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COMPARISON OF PERFORMANCE OF AN EARTHEN PLATE AND NAFION AS MEMBRANE SEPARATORS IN DUAL CHAMBER MICROBIAL FUEL CELLS

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Abstract

The performance of microbial fuel cells (MFC) employing an earthen plate as a membrane separator is compared to that using Nafion 117 in an identical up-flow dual-chambered cylindrical cell configuration. The MFC configuration is of a cylindrical outer cathode chamber separated by the membrane from a concentric rectangular inner anode chamber. The fuel cells, operated under continuous mode at hydraulic retention time of 12 hr, achieved average chemical oxygen demand removal efficiency of 60% and 48%, for the Nafion and earthen plate separators, respectively. The microbial fuel cells based on the earthen plate separator generated slightly lower average (28%) and maximum (48%) power densities than Nafion separator which is likely due to the higher membrane resistance. The earthen plate separator is 99% cheaper than the Nafion membrane, showing promise as an alternate separator for application to MFC technology.

Key words: chemical oxygen demand, earthen plate, microbial fuel cell, power density, proton exchange membrane

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1. Introduction

Microbial fuel cell (MFC) is an emerging technology, which has been paid much attention in recent years (Duteanu et al., 2010; Harnisch et al., 2008; Logan et al., 2006a, 2006b; Logan and Regan, 2006; Pant et al., 2016; Zhang et al., 2009; Zuo et al., 2008), for application to wastewater treatment with simultaneous electricity generation. An MFC is a bio-electrochemical system where bacteria catalyze the degradation of organic matter under anaerobic conditions to convert the energy stored in chemical bonds of organic compounds to electrical energy. The MFC performance is affected by many factors, such as reactor configuration, type of electrode material and membrane separator, solution pH, temperature, substrate type and concentration, hydrodynamic stress and feed rate, hydraulic retention time (HRT), inoculums selection or enrichment, cathodic electron acceptor selection (Oliveira et al., 2013, 2016; Pant et al. 2010, 2012). In the past decade many advances have been made in MFC research aimed at enhancing MFC power density output (Logan et al., 2006a; Min et al., 2005; Park and Zeikus 2003; Rabaey et al., 2004). However, MFC construction, operation and maintenance cost still need to be minimized to make this technology sustainable on economical ground (Logan and Regan, 2006; Zuo et al., 2008).

The characteristics of the membrane separator selected can significantly affect the performance of a MFC (Zhang et al., 2009). Nafion is the most widely used membrane separator because of the high proton conductivity (Harnisch et al., 2008; Logan et al., 2006a), but is not necessarily the best separator under

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neutral pH conditions used in MFCs. The high cost of Nafion makes the potential production cost of MFC commercially unacceptable (Pant et al., 2010). Many studies explore use of various other separators for MFC (Behera et al., 2010a; Lefebvre et al., 2011; Martin et al., 2010). For example, the use of a salt bridge (Min et al., 2005), ultrex membrane (Rabaey et al., 2004), a porcelain septum made from kaolin (Park and Zeikus, 2003), anion exchange membranes (Martin et al., 2010), bipolar membranes (Ter Heijne et al., 2006), microfiltration membranes (Sun et al., 2009), J-Cloths (Fan et al., 2007), a selemion HSF membrane (Logan et al., 2006a), ultrafiltration discs (Lefebvre et al., 2011) as separators in MFCs has been reported. We recently (Behera et al., 2010a, 2010b) reported on the treatment of synthetic and rice mill wastewater in fed-batch MFCs fabricated using an earthen pot acting as both anode half-cell container, and a membrane separator, with the performance compared to MFCs fabricated using a Nafion membrane separator (Jana et al., 2010). The earthen pot MFCs demonstrated better performance in terms of organic matter removal and electricity generation compared to the Nafion membrane, but the surface area available for ion transfer through the wall of earthenware was much larger than that in the Nafion-based MFC, making direct comparison difficult in that study. The thickness of the earthen pot membrane/anode container was also shown to affect both MFC internal resistances, and as a consequence MFC power generation (Behera and Ghangrekar, 2011).

