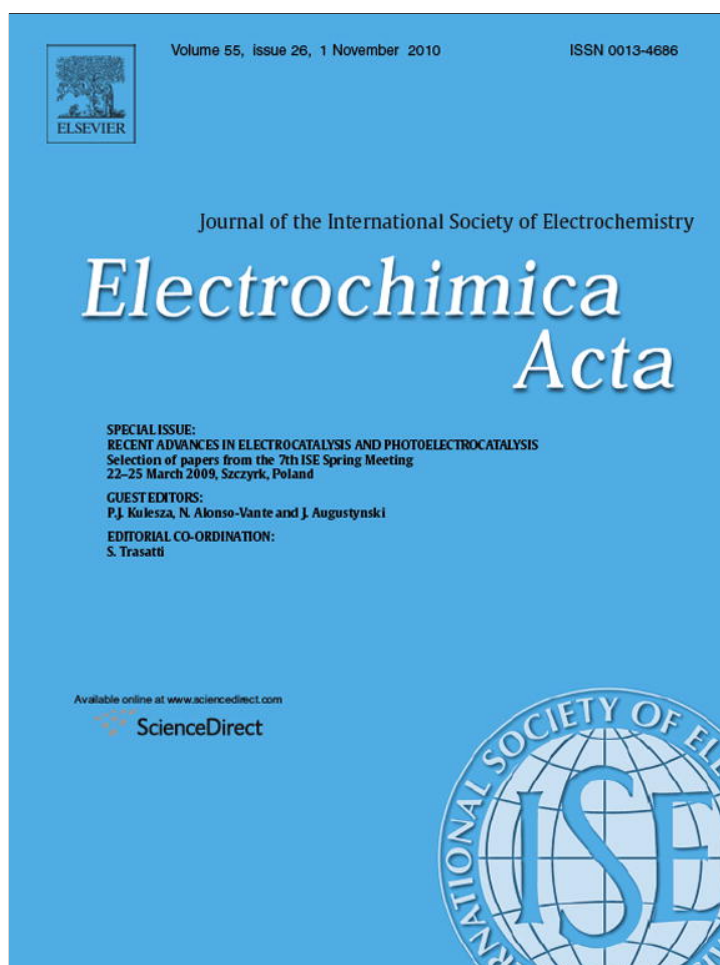


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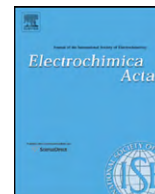


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Use of novel permeable membrane and air cathodes in acetate microbial fuel cells

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ARTICLE INFO

Article history:

Received 25 April 2009

Received in revised form

24 November 2009

Accepted 26 November 2009

Available online 1 December 2009

Keywords:

Microbial fuel cell

Zirfon® membrane

Non-platinized electrode

Air cathode

ABSTRACT

In the existing microbial fuel cells (MFCs), the use of platinized electrodes and Nafion® as proton exchange membrane (PEM) leads to high costs leading to a burden for wastewater treatment. In the present study, two different novel electrode materials are reported which can replace conventional platinized electrodes and can be used as very efficient oxygen reducing cathodes. Further, a novel membrane which can be used as an ion permeable membrane (Zirfon®) can replace Nafion® as the membrane of choice in MFCs. The above mentioned gas porous electrodes were first tested in an electrochemical half cell configuration for their ability to reduce oxygen and later in a full MFC set up. It was observed that these non-platinized air electrodes perform very well in the presence of acetate under MFC conditions (pH 7, room temperature) for oxygen reduction. Current densities of -0.43 mA cm^{-2} for a non-platinized graphite electrode and -0.6 mA cm^{-2} for a non-platinized activated charcoal electrode at $-200 \text{ mV vs. Ag/AgCl}$ of applied potential were obtained. The proposed ion permeable membrane, Zirfon® was tested for its oxygen mass transfer coefficient, K_0 which was compared with Nafion®. The K_0 for Zirfon® was calculated as $1.9 \times 10^{-3} \text{ cm s}^{-1}$.

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1. Introduction

Bioelectrochemical systems (BES) such as microbial fuel cells (MFCs) and microbial electrolysis cells (MECs) represent a promising technology for renewable energy production in form of electricity and hydrogen respectively. Besides they might have specific applications such as H_2O_2 production, chemical modification and CO_2 conversion to methane and/or electricity [1–4]. MFCs specifically might have short-term applications in simultaneous wastewater treatment and electricity production. Microbial production of electricity may become an important form of bioenergy in future because MFCs offer the possibility of extracting current from a wide range of soluble or dissolved complex organic wastes and renewable biomass [5]. However, the power densities of existing lab-scale MFCs (except sediment MFC) are too low for most envisioned applications. In fact, low power densities, with the existing design, are the most challenging problem currently faced by MFCs [6,7]. These devices which are capable of converting waste to electricity basically rely on the use of platinum (Pt) as a catalyst in the electrode and a PEM as an ion exchange membrane between the cathode and anode. In most studies so far Nafion® has been the membrane of choice [8]. Even the most recent literature men-

tions the use of cathodes with a Pt loading rate of 0.5 mg cm^{-2} [9]. The various challenges and constraints faced by oxygen cathodes in MFCs have been reviewed extensively [6,10]. Further, in order to achieve an efficient MFC operation (high cell voltage), a rapid and intense oxidation reaction at the anode (low anodic overpotential) and a very reactive and low cost cathode (high cathodic potential) is needed, the absence of which has caused cathode catalysis to be one of the limiting factors for MFC operation at the moment [11].

