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Evaluation of paper recycling industry wastewater treatment and simultaneous bioelectricity generation in a microbial fuel cell

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ABSTRACT

Microbial fuel cell (MFC) is an integrated technique for treatment of wastewater as well as electricity production. In this research, the performance of proton exchange membrane (PEM) was studied using double chambered microbial fuel Cell (DCMFC) and compared with conventional salt-bridge. The effect of potassium ferricyanide and potassium permanganate mediators in phosphate buffer solution (PBS) were evaluated. For both the configurations, graphite felt was used as a cathode and carbon cloth was used as an anode. The external resistance was fixed at 10 kΩ for both configurations. The results indicated that the PEM and potassium permanganate mediated MFC had better chemical oxygen demand (COD) removal efficiency and power density production than salt bridge. The maximum power density and COD removal efficiency were achieved as 15.075 mW/cm^2 and 75%, respectively. It was also found that the power density produced in membrane based fuel cell was 2.5 times the salt bridge fuel cell. The results showed the paper recycling wastewater was treated by PEM -MFC effectively and efficiently than salt bridge MFC and electricity was generated as a byproduct.

Keywords: MFC; Electrochemistry; Wastewater treatment; Mediator; Paper recycling wastewater

1. Introduction

Paper recycling is the process of converting the waste paper into usable products. In India, about 550 paper recycling units are using waste paper as a source of raw material [1]. Paper recycling industries require huge quantity of water for production, which increases the relative proportion of wastewater generation. In addition, the paper recycling industrial wastewater contains soluble organic matters and cellulose particles which are difficult to degrade by the conventional treatment methods [2]. For the past two decades, the paper recycling industrial effluent is treated by various conventional treatment methods namely sedimentation and flotation technique and precipitation and coagulation technique. Although, these techniques remove 83% of lignin, it requires secondary treatment [3,4]. In ultra-filtration, the chemical oxygen demand (COD) removal and total dissolved solids (TDS) removal efficiencies were achieved as 95% and 50%, respectively. However, the flux was reduced by 35% due to the membrane fouling, which requires pre-treatment [5]. In photo-Fenton process, the combination of $Fe^{2+}/H_2O_2/UV$ was employed to treat the paper recycling industrial effluent. But the reaction mechanism is too complex, generates huge quantity of sludge and lowers the degradation rate [6]. To overcome the limitations, it is important and urgent to find an alternative method to degrade the paper recycling wastewater. The research is focused on development of sustainable environment and to promote an economical and effective treatment technique for industrial wastewater [7]. The microbial fuel cell (MFC) is as an emerging technique, for the treatment of industrial wastewater and simultaneous electricity generation. Energy is considered as an essential building block of the entire process of growth and survival of all livelihoods. To avoid overexploitation of natural resources

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and reduce the water demand, MFC is an advanced method for treatment of wastewater and production of electricity [8]. Future economy of our country is significantly depending on the availability of energy resources [9]. Different types of wastewater have been successfully treated by MFC [10]. Microbes are maintained at an anaerobic condition enhances the degradation of cellulose which intern generates electricity rather than consumption of power [11].

Several studies have reported that, using DCMFC oxidizes the complex matter present in industrial wastewater in to simpler form [12,13]. Increase in concentration of PBS from 50 to 100 mM leads to increase in power density from 501 to 672 mW/m^2 [11]. Anode material up-gradation is an important parameter for power generation in DCMFC [14]. Material having high conductivity increases the porosity and thereby increasing the surface area of the electrode [15]. Similar studies were reported using distillery wastewater [9].

The present study investigates the treatment of paper recycling industrial wastewater using double chambered MFC with two different configurations, one with salt bridge and other with proton exchange membrane (PEM). In addition, the performance of electron acceptors viz., potassium permanganate and potassium ferricyanide is compared in terms of power generation and treatment efficiency.