The present paper focuses on an effort to better compare performances of MFCs operated using the earthen plate as membrane separator to that of Nafion separated MFCs; by fabrication of polyacrylate MFCs with earthen plate separator of the same exposed geometrical surface area to that of the Nafion separator. The comparison of MFC performance is evaluated by operation of both types of MFC under continuous mode feeding of acetate as synthetic waste, following initial batch incubation with acetate and anaerobic sludge, as inoculums, and using a model ferricyanide solution as oxidant in the catholyte.

2. Material and methods

2.1. Experimental set-up

The study was carried out in two identical up-flow dual chambered polyacrylic MFCs separated by the same exposed area of either a Nafion117 proton-exchange membrane (PEM, Sigma Aldrich) or an earthen plate membrane (EP, 3.7 mm thick). The earthen plates were prepared from soil (elemental composition: Al-32%, Si-47.80%, K-4.90%, Ca-1.20%, Ti-0.90%, Fe-13%) sourced from Midnapore, India and baked at 550-650°C for 6.0 hours, as described previously (Behera et al., 2010a). The cylindrical MFCs consisted of a cylindrical outer catholyte chamber separated from a rectangular polyacrylic inner anolyte chamber, with membranes inserted in two, opposite, faces of the inner chamber (Fig. 1). Each membrane was of 20 cm^2 surface area, making the total exposed membrane surface area of 40 cm^2. The working volume of the anolyte chamber of both MFCs was 350 mL, whilst that of the catholyte chamber was 1200 mL.

Two graphite plates of 70 cm^2 each were used as electrodes and placed in the anolyte and catholyte, giving a total surface area of 140 cm^2 each for anode and cathode in all MFCs. The electrodes were connected externally with titanium wire through an external resistance of 100 Ω. The cell culture medium anolyte was supplied to the MFCs from the bottom of the anode chamber using a peristaltic pump (Gilson, France) with effluent exiting at the top of the reactor. The catholyte consisted of 50 mM ferricyanide in 100 mM phosphate buffer (pH 7.0).

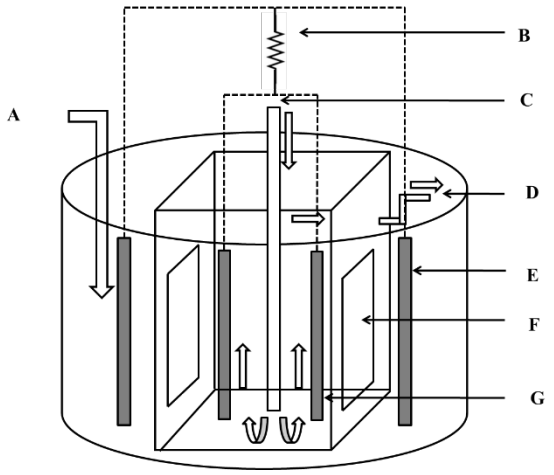

Fig. 1. Schematic diagram of the MFCs used in the study: (A) $K_3Fe(CN)_6$ catholyte (B) external resistance load (C) cell culture medium anolyte influent (D) anolyte effluent (E) cathode (F) membrane (G) anode

2.2. MFC operation

The MFCs were inoculated with anaerobic sludge collected from an anaerobic digester (Mutton Island wastewater treatment center, Galway). The granular sludge was crushed in a grinder for 10 seconds and 60 mL of the resultant sludge was added to the anode chamber to establish a sludge loading at 0.75 kg COD kg VSS^{-1} (Behera and Ghangrekar, 2009). Synthetic media containing acetate as a source of electron donor having COD of 1250 mg L^{-1} was used as feed. The acetate medium also contained (per L): 0.1 g KCl, 0.15 g NH_4Cl, 0.6 g Na_2HPO_4, 2.5 g $NaHCO_3$, 10 mL trace element solution and 10 mL vitamin solution (Katuri et al., 2012b) prepared according to Dsmz.de. (2003) (medium No. 826). The feed pH of 7.2-7.5 was maintained throughout the experiments and the MFCs were operated at temperatures that varied from 28 to 32°C. Initially both the MFCs were operated under batch mode condition to encourage the attachment of electrogenic bacteria to the graphite plate anodes. The first batch consisted of the inoculum supplemented with 290 mL

of acetate medium, with the two subsequent batches prepared by replacement of half of the anolyte volume by acetate medium (no additional inoculums). After these three feed cycles, the MFCs were operated in continuous mode at organic loading rate (OLR) of 2.5 kg COD m^{-3} d^{-1}, by maintaining a hydraulic retention time (HRT) of 12 h using the peristaltic pump. After reaching a steady state voltage output across the 100 Ω resistors, the performance of PEM-MFC and EP-MFC was evaluated by polarization experiments using a CH instruments 650 (USA) potentiostat.