Since the use of Pt as a catalyst is not feasible due to the high cost and future scarcity, there is an urgent need for some low cost sustainable alternatives [12]. Moreover, the use of PEM might not be feasible owing to their high cost, potential for biofouling, associated ionic resistance and its permeability to oxygen [13,14].

The aim of the present study, is to reduce these severe limitations by using in-house developed non-platinized, gas porous carbon electrodes as air cathodes and a novel ion permeable membrane, Zirfon® in a dual chamber MFC with acetate as the sole carbon source. The electrodes were tested in electrochemical half cells and complete MFC configuration for checking their ability for oxygen reduction under different conditions of air flow, presence or absence of acetate and microorganisms. The oxygen diffusion through the proposed membrane, Zirfon® was tested to compare it with Nafion®. The ionic resistance of this membrane was also calculated and compared with that of Nafion® and Fumasep®. Finally, a cost comparison of the proposed materials is made with existing MFC costs.

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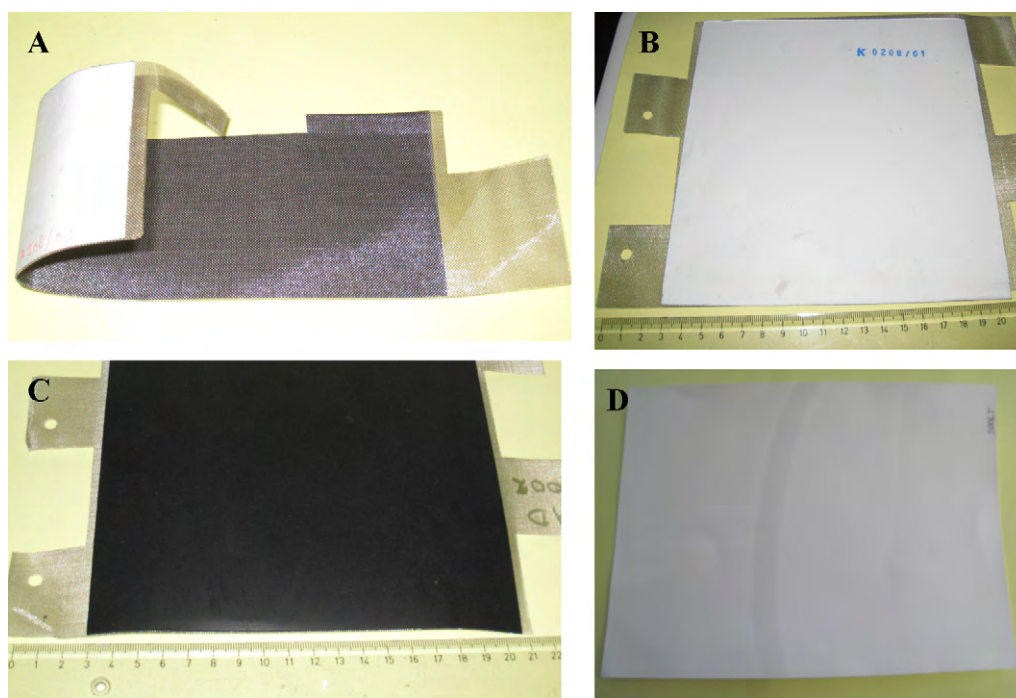


Fig. 1. Electrode and membrane used in this study. (A) PTFE layered activated charcoal electrode with current collector, (B) PTFE side of activated charcoal electrode, (C) activated charcoal electrode without PTFE layer, and (D) Zirfon® membrane.

2. Experimental

2.1. Buffer composition

Phosphate buffer solution (PBS) was composed of (mM): NH_4Cl (3.7), NaCl (6.8), $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (1.0), KH_2PO_4 (3.7) and yeast extract (10 mg L^{-1}). The pH was adjusted to 7.0. Ultrapure (Mili Q) filtered water was used for solution preparation, and all chemicals and reagents used were of analytical grade.

