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Treatment of dairy wastewater and performance comparison of three different electrodes in microbial fuel cell system

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ABSTRACT

Dual-chambered microbial fuel cells were tested in batch mode with three different electrodes: graphite rod, graphite felt, and carbon cloth as anodes and cathodes. Dairy wastewater, after sedimentation (initial chemical oxygen demand of 1,357 mg/L), was the feed for microbial fuel cell. The inoculum for the experiments conducted was a mixed culture of anaerobic microorganisms sourced from dairy wastewater. This study aimed to evaluate the performance of three electrodes in microbial fuel cell on the basis of their power generation and their wastewater treatment efficiency (chemical oxygen demand, total organic carbon content, turbidity), respectively. Carbon cloth produced the maximum open circuit voltage and power density (0.847 V and 1.36 W/m^2) followed by graphite felt (0.812 V and 1.22 W/m^2) and graphite rod (0.658 V and 0.78 W/m^2). From the polarization studies, it was found that external resistance of 100 Ω produced the maximum power density for all the three electrodes. Electron transfer ability of the electrodes in dairy wastewater was measured by cyclic voltammetry. Biofilm growth on the surface of the anode was visualized by scanning electron microscopy. Chemical oxygen demand removal efficiency was found to be more in microbial fuel cell with carbon cloths and was estimated to be 91.3%. There was a significant reduction of total organic carbon content and turbidity in wastewater by the deployment of the three electrodes. Even though the power generated through these microbial fuel cells is low, efficiency in water treatment was found to be high. Thus, microbial fuel cells can be used in dairy industries to treat wastewater and simultaneously generate power effectively.

Keywords: Microbial fuel cells; Dairy wastewater; Power generation; Chemical oxygen demand; Anaerobic; Biofilm

1. Introduction

Renewable energy or clean energy production has been the primary concern over the recent years. This has been due to the environmental impacts created by high energy demand and subsequent utilization of conventional non-renewable resources [1]. Wastewater treatment is also an environmentally impacting issue faced by wide varieties of industries. The utilization of waste materials for the generation of green energy serves two purposes, namely lessening environment damage due to indiscriminate waste disposal practices, and decreasing usage of conventional energy resources [2]. Wastewater has the potential chemical energy in the form of organic matters. These organic matters present in the wastewater can be converted directly into clean energy with the help of microbial fuel cells (MFC) [3]. Most of the conventional treatment processes do not support the recovery of the organic matters and are also energy-consuming activities [4]. In the early 90s, energy production from the wastewater through MFC was made possible by the interdisciplinary research carried out in the field of biochemistry, microbiology, and electrochemistry [5,6]. MFCs are bioreactor which converts

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the chemical energy present in the organic matter into electrical energy by the use of microorganisms and also simultaneously reduces the chemical oxygen demand (COD) of the wastewater. Enriched microbes in an anode have shown to produce higher current [7]. Even though the power generated from the MFCs is of very low density, however, their operations are very efficient in the wastewater treatment [8]. MFCs have the advantage of operating under normal conditions unlike other fuel cells, and also have versatility in fuel usage. For instance, a great variety of degradable waste sources can be feed inside MFCs [9]. Thus, MFCs have been found to be a promising technology utilizing the waste sources to generate power and concurrently treat the wastewater.

Various studies on MFCs have been tested with domestic, municipal, and industrial wastewaters, respectively. The sources of these wastewaters may be refineries, distilleries, breweries, and paper industries [8,10–15]. Especially, domestic, municipal, and food processing-related wastewaters attracted the attention of researchers due to the presence of the magnified volume of organic sources. India is the largest producer and consumer of milk and milk products. It also has a large number of dairy industries throughout the country [16]. Wastewater discharged from dairy industries in India also poses environmental issues to natural water resources. Hence, to deal with such situations an effective wastewater treatment method is necessary. Because dairy wastewater is easily biodegradable, various biochemical methods, namely membrane reactor and anaerobic sludge blanket reactor have been used for treatment purposes. Even though biological processes are economic processes, MFC has an additional advantage of generating power during wastewater treatment. In this study, dairy wastewater was taken as the fuel for the MFC system because it contains various proteins, carbohydrates, and fat content [16]. This metabolic content can be transformed into electrical energy by the oxidation of the organic matter using microorganisms. Few studies on MFCs using dairy wastewater has been carried out focusing on the different parameters such as MFC design, electrodes, and operating conditions. Such studies aimed to generate power and reduce COD [16–19].