2.3. Analyses and calculations

The cell voltages were recorded continuously using a digital multimeter with data acquisition unit (Pico data logger, UK) throughout the batch and continuous feed experiments by connecting anode and cathode across a 100 Ω external resistor. Current (I) was calculated using Ohm's law (I = V/R) with MFC power estimated by P = IV, where I is cell current and V is the cell voltage. Polarization studies were carried out for the MFCs by linear sweep voltammetry at a scan rate of 1 mVs^{-1} using a potentiostat (CH instruments, USA) with a two electrode assembly system. Power density was normalized to the anode surface area. Internal resistance of the MFCs was estimated from the slope of linear portion of the plot of voltage versus current (Picioreanu et al., 2007).

The suspended solid (SS), volatile suspended solids (VSS), and chemical oxygen demand (COD) levels were monitored according to APHA standard methods (APHA, 1998). Chemical compositions of earthen plates were estimated from Scanning Electron Microscopy (SEM, Hitachi SU-70) images with Energy Dispersive X-ray Microanalysis (EDX, Oxford Instruments). Nitrogen absorption isotherms measured with an ASAP2010 adsorption analyser were used to estimate earthen plate membrane specific surface area, using the Brunauer–Emmett–Teller (BET) method. Pore sizes distribution curves were calculated by the BJH method from the desorption branch (Ye et al., 2011).

Oxygen mass transfer coefficient (ko, cm/s) for the earthen plate was calculated as described previously (Chae et al., 2008). Briefly, both reactor chambers were filled with 50 mM phosphate buffer solution and the dissolved oxygen (DO) content of the anode chamber solution removed by purging N_2 gas, whilst that of the cathode chamber solution was saturated by continuous supply of air. The change in DO of the anode chamber solution was monitored using a DO meter (Cyber Scan, CD 650, Eutech Instruments, Singapore) and k_o estimated using Eq. (1):

$$k_0 = -\frac{v}{At} ln\left(\frac{C_0 - C}{C_0}\right) \quad (1)$$

where v is the liquid volume of anode chamber, A is the earthen plate cross-sectional area, c_0 is the saturated DO concentration, and c is the DO concentration at time t. The oxygen diffusion coefficient (Do, cm^2/s) through the membrane was calculated as $Do = koL$, where L = thickness of earthen plate membrane.

The coulombic efficiency (CE) was estimated using the ratio of charge produced (by integrating the measured current over time) to theoretical charge on the basis of consumed COD, with the theoretical charge production estimated as (F x n x w)/M, where F is Faraday's constant, n is the number of moles of electrons produced per mole of substrate (= 4 for wastewater COD), w is the daily COD load consumed in gram and M is molar mass of acetate (Logan et al., 2006a).

3. Results and discussions

3.1. MFC operation

After inoculation, a gradual increase in the voltage produced across the 100 Ω external resistance load was observed in both the MFCs over the first 48 hrs, with a slight delay in response of the PEM-MFC compared to that for the EP-MFC, leading to generation of a maximum voltage of ~0.3 V over the first batch feed cycle, (Fig. 2). In this set-up, it is presumed that the cathode does not limit the current, as the ferricyanide catholyte was supplemented periodically over the time course of the experiments resulting in a constant electron acceptor concentration close to 50 mM, as reported on previously (Katuri et al., 2012a; Kong et al., 2010). After two further batch fed cycles, represented by the arrows in Fig. 2, the feed was switched to continuous mode on the 7^{th} day of operation, providing a flow of 20 mM acetate to the chamber (HRT 12 hrs). The measured cell voltage for both MFCs stabilized after this, with average sustainable current generation across 100 Ω external resistances over the 7 to 26 day continuous feed time period (Fig. 2) of 5.14 mA and 4.34 mA for the PEM-MFC and EP-MFC, respectively. The cells thus provide average power densities of 190 mW/m^2 and 138 mW m^{-2}, respectively, under these steady-state conditions. Under these conditions both cells establish the same OCV of 0.68 V. This OCV is close to the 0.76 V observed by Kong et al. (2010) for a PEM-MFC fed with acetate and using potassium ferricyanide solution as catholyte.