2.2. Novel gas porous electrodes for use as air cathodes

Two different types of gas porous electrodes were tested as air cathodes. These were VITO graphite electrodes (VG) and VITO activated charcoal electrodes (VAC). These electrodes were tested both with (5 wt.% Pt) and without any presence of platinum as catalyst (0 wt.% Pt). The electrodes were unique in design and structure with a metallic mesh current collector stretching out at the edges. The activated charcoal electrode with and without a porous gas diffusion layer made of polytetrafluoroethylene (PTFE) is shown in Fig. 1(A–C). For the purpose of comparison, some commercially available electrodes were also tested under similar conditions. These electrodes were stainless steel mesh, activated carbon with cobalt and carbon paper electrode (Ballard AVCarb). All electrodes had an active projected surface area of 10 cm^2 .

2.3. Novel membrane for use as ion permeable membrane

The membrane used was a Zirfon® separator. Prior to use, the Zirfon® membrane was pretreated for wetting it by warming in lukewarm water for 2 h and then stored in PBS. The Zirfon® membrane used in the present study is shown in Fig. 1D. The mass transfer coefficient is a common measurement MFC studies (in neutral pH medium) for checking the oxygen permeability of membranes/separators and the cathode. It is important that the membranes used do not allow oxygen to diffuse from the cathode compartment to the anode compartment. Oxygen mass transfer

coefficient, K_0 for Zirfon® was determined using a non-inoculated MFC reactor. Since this was done using an air cathode MFC, the cathode gas compartment was continuously fed with a constant flow of air (21% O_2) (Fig. 2). The electrolyte in the feeding tank was continuously sparged with nitrogen gas to remove any dissolved oxygen (DO). The mass transfer coefficient of oxygen in the membrane, K_0 , was determined by measuring the DO concentration over time and using the equation [13]:

$$k_0 = -\frac{v}{At} \ln \left[\frac{C_s - C}{C_s} \right]$$

where V is the liquid volume (cm^3) in the electrolyte loop, A is the membrane cross-sectional area (cm^2), C_s is the saturated oxygen concentration (8 mg L^{-1}) in the electrolyte chamber, and C is the actual DO in the electrolyte chamber at time t (s). DO was measured using a Multi-340i OxiCal-SL probe (WTW, Germany). Other membranes, i.e. Nafion® (purchased from DuPont, USA) and Fumasep® (Fumatech GmbH, Germany) were used for comparison in this study.

2.4. Linear sweep voltammetry for characterization of single electrodes in half cells

The electrolyte used for these measurements was PBS as mentioned above. Characterization of electrodes as oxygen reducing (cathode) was carried out at room temperature in an electrochemical half cell with an active electrode surface area of 10 cm^2 . The schematic diagram of the half cell is shown in Fig. 2. A Pt disk laser welded on a Titanium (Ti) plate was used as a counter electrode. An Ag/AgCl -3 M KCl (+199 mV vs. SHE) was used as a reference electrode (Radiometer Analytical, France). Air (21% O_2) was fed to the air compartment at the PTFE side of the electrode at an overpressure of 5 mbarg. Electrochemical measurements were performed using a potentiostat type PRT 20-2 (Tacussel Electronique, France) connected to a function generator type GSTP (Tacussel Electronique, France). The cathodic polarization curves for the different electrodes were prepared by changing the potential of the working

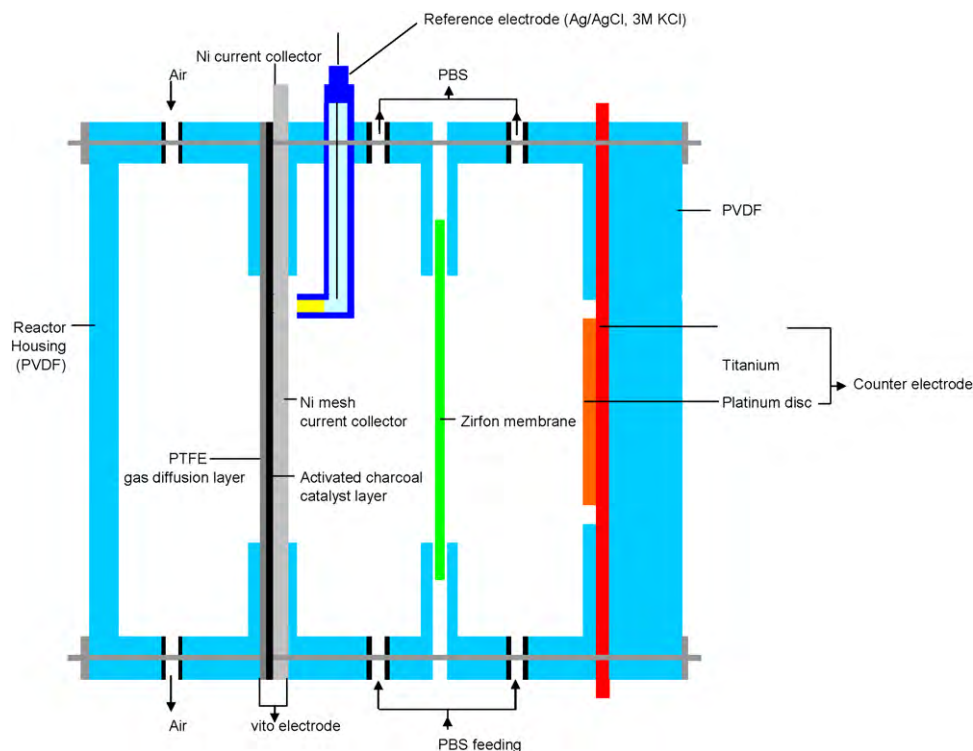


Fig. 2. Schematic representation of the half cell assembly for testing electrode for oxygen reduction and membrane for oxygen diffusion.