2. Materials and methods

2.1. Configuration of microbial fuel cell

2.1.1. Salt Bridge–MFC

Double chambered microbial fuel cell (DCMFC) was constructed as reported earlier [16]. The salt bridge which separates the chamber was prepared by dissolving potassium chloride (4%) and Agar (5%) in distilled water and then heated for 3 min. The hot solution was poured in polyvinyl chloride (PVC) pipe (1.7 cm diameter and 11.5 cm length) and allowed to solidify without any disturbances. The spacing between the anode and cathode was maintained as 12 cm. Carbon cloth was used as an anode and graphite felt was used as cathode, both the electrodes were procured from Sainergy fuel cell store, India. The electrodes were connected to external resistor and multimeter through a copper wire. Schematic diagram of double chambered MFC reactor was shown in Fig. 1. The anodic chamber was filled with paper recycling wastewater and maintained in an anaerobic condition. In the cathode chamber, two different catholytes namely PBS (2.452 g of potassium dihydrogen phosphate (KH_2PO_4) and 4.576 g of dipotassium hydrogen phosphate (K_2HPO_4) with 5 mM potassium permanganate and PBS with 5 mM potassium ferricyanide was used, and the performance of the catholytes were evaluated. The pH of the electrolytes were maintained at 6.9 by adding NaOH or HCl. Maintaining the pH at neutral condition results in enhanced power generation. Fluctuation in pH affects the performance of membrane, conductivity, ionic concentration of electrolyte and also inhibits the microbial activity [17]. The cathodic chamber was aerated throughout the experiment to increase the dissolved oxygen content.

2.1.2. PEM-MFC

In DCMFC, anodic and cathodic chambers were separated by PEM (Nafion®117) which was procured from Dupant & Co., USA. The MFC consist of two cylindrical chambers made of borosilicate, with capacity of 250 mL each. PEM was held by the clamp between two flattened chambers and interspace with an air-tight gasket. The size and spacing between

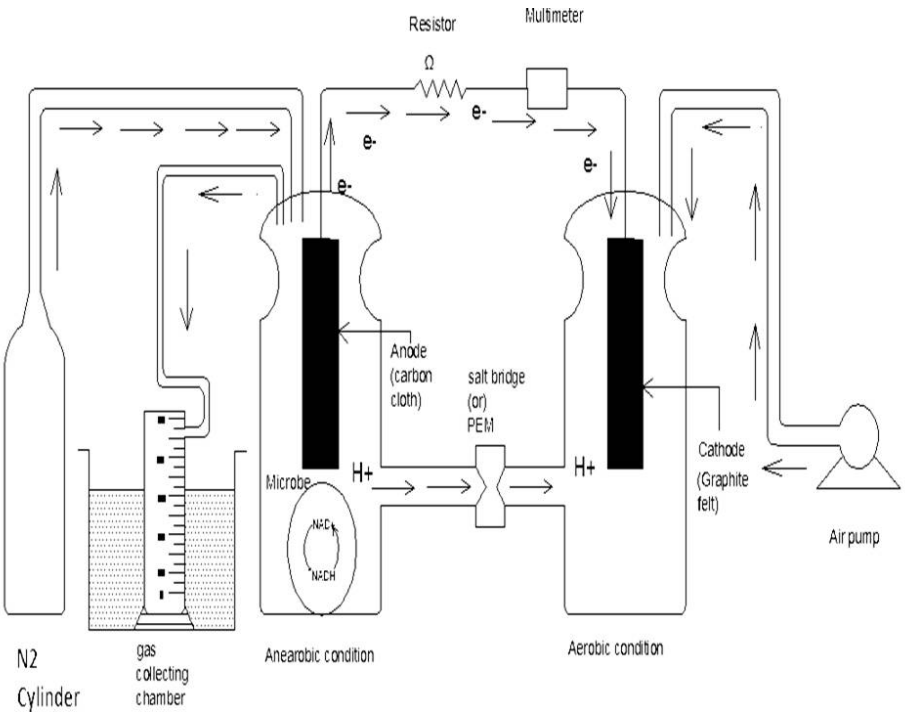

Fig. 1. Schematic of double chambered microbial fuel cell reactor.