This study utilized cost-effective carbon-based materials for bioelectricity generation rather than using highly expensive metal electrodes like platinum and platinum-coated materials. These carbon-based materials exhibit higher electrical conductivity, chemical stability, and biocompatibility [14,20]. Though past studies have used carbon-based electrodes for dairy wastewater treatment, this research explores the usage of carbon-based electrodes in dual-chambered MFC in similar working conditions. This study also compares the findings obtained from three types of carbon-based electrodes. The objectives of the study include the following: (i) characterization of the dairy wastewater used, (ii) isolation of microbes from dairy wastewater, (iii) evaluation of biofilm growth and electrochemical characterization of the electrodes, (iv) performance evaluation of electrodes by power generation, and (v) wastewater treatment efficiency of the electrodes in MFC.

2. Materials and methods

2.1. MFC design and operation

Dual-chambered MFC was fabricated using an acrylic material with two equal volume rectangular chambers (5 cm × 5 cm × 10 cm) as shown in Fig. 1. These chambers had a total/working volume of 200/150 mL. Nafion 117 was used as a proton-exchange membrane separating the anode and cathode chambers. Nafion 117 was used due to its good selective permeability and long-term stability [21–23]. To increase the porosity of the membrane, Nafion 117 was pretreated by boiling sequentially in 30% hydrogen peroxide solution, 0.5 M sulfuric acid and deionized water. Each of these three sequential boiling steps consumed 1 h [24]. Experiments were carried out by using three different electrodes namely, graphite rod (GR; surface area of 0.0017 m^2), graphite felt (GF; surface

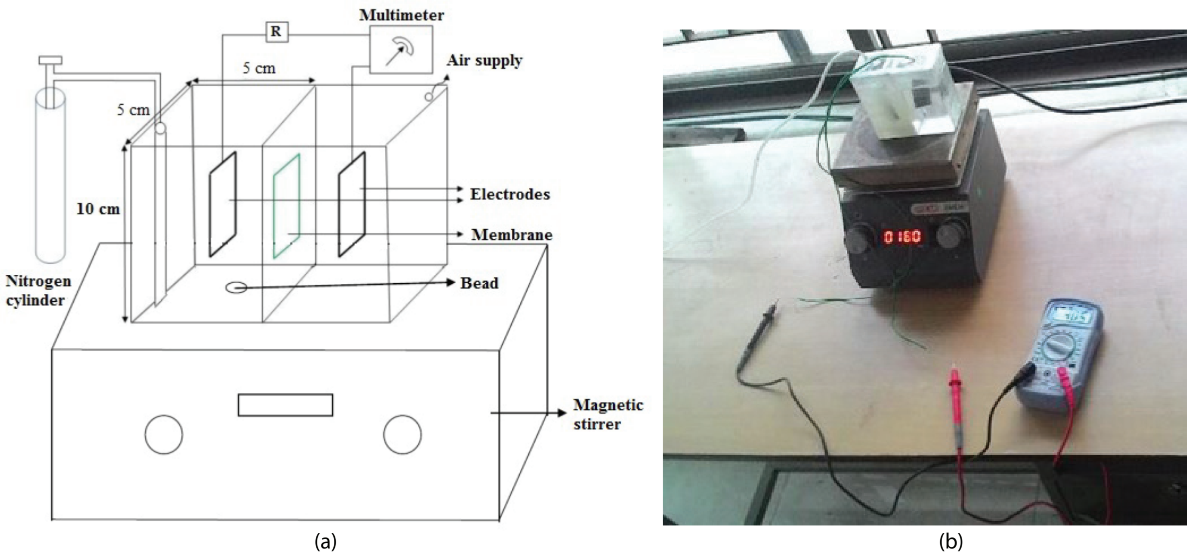

Fig. 1. (a) Schematic diagram of the dual chambered microbial fuel cell and (b) experimental setup.