electrode (cathode) starting from the open-circuit potential (OCP) to the near redox potential of hydrogen ($-550\text{ mV vs. Ag/AgCl}$) at a scan rate of 1 mVs^{-1} . The current was measured over a shunt resistance in the counter electrode loop.

A similar experiment was carried out under nitrogen flow at 5 mbarg as well to prove that it was indeed the oxygen which was being reduced at the electrode.

2.5. Microbial culture, medium and electrode characterization in full MFC

These measurements were performed in a MFC which has been in operation for 10 months. The anode was a carbon paper, cathode was non-platinized VG electrode and Fumasep was used as membrane. This MFC was inoculated with a mixed consortium of three natural mixed culture inocula namely windrow yard waste compost, thermophilic anaerobic compost of source separated kitchen waste plus not recyclable waste paper (IGEAN, Brecht, Belgium) and activated sludge from a sewage treatment plant (Aquafin, Desel, Belgium) [15]. Later, a pure culture of *Geobacter sulfurreducens* strain (No. 12127) procured from the DSMZ culture collection was also added to the inoculum mix. The medium consisted of sterilized PBS as described above. Sodium acetate (10 mM) and trace metal solution (1 mL L^{-1}) were further added to the PBS medium through a $0.22\text{ }\mu\text{m}$ filter. The final medium was flushed with N_2/CO_2 (80:20) for 30 min prior to use. All experiments were performed at a room temperature ($25\text{ }^\circ\text{C}$) and ambient atmospheric pressure. Monitoring and control of the cell was partially automated. Anode potential, cathode potential and cell voltage were monitored continuously by using LabVIEW Signal Express 2.5 software interfaced with a National Instrument data acquisition card (N-cDAQ-9172). Both the electrode potentials were measured against one Ag/AgCl-3 M KCl reference electrode which was placed inside the anodic compartment, in close proximity of the anode. The power density curve in the full MFC was calculated from voltages and currents obtained by varying the external resistance between anode and cathode

from 1500 to $3\text{ }\Omega$, with resting time at each resistance at least 15 min.

2.6. Resistance measurement of the membranes

In order to check the specific ionic resistance (ρ) of Zirfon®, Nafion® and Fumasep® membranes, a set up was designed with two electrodes (Ti and Pt laser welded on Ti) with one of these membranes (10 cm^2 active area) separating them. The distance of each electrode from the membrane was 2 cm. Both the compartments were filled with PBS (pH 7). Ag/AgCl-3 M KCl reference electrode were placed on each side of the membrane. A constant current of 5 mA was maintained between the two electrodes using the potentiostat in intensiostat mode.

3. Results and discussion

The maximum current densities at $-200\text{ mV vs. Ag/AgCl}$ obtained with different electrodes when tested for oxygen reduction under half cell conditions, are shown in Table 1. Since the oxygen evolving at the counter electrode will diffuse through the electrolyte towards the working electrode and interfere with the measurements performed there, the counter electrode was

Table 1

A comparison of maximum current produced by different electrodes at an applied voltage of $-200\text{ mV vs. Ag/AgCl}$ under air flow (RT, 5 mbarg).

Air electrode	Reduction current ($\text{J}/\text{mA cm}^{-2}$)
Stainless steel	-0.043
Activated carbon + Co	-0.440
Carbon paper (Pt)	-0.004
Graphite + Pt-black	-0.750
VG electrode with PTFE layer (5 wt.% Pt)	-0.750
VG electrode with PTFE layer (0 wt.% Pt)	-0.425
VAC with PTFE layer (5 wt.% Pt)	-1.200
VAC with PTFE layer (0 wt.% Pt)	-0.607