the electrodes, electrolyte solution, resistance used and pH maintained were same as that of salt bridge. The cathodic chamber was aerated with a fish tank aerator to increase the dissolved oxygen content. The total experimental setup was operated at room temperature in fed batch mode. Before starting the experiment, both compartments were sterilized for 20 min using autoclave. The Nafion®117 membrane was pretreated with hydrogen peroxide solution followed by distilled water each for 1 h. Also, the electrodes were pretreated by soaking in 1 M HCl followed by 1 N NaOH to overnight.

2.2. Inoculum and wastewater collection

The raw Paper recycling wastewater was collected from a paper recycling industry in Tamil Nadu and stored below 4°C until further use. The characteristics of paper recycling wastewater are presented in Table 1. Small quantity of sludge and soil were collected from paper recycling mill premises, which was used as an inoculum. Paper recycling wastewater was mixed in the mineral salt medium solution with 1:1 ratio, in which the collected sludge and soil were added. The solution was stirred at 120 rpm for 7 d and then centrifuged at 10,000 rpm for 20 min. Finally, the soil pellets were collected and added to the fresh mineral salt medium for sub-culturing the inoculum. When the voltage dropped below 50 mV, the reactor solution has to be replaced. A small quantity of anolyte solution was left in the reactor so as to maintain the biomass for the next cycle.

2.3. Analysis and calculation

2.3.1. Electrochemical analysis

Cyclic voltammetry was done in the potential window –0.5 and +0.5 V at a scan rate of 10 mV/s, using 3 electrode setup. A Pt wire, Ag/AgCl and carbon cloth electrodes were used as counter electrode, reference electrode and working electrode, respectively. The voltage across the external resistor was monitored at 10 h interval using a multimeter. Voltage, power, current density (C.D) and power density (P.D) values were calculated using Eqs. (1)–(4), respectively.

$$\text{Voltage } V = I \times R \tag{1}$$

$$\text{Power output } P = I \times \text{V} \tag{2}$$

Table 1
Characteristics of paper recycling wastewater

Parameters	Average value
BOD_3, mg/L	615
COD, mg/L	1,792
Chlorides, mg/L	257
Oil and grease, mg/L	78
pH	7.12
TSS, mg/L	158
TDS, mg/L	1,428
Sulphates, mg/L	85
Color	Turbid

$$\text{Current density (C.D)} = I_{\max} / A \tag{3}$$

$$\text{Power density (P.D)} = P_{\max} / A \tag{4}$$

where I is the harvested current in (A), V is the voltage in (V), R is the resistor in (Ω), P is the power in Watts and 'A' is the surface area of the electrode. Current density is the ratio of the maximum current to the surface area of the electrode and power density is ratio of maximum power to the surface area of the electrode.

2.3.2. Physico- chemical analysis

COD was determined by open reflux, dichromate titrimetric method as per standard methods [18]. To known volume of sample, a known amount of potassium dichromate and mercuric sulfate was added to remove chloride ion interference. The mixture was open refluxed with concentrated sulfuric acid and silver sulfate reagent at 150°C for 2 h. The amount of unreacted dichromate was determined by titration against a standard ferrous ammonium sulfate (FAS) solution using ferroin as the indicator. The difference between the dichromate originally added and the dichromate remaining unreacted gives the amount of dichromate used for the oxidation of organic compound was calculated by Eq. (5).

$$\text{COD (mg /}I\text{)} = ((A - B) \times 8000 \times M) / \text{volume of sample} \tag{5}$$

where A is mL of FAS consumed for blank, B is mL of FAS consumed for sample and M is the molarity of FAS.

