energy production. The MFC operation achieved maximum efficiency by using C-C electrode, with maximum of 189.1mV voltage production and 89.1% and 99.91% of COD and sulfide removal respectively mainly due to the carbon's surface morphology that is highly porous, high corrosion resistance, do not possessed toxicity to bacteria and has excellent conductivity and chemical stability allowing higher microbial adhesion and growth in which has caused the concentration of the microorganisms to be increased resulted in better MFC performance in treating spent caustic wastewater and energy production.

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