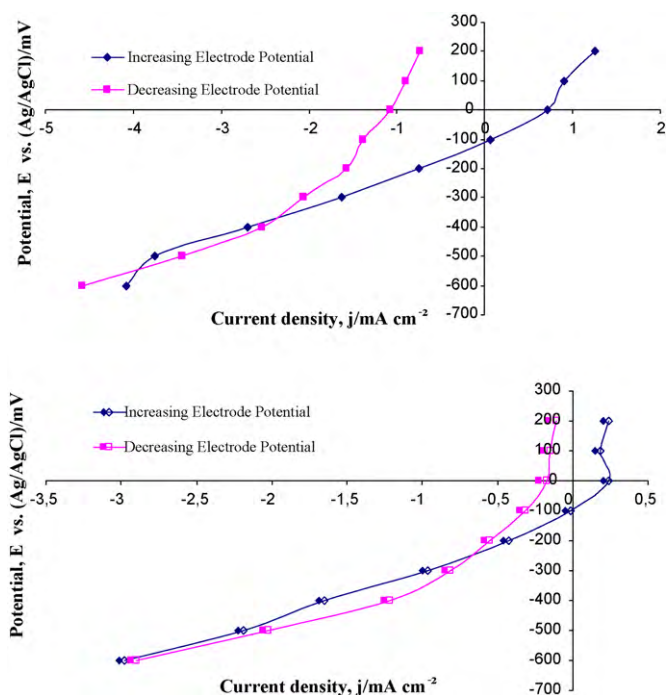


Fig. 3. Polarization curve of (A) VG electrode (5 wt.% Pt); (B) VG electrode (0 wt.% Pt) under air flow (5 mbarg) and PBS (pH 7) as electrolyte, RT.

separated from the working electrode with a Zirfon® separator (Fig. 2).

Fig. 3a and b, respectively, shows the cathodic polarization curves of VG electrode with 5 wt.% Pt ($0.25 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$ electrode surface area) and without the presence of Pt. As can be expected and seen from Fig. 3a and b, at every voltage point, the current density was expectedly higher for graphite with 5 wt.% Pt than for graphite without Pt. At $-200 \text{ mV vs. Ag/AgCl}$, the current density for graphite (5 wt.% Pt) was -0.75 mA cm^{-2} while the corresponding figure for non-platinized graphite was $-0.425 \text{ mA cm}^{-2}$. Fig. 4a and b shows the cathodic polarization curves of VAC electrode (5 wt.% Pt) under air flow and nitrogen flow, respectively. The current density was significantly low in case of nitrogen flow thus proving that the high current density obtained under the air flow was indeed due to the reduction of oxygen at the electrode. At $-200 \text{ mV vs. Ag/AgCl}$, a current density of -1.20 mA cm^{-2} was observed under air flow mode while the corresponding figure for nitrogen flow was -0.31 mA cm^{-2} .

In order to check the effect of presence of acetate, trace elements and microbial inoculum which are normally present in a MFC, on the performance of these electrodes for oxygen reduction, some additional tests were performed in the presence of these anodic materials. In general, the presence of acetate and microorganisms had a positive influence on the performance of the proposed electrodes as air cathodes. Fig. 5 shows the polarization curve of VAC electrode (5 wt.% Pt) under air flow and PBS, 10 mM acetate, trace elements and microbial inoculum as electrolyte. The current density (-4.14 mA cm^{-2} at $-200 \text{ mV vs. Ag/AgCl}$) was highly improved compared to the situation when only PBS was used as electrolyte (-1.2 mA cm^{-2}). Fig. 6a and b shows the cathodic polarization curves of VAC electrode (0 wt.% Pt) under air flow and nitrogen flow, respectively. As mentioned before, the current density was much lower under nitrogen flow than air flow. At $-200 \text{ mV vs. Ag/AgCl}$, a current density of -0.61 mA cm^{-2} was observed under air flow mode while the corresponding figure for nitrogen flow was almost ten times lower, 0.058 mA cm^{-2} . Also, these current values at $-200 \text{ mV vs. Ag/AgCl}$ are very obviously significantly lower (50%)

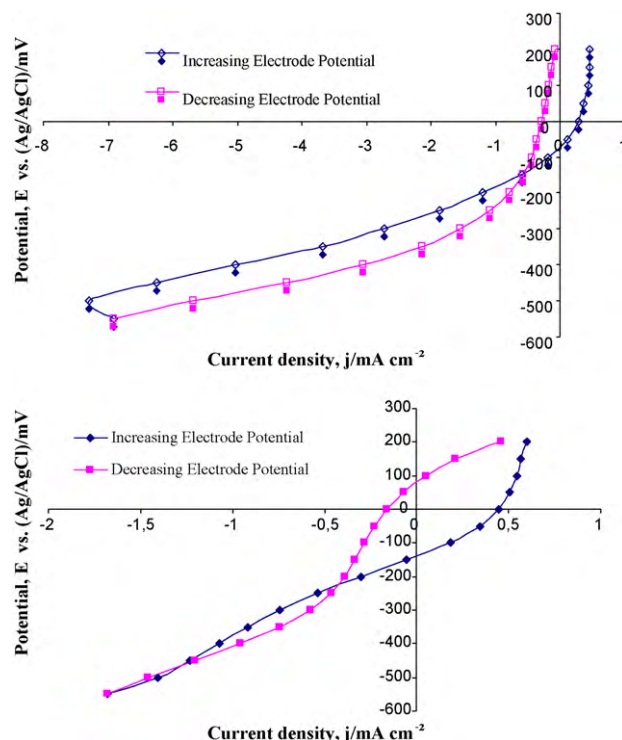


Fig. 4. Polarization curve of VAC electrode (5 wt.% Pt) under (A) air flow (5 mbarg); (B) nitrogen flow (5 mbarg) and PBS (pH 7) as electrolyte, RT.

than the ones obtained for VAC electrode (5 wt.% Pt) under similar conditions.