area of 0.0038 m^2), and carbon cloth (CC; 30 wt% wet proofed; the surface area of 0.00316 m^2). Each of these materials was simultaneously used as both anodes and cathodes for a single experimental setup. Three different setups were made, and subsequently, experiments were carried out. Insulated copper wires were used to connect the electrodes with the multimeter. An external load of 100 Ω resistor was applied to form a closed circuit. Nitrogen purging was performed twice a day in an anode compartment. This activity was carried out with an intention to maintain an anaerobic environment by removing dissolved oxygen. Mixing at 150 rpm using a magnetic stirrer (REMI 1MLH) was provided to anolyte to avoid sludge formation in the system.

2.2. Inoculation and operation

Real field dairy wastewater was collected from MILMA Kunnamangalam, Kozhikode district of Kerala and stored at 4°C [14]. Characteristics of dairy wastewater were analyzed as shown in Table 1. Dairy wastewater of volume 150 mL (pH adjusted to 7 using Systronics digital 335 pH meter) was used as anolyte, and 150 mL of 10^{-3} M ferricyanide of pH 7 were used as catholyte. Mixed culture of anaerobic electrogenic bacteria was isolated from dairy wastewater by serial dilution method and used for biofilm formation in the anode. Thioglycollate broth (composition: L-cystine, 0.5 g/L; dextrose, 5.5 g/L; pancreatic digest of casein, 15.0 g/L; sodium chloride, 2.5 g/L; sodium thioglycolate, 0.5 g/L; and yeast extract, 5.0 g/L) was used as medium for isolation of anaerobic bacteria [25,26]. Because pure cultures require high-controlled operating environment for isolation and maintenance, such kind of cultures is not preferred. The usage of mixed culture instead of pure culture has led to reduced expenditure in this experimental setup [27–30].

2.3. Analytical measurements and calculation

Digital multimeter (V&A TECH MAS830, ATEL Electronics, Poland) has been used to record cell voltage over time. The current flow (I), the power (P), the current density (I_D), and the power density (P_D) were estimated from the following equations:

$$I = V/R \tag{1}$$

$$P = IV \tag{2}$$

Table 1
Characteristics of the dairy wastewater

Parameters	Value
Color	White
pH	6.8
COD (mg/L)	1,357 ± 15
Turbidity (NTU)	295
Total organic carbon (TOC) (ppm)	525
Total Kjeldahl nitrogen (TKN) (mg/L)	9.18
Phosphorous (mg/L)	4.95

$$I_D = I/A \tag{3}$$

$$P_D = P/A \tag{4}$$

where V is the cell voltage, R is the resistance, and A is the surface area of the electrode. An external resistance was applied to polarize the cell (30–1,000 Ω), and the current variation under closed circuit conditions was monitored to obtain polarization and power density curves. The electrochemical activity of the electrodes in dairy wastewater was evaluated using cyclic voltammograms. Cyclic voltammetry (CV) studies were carried out using electrochemical workstation (CH Instruments, CHI760, Austin USA) over the scanning range of –0.8 to +0.8 V at a scan rate of 50 mV/s. Three electrode methods were adapted with an anode electrode as a working electrode, the platinum wire as a counter electrode, and Ag/AgCl as the reference electrode [27,31].

Coulombic efficiency measures the efficiency of conversion of chemical energy present in dairy wastewater into electrical energy. Coulombic efficiency (C_E) was calculated using the following equation [17,31–33]:

$$C_E = \frac{M\int_0^t I dt}{FbV_{\text{An}}\Delta\text{COD}} \tag{5}$$

where M is the molecular weight of oxygen, I is the output current, F is Faraday's constant, b is the number of electrons exchanged per mole of oxygen, V_{An} is the volume of anolyte, and ΔCOD is the change in concentration of COD over time.

The efficiency of the MFCs in wastewater treatment was evaluated by examining the treated wastewater in the anode chamber using three measures, namely, COD (determined as per APHA [34]), total organic carbon (TOC), and turbidity (using ELICO CL52D NEPHELOMETER).