Biochemical oxygen demand (BOD) was determined by titrimetric method as described by standard method [18]. The dilution water was prepared by aerating the required volume of distilled water in a suitable bottle. 1 mL nutrients solution viz., phosphate buffer, magnesium sulfate, calcium chloride and ferric chloride were added for each litre of dilution water. A known volume of sample was taken from two BOD bottles and remaining filled with dilution water. One bottle was kept for initial DO determination and the other bottle was incubated at 27°C for 3 d for determining final DO. 1 mL manganese sulfate solution and 1 mL alkali-iodide-azide reagent were added in each bottles. After settlement of precipitate, 1 mL of concentrated sulfuric acid was added to dissolve the flocs. Similarly the blank was prepared with dilution water. 200 mL of sample was taken from each bottle and titrated against sodium thio-sulfate (0.025 N) solution until it turns to pale yellow color. Few drops of starch solution were added and again the titration was continued until disappearance of blue color. The BOD value was calculated by Eq. (6).

$$\text{BOD (mg /}I\text{)} = ((\text{Intial DO} - \text{Final DO}) / \text{percentage of Dilution}) \times 100 \tag{6}$$

TDS content was determined by gravimetric method as per standard method [18]. A known volume of sample was filtered through Whatmann filter paper (0.45 μ) and the filtrate was dried on the hot plate until complete dry. The increase in weight of dish indicated the TDS content present in wastewater was calculated by Eq. (7).

$$\text{TDS (mg /}I\text{)} = ((\text{Intial weight} - \text{Final weight}) \times 1000) / (\text{volume of sample taken}) \quad (7)$$

2.3.3. Scanning electron microscope

After digestion period, carbon cloth was removed from the anode chamber to observe the bio-film formation over the electrode. The bio-film formation was fixed by soaking the electrode in 2.5% glutaraldihyde solution followed by ethanol series wash (20%, 40%, 60% and 70%) to dehydrate the electrode. After that the electrode was air dried and activated carbon powder coated for clear observation.

3. Results and discussion

3.1. Comparison of salt bridge-MFC and PEM-MFC in power density production

Paper recycling industrial wastewater was treated by DCMFC at room temperature with the initial pH of 6.9. Over 13 d of digestion period, the maximum voltage produced by PEM-MFC was 1,276 mV at 60th h. The voltage production was gradually decreased due to depletion of the nutrients. The change in voltage production was influenced by the degradation of wastewater under the microbial activity in growth phase [19]. But the maximum voltage produced by salt bridge-MFC was 894 mV at 48th h which was lower than PEM –MFC as shown in Fig. 2. From the results, it was observed that the lag phase of microbial growth in PEM-MFC and salt bridge-MFC were achieved at 25th and 75th h, respectively [20]. From the results, the voltage production of PEM-MFC was better compared to salt bridge-MFC due to lower internal resistance [21]. The salt bridge-MFC produced less power output than PEM-MFC, due to higher internal resistance in salt bridge [20].

The maximum power density production was calculated by the Eq. (4), from the ohms law equation and basic power Eqs. (1) and (2), respectively. The power density values of PEM-MFC (15.076 mW/cm^2) was greater than salt bridge (7.4 mW/cm^2) as shown in Fig. 3. In the presence of Geobacter microorganism, the power density obtained from PEM-MFC (38 mW/m^2) was very much higher than salt bridge-MFC (2.2 mW/m^2) [21]. The power density produced in salt bridge-MFC was low, since the internal resistance in the salt bridge was 15 times higher than for PEM-MFC [20]. Dairy industry wastewater was used in catalyst less membrane MFC produced the power density of 621.1 mW/m^2 and COD removal efficiency of 90% [22]. The COD removal efficiency of ethanol stillage wastewater by using PEM-MFC and salt bridge were achieved as 73% and 70%, respectively [20]. The power density produced as 230 mW/m^2 and COD removal efficiency was achieved as 89% for food processing industrial wastewater [23]. From the results it was clearly noted that, the COD removal efficiency of paper recycling industrial wastewater in PEM-MFC (75%) was higher than the salt bridge-MFC (72%).