The application with an air cathode to use the oxygen freely available in the air as electron acceptor has been a remarkable improvement in MFC research, which has advantages over the chemical electron acceptors in terms of reduced costs and improved process sustainability [16]. However, MFCs using oxygen as electron acceptor suffer from limitations such as (i) high cathodic overpotential and (ii) inability of cation exchange membranes (CEMs) to selectively transport protons due to competition with other available cations (Ca^{2+} , Na^+ , Mg^{2+}) leading to proton build up in the anode thereby lowering the pH and reduced activity of bacteria in the anodic biofilm area for substrate oxidation [17,18]. To overcome these limitations, efforts have been made in the past to develop electrodes for cathodic oxygen reduction having low cost, high electrochemical activity and long stability. Very recently, the use of pyrolyzed and plasma-treated non-precious catalysts for oxygen reduction were evaluated for use in MFCs [19]. Among dif-

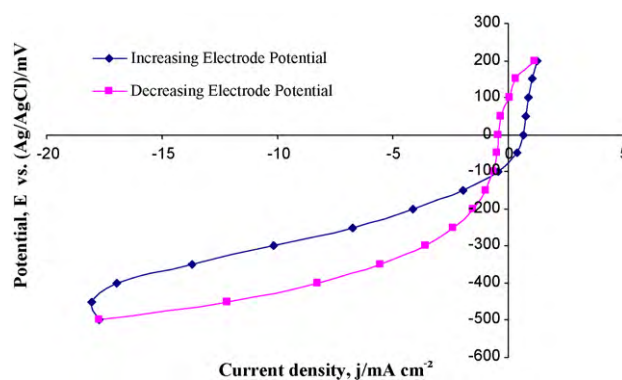


Fig. 5. Polarization curve of VAC electrode (5 wt.% Pt) under air flow (5 mbarg) and PBS, 10 mM acetate, trace elements and microbial inoculum as electrolyte, RT.

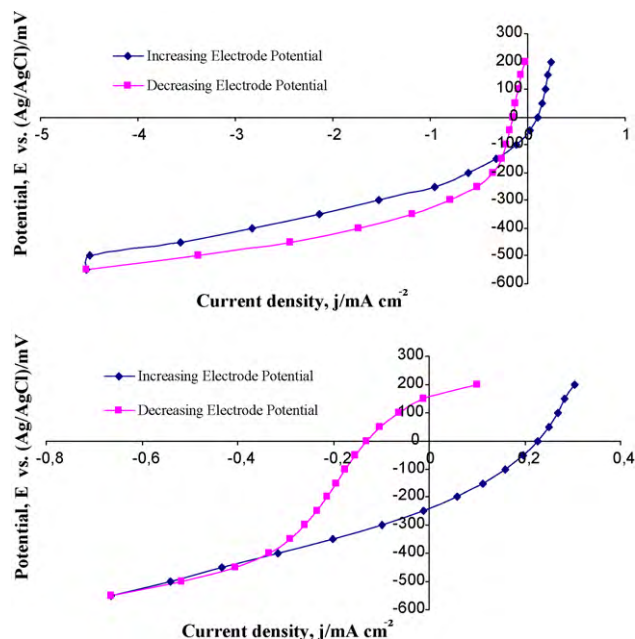


Fig. 6. Polarization curve of VAC electrode (0 wt.% Pt) under (A) air flow (5 mbarg); (B) nitrogen flow (5 mbarg) and PBS (pH 7) as electrolyte, RT.

ferent supports tested, Vulcan XC-72 having the highest surface area (BET of $220 \text{ m}^2 \text{ g}^{-1}$) showed the best current density at 0 V vs. Ag/AgCl ($-0.675 \text{ mA cm}^{-2}$). The electrodes based on activated charcoal mentioned in the present study had an active surface area of more than $10,000 \text{ m}^2 \text{ g}^{-1}$ due to the inherent structure of the material and the reactive organic groups that are building up this large BET-surface. This could account for their high electrochemical activity for oxygen reduction. This seem to agree with a previous finding that the overpotential towards oxygen reduction can be diminished by the use of high specific surface areas to increase the reaction interface instead of applying a catalyst [20]. Also this is one of the first reports for the use of activated charcoal being used as a catalyst for oxygen reduction in MFCs. Previously, three different carbon-based materials, activated carbon cloth (ACC), carbon fiber veil (CFV), and graphite foil (GF) were used as anode materials for in situ electrochemical sulfide oxidation in a MFC [21]. A maximum current density of 2.2 mA cm^{-2} was obtained with the MFC configuration with the ACC anode in combination with a platinumized ELAT cathode. Other effort to replace Pt at the cathode involved a coating of carbon cloth with iron(II)phthalocyanine or cobalt tetramethoxyphenylporphyrin (CoTMPP) as catalyst suspended in an air sparged solution [22].