2.4. Morphology studies

Anodes used in MFC were observed for its biofilm growth by scanning electron microscopy (SEM) (Hitachi SU-6600, Japan). Samples were washed five times with phosphate buffer saline, and the washed samples were fixed with 2.5% glutaraldehyde and kept for 24 h. Glutaraldehyde and phosphate buffer are useful for preserving field samples. Glutaraldehyde is very useful in stabilizing the surface as well as intracellular structures [35–37]. Fixed samples were washed three times with sodium phosphate buffer and were dehydrated in a series of graded alcohols (10%, 30%, 50%, 80%, and 100% of ethanol). All specimens were dried and coated with a thin layer of gold [38,39]. Microbes in the biofilm usually differ with the change in the wastewater types [40].

3. Results and discussion

3.1. Characterization of biofilm growth in the anode materials

The biofilm formation on anode materials is one of the essential factors to be considered in the MFC operation. Electron transfer mechanism and the biodegradability of the organic matters in the substrate are directly related to the biofilm formation on the anode material [41]. Hence, it is mandatory to check the biofilm growth on the surface of the material. This can be carried out using the SEM analysis of the electrodes after performing the experiments. SEM images of the three electrodes are shown in Fig. 2. Images confirm the biofilm formation on the surface of the electrodes. The thickness and the concentration of the biofilm formation depend on the significant organic sources present in the wastewater. It also depends on the surface properties of the anode material for adhesion of microbes. All the three electrodes were found to have good biofilm formation, which was reflected on the COD reduction in the treated wastewater. The different shapes (rod and oval) of microorganisms reveal the presence of various types of microbes in the anodic biofilm.

3.2. *Performance of MFC on power generation*

The open circuit voltages (OCV) and the closed circuit voltages (CCV) of the MFCs fed with dairy wastewater using the three electrodes namely, GR, GF, and CC are measured. These OCV and CCV values are shown in Figs. 3 and 4. The OCV and the CCV values for the MFCs were monitored continuously with a digital multimeter. The MFC setups were operated for around 2 weeks. In general, CCV values of MFCs are lesser than the OCV due to the load given to the circuit. The CC attained a maximum OCV value of 0.847 V on the fifth day. On the other hand, GF reached the maximum OCV value of 0.812 V on the sixth day, and GR produced the