3.2. Effect of mediator on salt bridge –MFC and PEM-MFC

Experiments were conducted to investigate the effect of mediators on microbial growth and removal efficiency of PRWW. Potassium permanganate ($KMnO_4$) and potassium ferricyanide ($K_3Fe(CN)_6$) were used as mediators at the concentration of 5 mM with phosphate buffer solution. Among the two mediators, cell voltage produced by $KMnO_4$ (1,276 mV for PEM) was higher than $K_3Fe(CN)_6$ (780 mV for PEM) as shown in Fig. 4. The $KMnO_4$ was found to be an attractive mediator because it has minimum effect on the microbial growth. After starting up the reaction $K_3Fe(CN)_6$ mediated catholyte was replaced by 30 mM $KMnO_4$ (3.6 mS/cm), hence the performance fuel cell have been improved in terms of power density production [24]. The power generation can be increased by increasing the solution conductivity using concentrated PBS which also improves the treatment efficiency of wastewater [25]. Power density produced by $KMnO_4$ and $K_3Fe(CN)_6$ mediators in PEM-MFC was 14.01 and 4.91 mW/cm^2, respectively. Similarly, the power density produced by salt bridge-MFC was 7.4 and 3.96 mW/cm^2 for

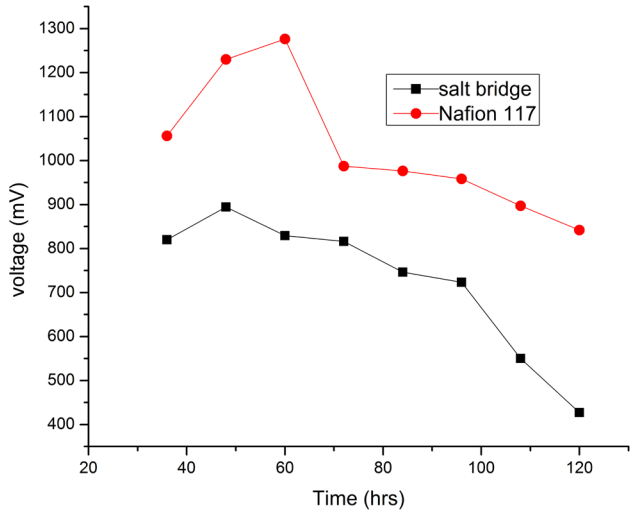

Fig. 2. Comparison of voltage production in PEM-MFC and salt bridge.

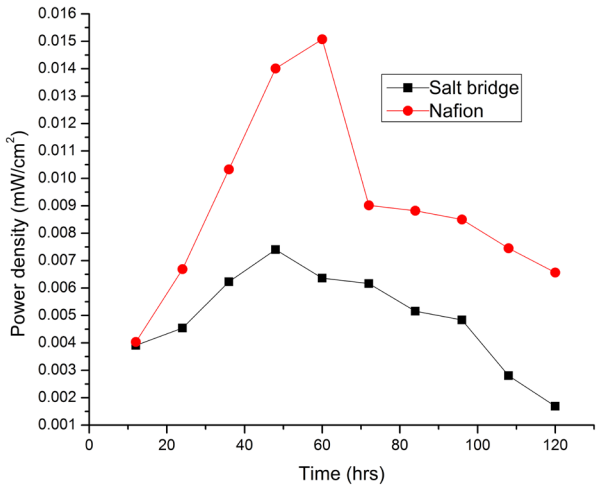

Fig. 3. Comparison of Power density production in PEM-MFC and salt bridge-MFC.