Another interesting feature of the proposed electrode structure is the integration of a current collector in the form of a metallic (SS, Ni, Ti, etc.) mesh uniformly in contact with the active catalyst layer and stretching out at the edges (Fig. 1). Conventionally, the MFCs use either a wire or rod as current collector [23,24] which are not so efficient in transferring the electrons from the anode to the external circuit since part of the electrode which is not in contact with the current collector is not able to transfer its electrons as efficiently. The use of edge current collectors in these novel electrodes solves that problem. Also these edges allow to connect several single cells electrically in series and/or parallel to make a large reactor unit or stack.

The cell voltage–current density ($V-I$) curve and power density–current density ($P-I$) curve for the MFC inoculated with mixed consortium including *G. sulfurreducens* and non-platinized VG electrode as air cathode (70 cm^2) as described in Section 2.5 above, with 10 mM acetate as substrate is shown in Fig. 7. The

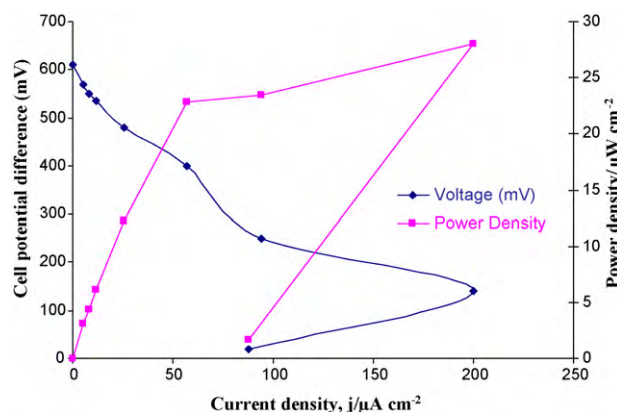


Fig. 7. A $V-I$ and $P-I$ curve for the MFC with carbon paper anode, acetate and mixed consortium including *G. sulfurreducens* as anolyte and non-platinized VG electrode as air cathode.

maximum power achieved was $28 \mu\text{W cm}^{-2}$ at 140 mV cell voltage and 0.2 mA cm^{-2} of current density. This was much higher than the power reported ($1 \mu\text{W cm}^{-2}$) using a cathode electroplated with Pt at a loading rate of 0.15 mg cm^{-2} and *Shewanella oneidensis* as the microbial inoculant [25]. However, in the present study, after reaching the maximum power, the performance dropped suddenly. This could be attributed to the fact that at this power density, due to high rate of acetate oxidation, there is an intense localized proton accumulation within the anode biofilm leading to an overall acidification of the anode medium [26,27]. It has been reported recently that dropping the pH of the culture medium from 7 to 6 severely limits the growth of *G. sulfurreducens* [27]. Using confocal laser scanning microscopy (CLSM) and atomic force microscopy (AFM), anodic biofilms up to $100 \mu\text{m}$ were measured in this MFC [28].

The Zirfon® separator was used as a replacement of conventional ion exchange membranes namely, Nafion® and Fumasep®. The standard type of Zirfon® separator is a macroporous organo-mineral material containing 85 wt.% of a hydrophilic ZrO_2 powder with a high specific surface area ($22 \text{ m}^2 \text{ g}^{-1}$) to assure an optimum wettability and 15 wt.% polysulfone (PSF) which gives the material its mechanical strength [29,30]. The properties of the novel ion permeable membrane, Zirfon® are given in Table 2.

The oxygen diffusion through the assembly of the two electrodes (anode and cathode) and Zirfon® membrane in three different runs is shown in Fig. 8. The resulting oxygen mass transfer coefficient (K_0) for Zirfon® was calculated as $1.9 \times 10^{-3} \text{ cm s}^{-1}$ which is comparable with K_0 values reported for a cathode coated with 4 gas diffusion layers ($2.3 \times 10^{-3} \text{ cm s}^{-1}$) [31]. This is despite the fact that the mode of oxygen diffusion in Zirfon® and gas diffusion layers is quite different. In case of diffusion layers, it is the oxygen in gaseous form which is diffusing through the cathode, while in case of Zirfon® the diffusion is in the form of dissolved oxygen due to the high hydrophilicity of Zirfon®. It has been reported that the oxygen flux through the air cathode (Nafion® bounded platinumized

Table 2
Properties of the novel ion permeable membrane, Zirfon® (500 μm).