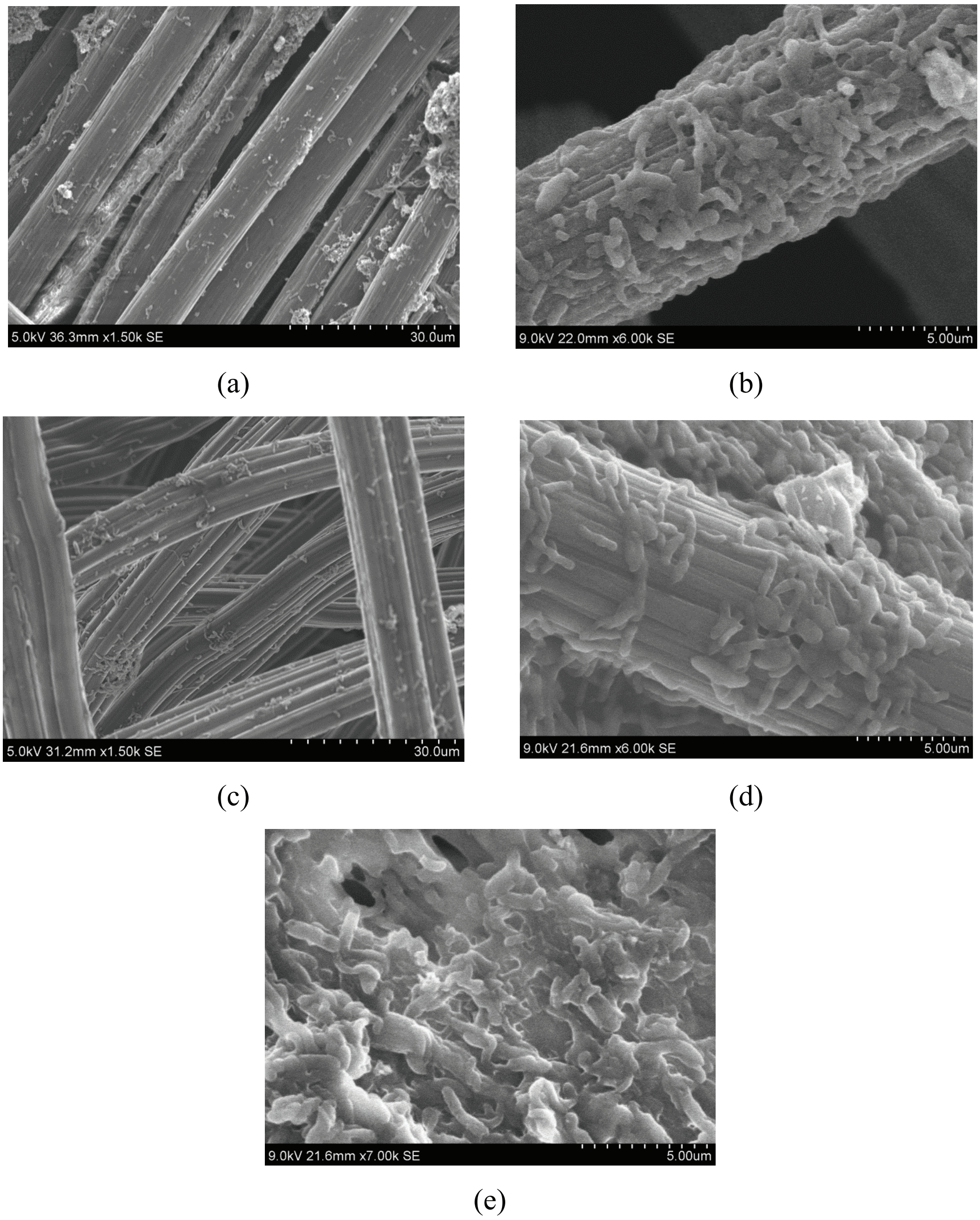

Fig. 2. SEM images of the three anodes with microbial adhesion: (a), (b) carbon cloth; (c), (d) graphite felt; and (e) graphite rod.

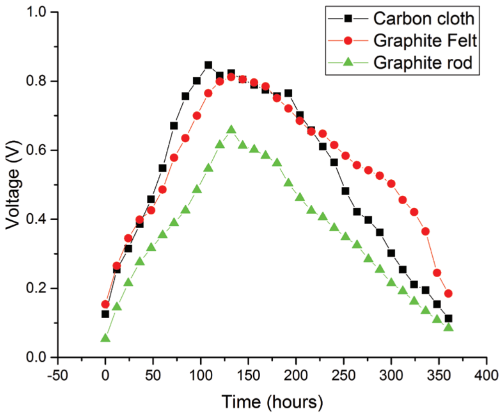

Fig. 3. OCV of MFCs with three different electrodes.

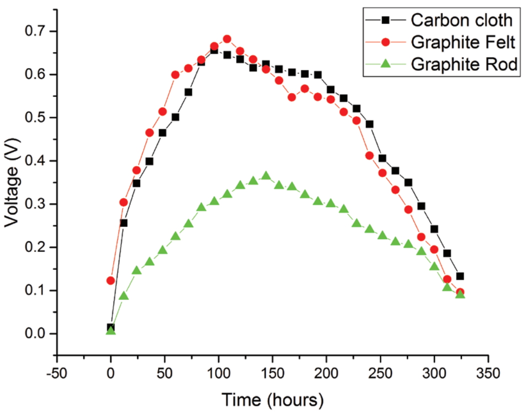

Fig. 4. CCV of MFCs with three different electrodes.

highest OCV value of 0.658 V on the sixth day. Though the operating conditions are the same for all the electrodes, CC had better voltage than GF and GR. This may be due to the following reasons: the electrical conductivity of the material, better biofilm formation on the electrode surface, and faster electron transfer mechanism at the interface between biofilm and anode surface leading to higher electrochemical activity.