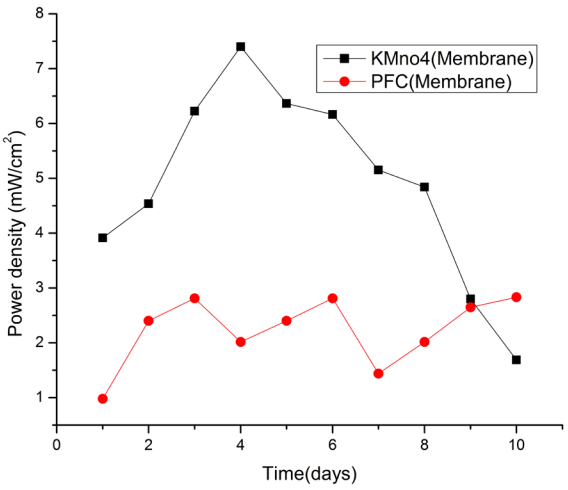

Fig. 4. Comparison of Power density production with potassium permanganate and potassium ferricyanide in PEM-MFC.

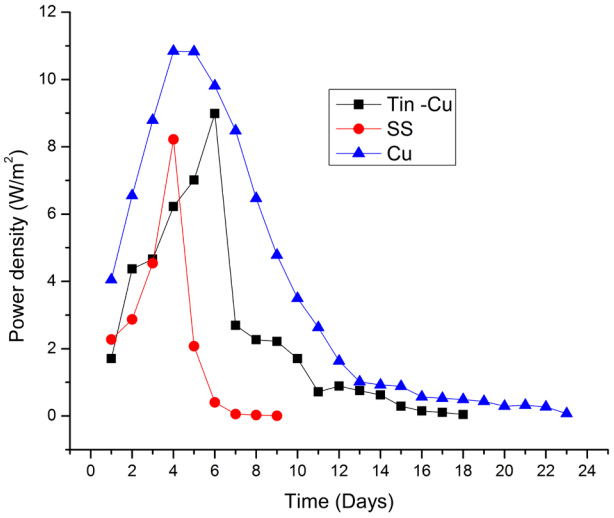

Fig. 5. Comparison chart between stainless steel, copper and tin coated copper wire.

$KMnO_4$ and $K_3Fe(CN)_6$ mediators, respectively. In general the mediators inhibit the growth of bacterial cell [20]. Potassium ferricyanide has high inhibiting effect on the microbial community. The structure of lignocellulose molecules present in PRWW is similar to humic acid, which has been already stated as a superior mediator for bioelectricity production in MFC [26,27]. In addition, lignin mediated fuel cell achieved as 81% of COD removal efficiency and 93 W/m^2 of power density production from PEM-MFC [20]. In addition to that the performance of potassium permanganate mediated MFC with paper recycling industrial wastewater enhanced the power generation and treatment efficiency.

3.3. Effect of current collector

Current collector is also an important parameter for power density production. This work explains the conductivity of different current collectors viz., stainless steel, copper and tin coated copper wire. Among the three collectors, copper wire produced more power output than others which was represented in Fig. 5. Many of the research works has used copper wire as a current collector [7,28]. Granular activated carbon (GAC) surrounded by stainless steel mesh cage has produced 410 mV of voltage in fuel cell [24]. Aluminium plate current collector has generated 2,212.57 mW/m^2 of power density which was higher than other current collectors [29]. Coarser stainless steel mesh electrode produced higher power density (1,616 mW/m^2) than finer mesh electrode (599 mW/m^2) [30]. Stainless steel insulated copper wire generated the open circuit voltage of 582 mV in MFC [25].