To examine the catalytic activity of the anodic biofilm slow scan cyclic voltammograms were recorded in situ in the cell culture medium. Sigmoidal shaped voltammograms shown in Fig. 3, permit estimation from the first derivative of the CVs (not shown) of acetate oxidation centered at −0.44 vs. Ag/AgCl. The sigmoidal shaped CV is indicative of catalytic oxidation of the substrate by the biofilm and heterogeneous electron transfer to the electrode, with similar responses reported on for acetate oxidation by anodic biofilms of *Geobacter sulfurreducens* (Katuri et al., 2010, 2012b; Marsili et al., 2010) and by mixed culture biofilms (Liu et al., 2008). From the difference in the ferricyanide reduction potential in buffer of

~+0.24 V vs. Ag/AgCl and that of the redox couple centred at −0.44 vs. Ag/AgCl an estimate of the cell voltage of ~0.68 V was obtained, agreeing well with the OCV measured for both cells.

Efficient proton and ion transfer between anolyte and catholyte is important in MFC systems, to maintain electro-neutrality during current flow. The ability of the earthen plate separation membrane to permit ion-exchange was therefore evaluated and compared to that of the Nafion PEM. For this experiment, 1 M H_2SO_4 was added to the anolyte chamber of both MFCs and the pH change in the catholyte, initially containing distilled, deionized, water was measured as a function of time.

The catholyte pH decreased with time as protons migrated from anolyte to catholyte with the extent of proton exchange through the earthen plate membrane similar to that observed for proton exchange through the Nafion separator (Fig. 4). The specific surface area of the earthen plate membrane, as determined by nitrogen absorption, is relatively high at 39.8 $m^2 g^{-1}$, whilst providing a pore volume of 0.068 $cm^3 g^{-1}$ and an average pore diameter of 19.2 Å, indicating mesoporous structure of the ceramic membrane (Colomer, 2006). For comparison, narrow hydrophilic pores of 10–60 Å diameter are reportedly available for mass transport in Nafion 117 (Fang et al., 2004).

3.2. Organic matter removal

Initially both MFCs were operated in batch mode to avoid the washout of the sludge inoculum and thus encourage attachment of bacteria to the anode surface. After ~7 days of operation in batch mode, MFC operation was switched to continuous mode at an OLR of 2.5 kg COD $m^{-3} d^{-1}$. Steady state conditions for stable substrate degradation, represented by COD removal, and electricity generation were already established at this stage (Fig. 5).

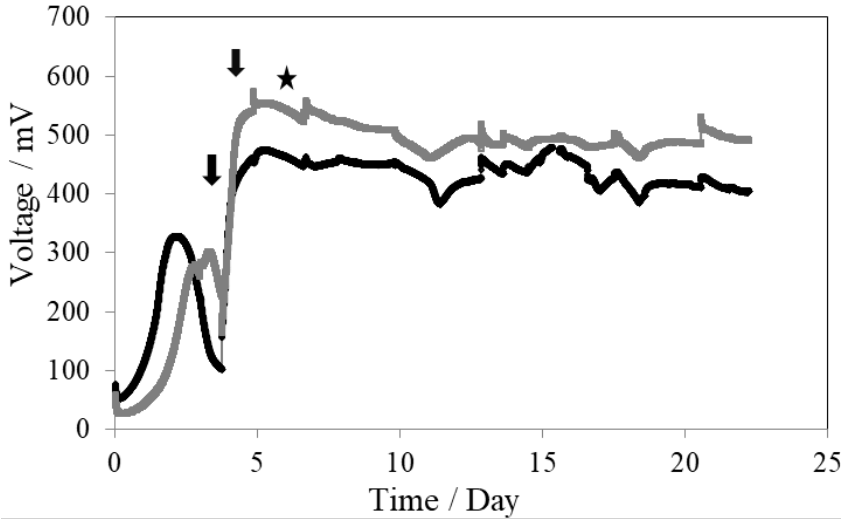

Fig. 2. Voltage generations as a function of time for the PEM-MFC (grey) and the EP-MFC (black). Arrows indicate replacement with fresh feed and star indicates the time when the reactors were switched from batch to continuous mode operation