The OCV values obtained in this study were higher in comparison with that of the MFC fed with dairy wastewater tested with plain graphite plate under anaerobic condition (maximum OCV of 780 mV) [24]. GF showed the maximum CCV of 0.682 V in comparison with that of the CC (0.656 V) and GR (0.364 V), respectively. From the results, it can be inferred that the dairy wastewater used in this study has the potential organic matters for power generation.

Fig. 5 shows the effect of varying resistances to power density. The power density increased with a corresponding raise in resistance in the interval 0–100 Ω. Subsequently, the power density started to decrease with further increase in the resistance. In general, at a lower external resistance, the electrons easily pass through the circuit and swiftly oxidize

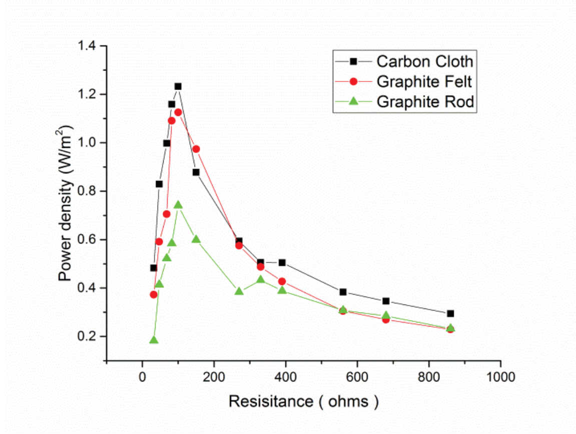

Fig. 5. Performance of MFC to varying resistances.

the organic material at the anode chamber [18]. In MFC, the maximum power densities are attained when the external resistance is equal to the internal resistance [42].

Polarization and power curves of the MFCs for the three electrodes are shown in Figs. 6–8. The polarization of the MFC was performed under varying external resistances (32–860 Ω). The optimum resistance was found to be 100 Ω for all the three MFCs producing the maximum power densities (CC: 1.23 W/m^2, GF: 1.13 W/m^2, and GR: 0.74 W/m^2). The maximum power densities obtained were much higher than the ones reported in the earlier study [43]. The earlier study had used dairy wastewater as a substrate in MFC and produced a power density of 419 mW/m^2 [43].

From the CCV, current, power, current density, and power density were calculated using Eqs. (1)–(4). CC produced the maximum power density of 1.362 W/m^2 (at the 96th h) in comparison with both GF (1.224 W/m^2 at the 108th h), and GR (0.779 W/m^2 at the 144th h), respectively. However, GR produced the maximum current density of 2.141 A/m^2, followed by CC (2.076 A/m^2) and GF (1.795 A/m^2), respectively. The results obtained for power density in this study was higher than ones obtained in the earlier study [18]. The earlier study produced the power density of 0.621 W/m^2 using the MFC with graphite plate as an anode for dairy wastewater treatment [18]. A similar study using dairy wastewater in MFC with graphite plate as an anode under anaerobic condition also produced a lesser power density of 161 mW/m^2 [24]. The coulombic efficiency of the MFC was evaluated based on the COD value. CC showed the higher CE of 22.26% than the other two electrodes (GF: 17.21% and GR: 0.98%). The coulombic efficiency was found to be comparable with that of various MFCs tested with dairy wastewaters, as shown in Table 3.

3.3. Electrochemical characterization of anode materials

Electron transfer efficiency and electrochemical activity of the anode was analyzed using CV. The CV plots for the three electrodes CC, GF, and GR in dairy wastewater are shown in Fig. 9. From the results, it is understood that CC has a better electrochemical activity than that of both GF and GR. Higher the peak current value, larger the oxidation current densities [44].