3.4. Scanning electron microscopy images of electrodes

Surface morphology of electrode was analyzed by scanning electron microscopy (SEM) at 5 KV with a zeiss instrument. Figs. 6 and 7 clearly illustrate the attachment of microorganism over the electrode surface and bare electrode, respectively. In this study, carbon cloth was used as anode

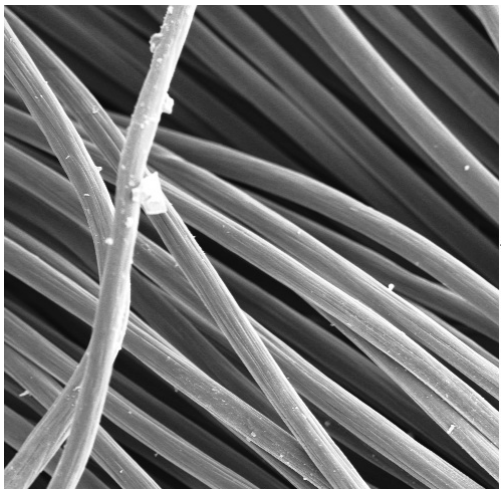

Fig. 6. SEM image of bare carbon cloth.

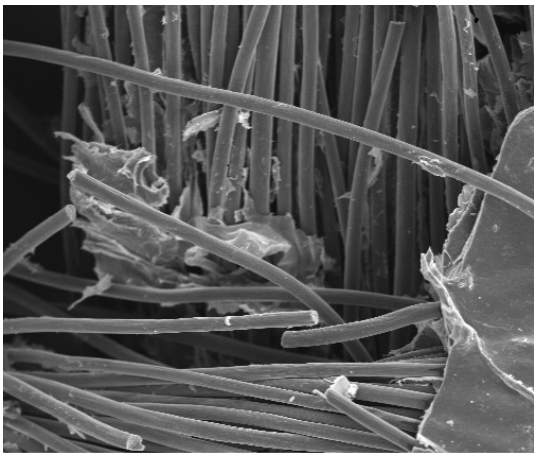

Fig. 7. SEM image of biofilm formed carbon cloth.

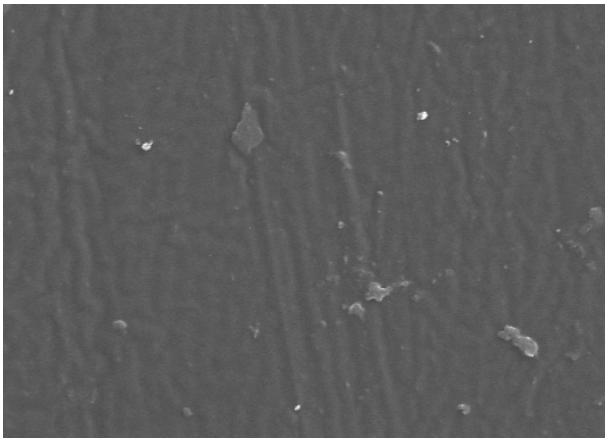

Fig. 8. SEM image of Nafion®117 before treatment.

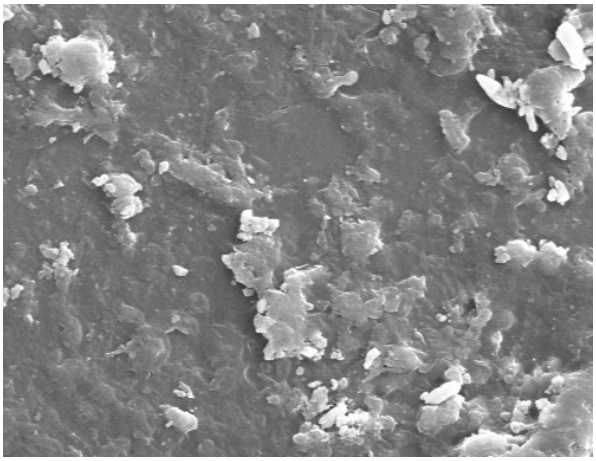

Fig. 9. SEM image of Nafion®117 after treatment.