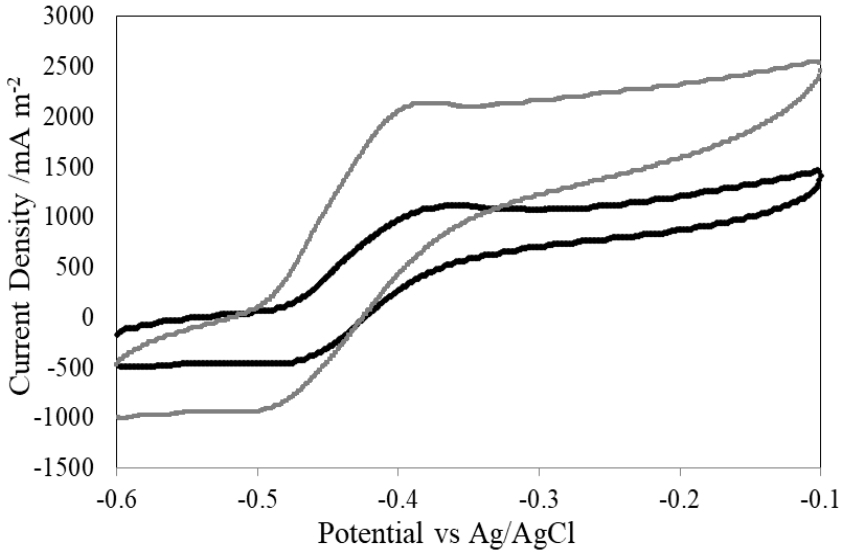

Fig. 3. Cyclic voltammograms (1 mV/s) of anodic biofilm, recorded in-situ on the 19th day of operation of the cells under 100 Ω external resistors, for the PEM-MFC (grey) and EP-MFC (black) systems

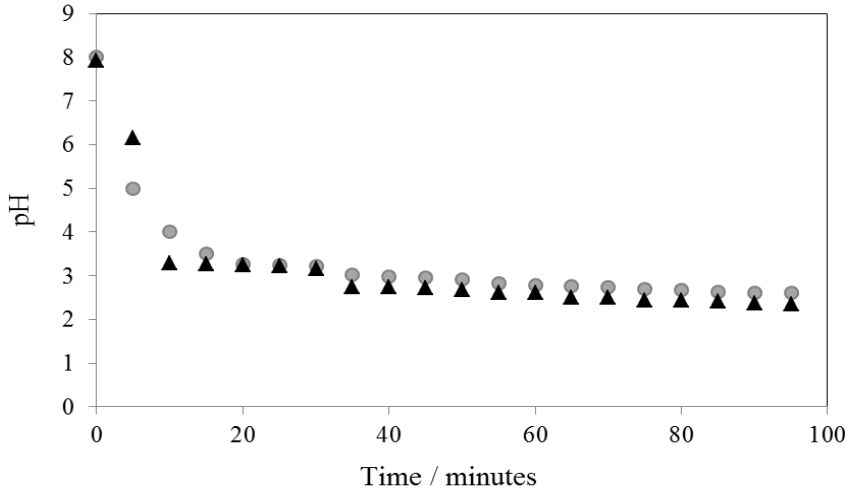

Fig. 4. A comparison of the change in pH of the catholyte chamber, initially containing distilled deionized water, as a function of time upon introduction of 1 M sulphuric acid into the anolyte chamber for PEM (grey circles) and earthen plate (black triangles) MFCs