Feature	Value
Permanently hydrophilic	Perfect wettable in aqueous solutions
Dimensionally stable	No shrinkage effects
Robust; flexible	Can be reinforced with a fabric
High gas bubble point	>7 barg
Mean pore size	0.08 μm
Ionic resistance	2727 $\Omega \text{ cm}$ in PBS, pH7, RT
Can be casted on electrodes	Planar, tubular
Thickness	Between 250 and 1000 μm

Table 3

A cost comparison of major components of a MFC.

Components	Present material cost (€/m ²)	Future material cost (assumed) (€/m ²)	Cost of proposed material including manufacturing cost (€/m ²)
Anode	100 (graphite felt)	5 (Graphite paper)	40 (PTFE bounded activated charcoal + current collector)
Membrane	400 (Nafion®)	10 (CEM)	45 (Zirfon®)
Cathode	500 (Pt catalyzed)	5 (Biocathode)	52 (activated charcoal + PTFE layer + current collector)
Current collector	25	10	–

The present and future values as given in column 2 and 3 are from Rozendal et al. [35].

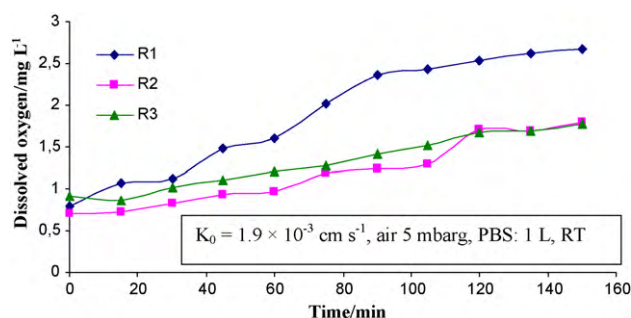


Fig. 8. Oxygen diffusion through the assembly of electrodes and Zirfon® membrane in an uninoculated MFC.

carbon cloth) is 2.7 times higher than that through the same cathode containing a Nafion® PEM [32]. The K_0 for Nafion® 117 has been reported as $2.8 \times 10^{-4} \text{ cm s}^{-1}$ [13]. However, when we tested our proposed gas porous activated charcoal electrode without the presence of Zirfon® membrane, the oxygen mass transfer coefficient (K_0) was $32 \times 10^{-3} \text{ cm s}^{-1}$, an increase of 16.8 times than the situation when Zirfon® was present. This high oxygen flux through the cathode can be expected as these are highly gas porous electrodes. Furthermore, when this membrane was used in place of a conventional PEM (Nafion® or Fumasep®), there was no reduction in the performance of the electrodes for oxygen reduction at the cathode and acetate oxidation at the anode.

Under similar measuring conditions, the specific resistance (ρ) of PBS was measured as $354 \Omega \text{ cm}$ (2.82 mS cm^{-1}). ρ for Zirfon® (550 μm) was calculated as $2727 \Omega \text{ cm}$ (0.365 mS cm^{-1}). The ρ values for Fumasep® (100 μm , PBS, RT) and Nafion® (100 μm , PBS, RT) were $16,000 \Omega \text{ cm}$ (0.062 mS cm^{-1}) and $17,000 \Omega \text{ cm}$ (0.058 mS cm^{-1}), respectively.

These electrodes and membranes were tested on MFCs which were always fed with acetate as the substrate since acetate is the end product of several metabolic pathways for higher order carbon sources (including the Entner–Doudoroff pathway for glucose metabolism) [33]. However, it is possible to use them in systems where other substrates are being used.

The high cost of cathodes due to the presence of Pt is one of the major drawbacks for its large-scale application. Even though relatively low Pt loadings of 0.1 mg cm^{-2} perform almost as well as loadings of 0.5 mg cm^{-2} [34], the prices for chemical cathode catalysts with precious metals remain high. A cost comparison was made between the proposed novel materials for use in MFC with the existing costs and future proposed costs [35]. As can be seen from Table 3, the cost of our studied materials is significantly less compared to the materials which are currently in use in MFC research and there is a possibility of reducing these even further with improvements in their design and material built-up.

4. Conclusions

It can be expected that use of these sustainable low cost air cathodes and ion permeable membranes will overcome several of the current limitations in the development of large-scale MFCs,

next to the development of an efficient reactor design. The use of these novel electrodes is expected to remove one of the major bottlenecks, which is the lack of an efficient, low cost, gas porous electrode in MFC research. Another innovation which is being explored currently is integrating the Zirfon® membrane directly onto the electrode (anode and/or cathode) surface thereby reducing the spacing between the electrode and the ion permeable membrane substantially and then using this sandwich type configuration in the MFC. It is also possible to use the same Zirfon® coated electrode in such a way that an extra feeding loop of PBS is introduced between anode and cathode leading to even lower oxygen diffusion into the anode compartment.

Acknowledgement

This research is supported by a grant from Milieu en Energiotechnologie InnovatiePlatform (MIP) under the project 'Sewage Plus: Second life of sewage as a matrix for dilution of organic waste streams' (M8463).

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