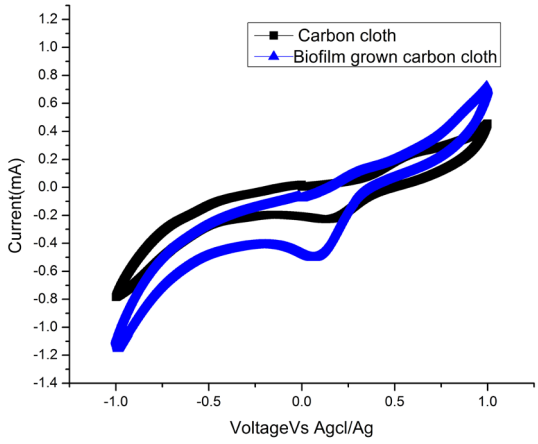

Fig. 10. Cyclicvoltametry of bare carbon electrode compared with biofilm formed electrode.

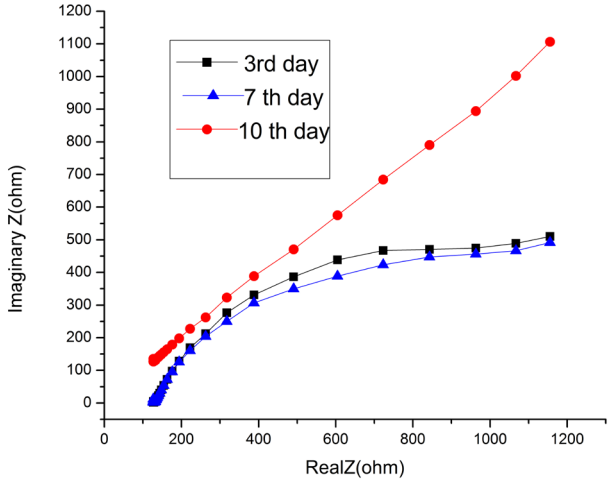

Fig. 11. Nyquits plot of electrode in EIS technology.

for both PEM-MFC and the salt bridge-MFC is hydrophobic in nature. The microbial attachment would increase the electron transfer rate, however thicker bio-film can also affect the electron transfer rate due to high internal resistance [31–33]. Figs. 8 and 9 illustrate the SEM images of Nafion®117 membrane before and after wastewater treatment, respectively. At the end of the third cycle, the voltage production got decreased due to membrane fouling.

3.5. Electrochemical measurements

Cyclic voltammetry and electrochemical impedance spectroscopy (EIS) were analyzed using three electrode setup. A Pt wire, Ag/AgCl and carbon cloth electrodes were used as counter electrode, reference electrode and working electrode, respectively. Bio-catalytic activity of microbes developed the clear redox peaks in voltammogram [34,35]. Voltage produced by bare carbon cloth was less than bio-film formed electrode, because the microbial growth enhanced the oxygen reduction rate of the electrode. Fig. 10 clearly shows that voltammogram of bare carbon cloth and microbial attached electrode. EIS results were plotted in Fig. 11. EIS plot illustrate that, the resistance values of electrode at 3rd, 7th and 10th d of the treatment. Third day the resistance value was high and at the 7th d the resistance value was reduced due to bio-catalytic activity of microbes. However, at the 10th d the internal resistance of electrode has increased due to thicker bio-film formation. Increase in Ohmic resistance values would decrease the current production [21].

4. Conclusion

This study proves that the double chamber MFC is an emerging technique to treat the paper recycling wastewater as well as electricity could be generated with an aid of bio-catalyst in the anode. The results clearly indicate that the electricity generated from PEM-MFC was twice the salt bridge-MFC. The COD removal efficiency was achieved as 79%.

From the experimental analysis, potassium permanganate was identified as an ideal mediator. It was found that potassium permanganate has no inhibiting effect on microbial growth and it enhanced the electricity generation. Copper current collector generated more voltage than other three collectors, due to the internal resistance. It can be concluded that PEM-MFC is a promising technique to treat the paper recycling wastewater and simultaneous electricity production.

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