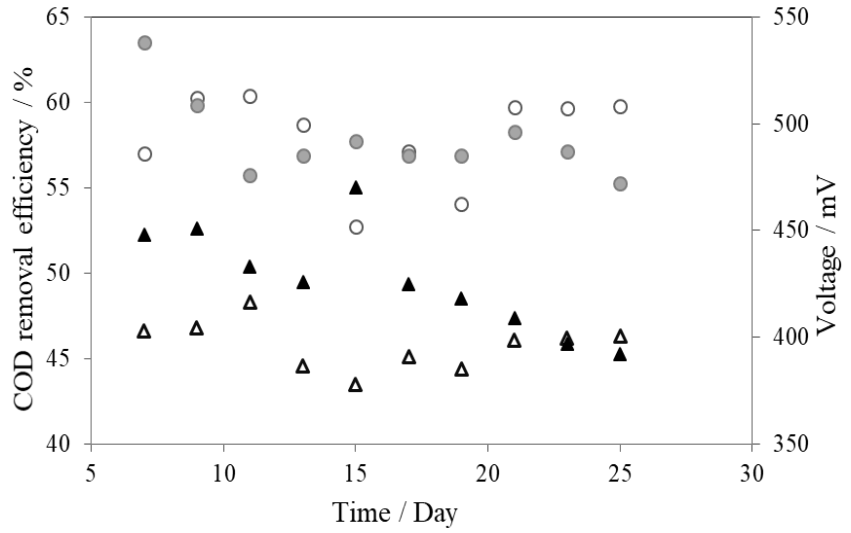

Fig. 5. COD removal efficiency (open) and voltage generation across the 100 Ω resistance (closed) of PEM-MFC (circles) and EP-MFC (triangles) as a function of time during continuous feed of MFCs (commenced at day 7)

During the subsequent steady state operation in continuous feed mode over an 18 day period, the PEM-MFC demonstrated an average COD removal efficiency of 60%, compared to an average COD removal efficiency of 48% for the EP-MFC. The earthen plate material may be permeable to oxygen (Behera and Ghangrekar, 2011), which could be one reason for the relatively lower COD removal efficiency for the EP-MFC compared to the PEM-MFC. An oxygen mass transfer coefficient, k_0, and oxygen diffusion coefficient, D_0, of 6.11×10^{-6} cm/s and 2.44×10^{-6} cm^2/s were determined, respectively, for the 3.7 mm thick earthen plate membrane, as described in the experimental section. Comparison of these values to those previously reported for oxygen diffusion through Nafion 117 of 0.175 mm thickness of 2.80×10^{-4} cm/s and 5.35×10^{-6} cm^2/s, respectively (Chae et al., 2008) implies that the earthen plate is not as permeable to oxygen as the Nafion membrane, and that this is not the major factor contributing to differences in COD removal efficiency between the two MFCs.

3.3. Polarization studies

At the end of the continuous feed period (day 25) polarization studies were carried out for the MFCs by slow scan linear sweep voltammetry yielding the polarization curves (Fig. 6) with a maximum power density of 250 mW m^{-2} at 35 Ω resistance for the PEM-MFC and 145 mW m^{-2} at 67 Ω resistance for the EP-MFC. Maximum volumetric power density of 10.0W m^{-3} and 5.8 W m^{-3} were estimated based on anolyte volume from the polarization curves for the PEM-MFC and EP-MFC, respectively.

Internal resistances of 36 Ω and 70 Ω were estimated from the slope of the linear portion of the plot of cell voltage versus current (Fig. 6), for the PEM-MFC and EP-MFC, respectively. The earthen plate membrane is substantially thicker (~3.7 mm)

than the Nafion 117 (~0.17 mm), which might contribute towards the higher internal resistance for these cells. The higher internal resistance, and thus lower current density during MFC operation is thus proposed to be the main factor contributing to the lower power density, and COD removal efficiency for the EP-MFC reactor, compared to the PEMFC. For example, Jung et al. (2007) report internal resistances of dual chamber MFCs using a range of membrane separators to be from 1239 to1344 Ω, depending on factors such as electrode spacing (Chae et al., 2008) and membrane, with higher internal resistance resulting in MFC with lower power densities in this configuration. Lower internal resistances of 84-98 Ω, and higher power densities, were obtained for MFCs in a single chamber, air-breathing cathode configuration with much closer electrode spacing (Ye et al., 2011). Using a dual chamber Nafion-separated MFC system similar to the configuration in this report, (Katuri et al., 2012a) report a maximum power density of 6.4 W m^{-3} based on anolyte volume for the MFC operating on slaughterhouse waste-waters, whilst others (Kong et al., 2010) observe maximum power density of 4.35 W m^{-3} using acetate as feed. Improvement in power density may be achieved, as reported on recently (Zhang et al., 2011) by use of high surface area anodes, glass fiber as a separator, in a single chamber, air-breathing cathode, MFC configuration, providing a power density of 75 W m^{-3}.