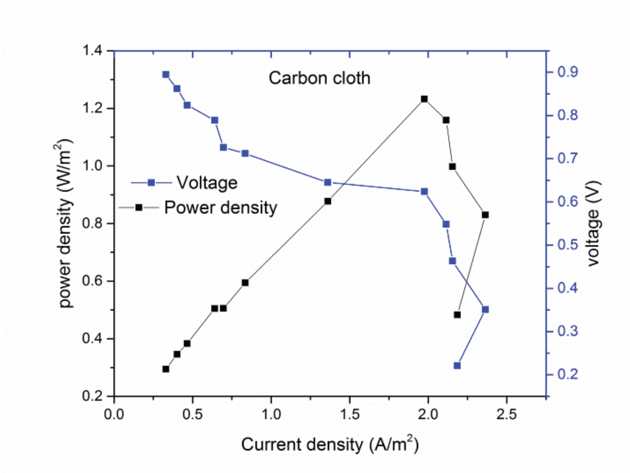

Fig. 6. Polarization and power curves for the MFC with CC as anode.

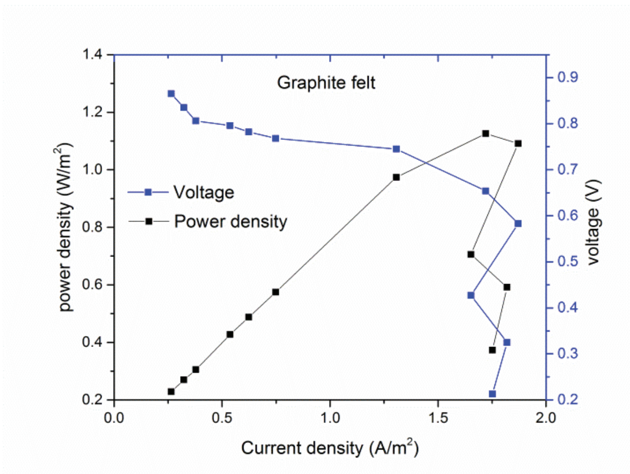

Fig. 7. Polarization and power curves for the MFC with GF as anode.

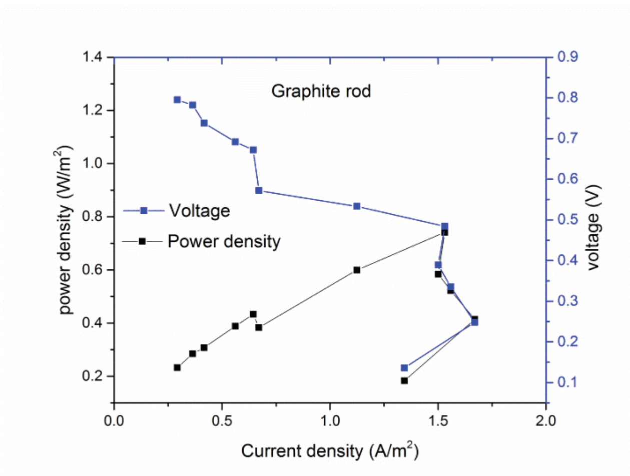

Fig. 8. Polarization and power curves for the MFC with GR as anode.

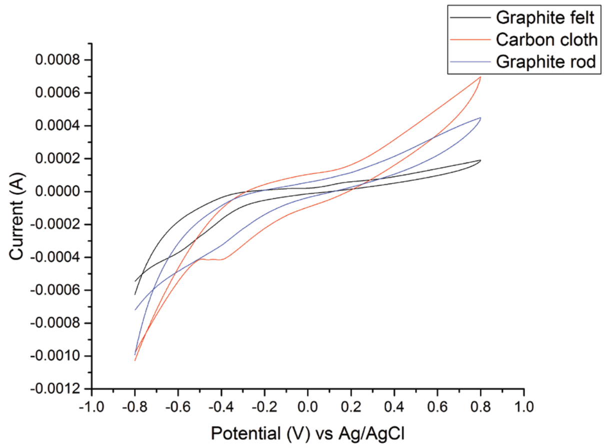

Fig. 9. CV for three electrodes.