Table 1 shows the comparison of the performance of the EP-MFC and PEM-MFC. From the polarization curve and the data in Table 1 the EP-MFC generates ~40% less maximum power density, with an internal cell resistance 48% higher than the PEM-MFC. Over the 18 day period of steady state operation in continuous feed mode the same, although low (<10 %), coulombic efficiencies are estimated for both the PEM-MFC and the EP-MFC under the 100 Ω resistance load. Many different membrane separator configurations have been studied in MFCs, such as the use of single chamber porous air cathode (Park and Zeikus, 2003), tubular air cathode system with an outer cathode and an inner anode (Rabaey et al., 2005), and more recently (Zhang et al., 2010) nylon and glass fiber filter separators in air-cathode single-chamber microbial fuel cells.

A potential disadvantage to all these configurations is the susceptibility of the membrane separators to degradation (Zhang et al., 2009) or mechanical deformation (Zhang et al., 2011) over long periods. The use of an earthen plate separator as an alternative membrane provides a low cost, durable, option, with little loss in performance compared to the same exposed area of a Nafion PEM as a benchmark.

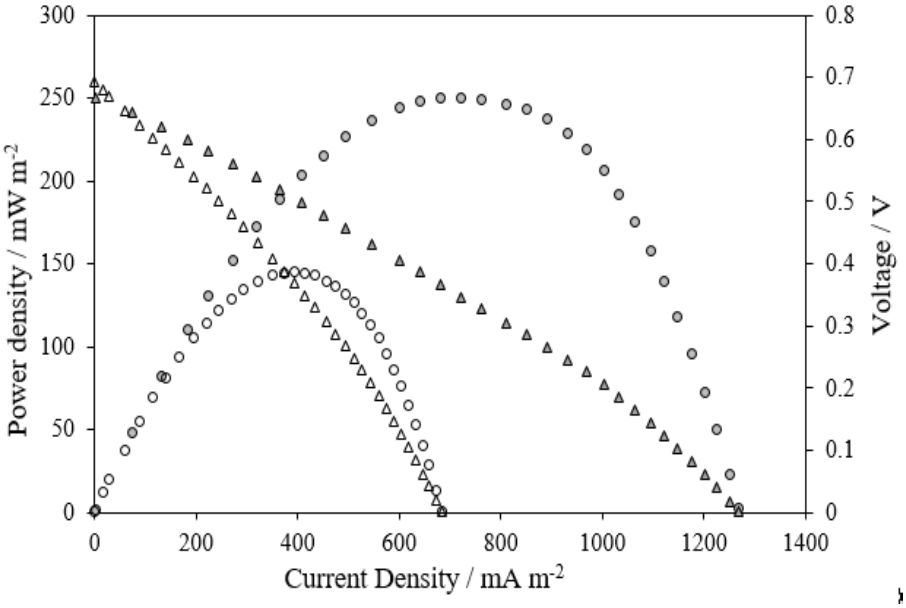

Fig. 6. In-situ polarization curves, recorded on day 25, cell voltage and power density versus current density for PEM (grey closed) and earthen plate (black open) MFCs

Table 1. Comparison of performance of PEM-MFC and EP-MFC

MFCs	*Voltage with 100 Ω load (V)*	*Power density with 100 Ω load (mW m^{-2})*	*Maximum Power density (optimum resistance) (mW m^{-2})*	*Volumetric power density with 100 Ω load (W m^{-3})*	*Maximum volumetric power density (optimum resistance) (W m^{-3})*	*Internal Resistance (Ω)*	*Maximum Coulombic Efficiency (%)*
PEM-MFC	0.52	190	250 (35 Ω)	7.6	10.0 (35 Ω)	36	25
EP-MFC	0.44	138	145 (67 Ω)	5.5	5.8 (67 Ω)	70	17

4. Conclusions

This study demonstrated by comparison to Nafion, that an earthen plate membrane may provide a low-cost, more durable, alternative to Nafion in continuous flow MFCs. The MFC using Nafion membrane as separator showed slightly better performance in terms of organic matter removal and electricity generation than the MFC based on earthen plate membrane separators; however both show comparable coulombic efficiency.

The performance of earthen plate MFC may be enhanced by increasing the membrane surface area or by decreasing the membrane thickness. The 40 cm^2 Nafion 117 membranes used in the present experiment costs approximately €30.0 whilst the same area of the earthen plate costs approximately €0.025, making it 99% cheaper. Utilization of such low cost separators may contribute to further developing economical MFC technology.

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