3.4. Performance of MFC on wastewater treatment

MFCs with different anode materials were tested for COD removal. This was performed to evaluate the potential of these MFCs to act as a wastewater treatment system. Because the influent dairy wastewater contains organic matters and some nutrients, such as nitrogen and phosphorous, the changes in the concentration of these compounds were also analyzed. Table 2 provides the information about the differences in the COD, TOC, turbidity, total Kjeldahl nitrogen (TKN), and phosphorus content of the effluent respectively. There is no significant difference in the effluent characteristics treated using different anode materials. The COD reduction was found to be almost 90% for all the three electrodes (CC: 91.3%, GF: 88.43%, and GR: 90.05%). The results were relatively higher in comparison to ones obtained using dual-chambered MFC possessing (initial COD of 1,487 mg/L) carbon paper as anode and dairy wastewater as substrate showed 81.29% COD reduction [13]. The results obtained in this study were also comparable with the findings reported in various studies operated at similar working conditions, as shown in Table 3. The TOC reduction of wastewater was also found to be in the range between 85% and 90%. The CC exhibited the higher reduction (89.7%) than the other two electrodes (GF: 88.4% and GR: 85.7%). A significant change in turbidity of wastewater was found. Clarity of the treated water confirms the change in the turbidity of the water. The percentage turbidity reduction was more in GF (89.49%) when compared with both CC (88.48%) and GR (88.31%). In the previous study, MFCs have been tested and proved to reduce nitrate in the wastewater [45]. However, in this study, increase in the nitrogen content was observed because TKN was converted into nitrogen, nitrate, or nitrite using any subsequent nitrification process. Also, increase in the phosphorus was found in the effluent. This is due to the conversion of phosphorus into orthophosphate by chemical mechanisms [46]. This increase in orthophosphate happens under an anaerobic condition at low redox potential [17].

4. Conclusion

From this study, the effect of CC, GF, and GR as anode and cathode materials in MFC fed with dairy wastewater

Table 2
Comparison of electrodes on power generation and wastewater treatment

Parameters on power generation		Carbon cloth	Graphite felt	Graphite rod
Maximum OCV (V)		0.847 at the 108th h	0.812 at the 132th h	0.658 at the 132th h
Maximum CCV (V)		0.656 at the 96th h	0.682 at the 108th h	0.364 at the 144th h
Maximum current density (A/m^2)		2.076	1.795	2.141
Maximum power density (W/m^2)		1.362	1.224	0.779
Coulombic efficiency (%)		22.26	17.21	0.98
Wastewater treatment	Initial	Final (after treatment)		
COD (mg/L)	1,357	118.059	157.005	135.022
Turbidity (NTU)	295	33.984	31.005	34.486
Total organic carbon (ppm)	525	54.075	60.9	75.075
TKN (mg/L)	9.18	24.12	25.08	21.82
Phosphorous (mg/L)	4.95	6.10	6.56	6.34

Table 3
Studies related to COD removal efficiency and coulombic efficiency during wastewater treatment

MFC configuration	Feed mode	Inoculum	COD removal (%)	CE (%)	References
Dual chamber	Fed batch	Dairy wastewater	83	14	[10]
Dual chamber	Batch	Dairy wastewater	64.5–74.6	5–12	[19]
Dual chamber	Batch	Dairy wastewater	91	17	[24]
Dual chamber	Batch	Dairy wastewater	95	14	[46]

was tested. Based on the power generation, CC was found to exhibit maximum power density compared with the other two electrodes. Cost of the process is mainly based on the electrode materials, membrane, and its durability. Use of less expensive carbon-based electrodes and a mixed consortium makes the process economically feasible. Still, the process can be made economically possible by replacing the expensive Nafion membrane with low cost and selective permeability property membrane. Because the study is operated at room temperature and almost neutral environment, the durability of the electrodes are longer and can be reused. All the three electrodes showed a similarity in the wastewater treatment. The COD, TOC, and turbidity reduction were found to be similar for all electrodes and possess little variation. Treated effluent was found to contain lower levels of COD and turbidity. Therefore, it may be safe for disposal without any further treatments. In comparison with various factors, CC can be suggested as a better anode material for treating dairy wastewater with simultaneous power generation. Identification of isolated anaerobic organisms (used for this study) using 16sRNA sequencing and modification on CC anode surface to increase the electrochemical activity and biofilm adhesion would be scope for future work. Because milk-processing operation runs almost through the year, large sources of wastewater have potential to produce renewable energy from the treatment, which would be helpful for the sustainable wastewater management and simultaneous renewable energy production.

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