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Regeneration of the power performance of cathodes affected by biofouling

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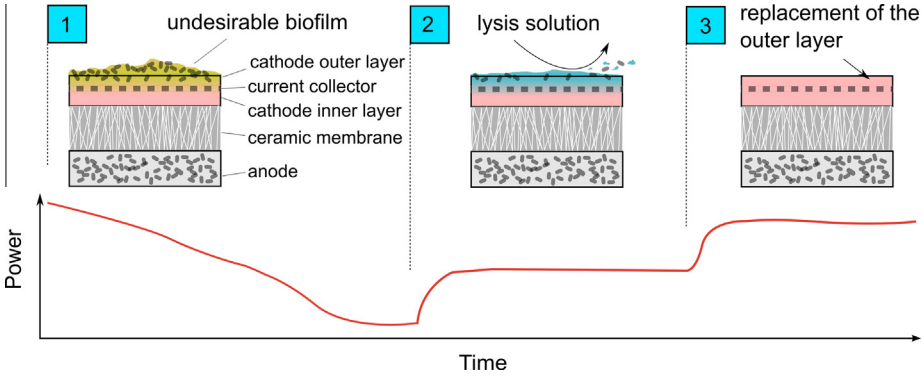
HIGHLIGHTS

- Detrimental biofilm growth on cathodes has been successfully removed, allowing full power recovery.
- An *in-situ* chemical regeneration method, for bio-fouled cathodes is proposed.
- Introducing multiple sacrificial layers for cathode electrodes, can be a good recovery mechanism.

GRAPHICAL ABSTRACT



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ABSTRACT

Air cathode microbial fuel cells (MFCs) were used in a cascade-system, to treat neat human urine as the fuel. Their long-term operation caused biodeterioration and biofouling of the cathodes. The cathodes were made from two graphite-painted layers, separated by a current collector. The initial performance of the MFCs was reaching average values of 105.5 ± 32.2 μW and current of 1164.5 ± 120.2 μA. After 3 months of operation the power performance decreased to 9.8 ± 3.5 μW, whilst current decreased to 461.2 ± 137.5 μA. Polarisation studies revealed significant transport losses accompanied by a biofilm formation on the cathodes. The alkaline lysis procedure was established to remove the biomass and chemical compounds adsorbed on the cathode's surface. As a result, the current increased from 378.6 ± 108.3 μA to 503.8 ± 95.6 μA. The additional step of replacing the outer layer of the cathode resulted in a further increase of current to 698.1 ± 130 μA. Similarly, the power performance of the MFCs was recovered to the original level reaching 105.3 ± 16.3 μW, which corresponds to 100% recovery. Monitoring bacterial cell number on the cathode's surface showed that biofilm formed during operation was successfully removed and composed mainly of dead bacterial cells after treatment. To the best of the authors' knowledge, this is the first time that the performance of deteriorating cathodes, has been successfully recovered for MFCs *in-situ*. Through this easy, fast and inexpensive procedure, designing multilayer cathodes may help enhance the range of operating conditions, if a biofilm forms on their surface.

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1. Introduction

Microbial Fuel Cell (MFC) is a bioelectrochemical reactor in which electroactive bacteria convert the energy stored in various chemical substrates, into electricity [1]. A number of factors are influencing the overall MFC performance and complex mathematical models have been proposed to describe these systems [2].

The MFC technology is a sustainable way of producing energy, which has been used in a variety of applications, the most common of which include treating wastewater and nutrient recovery [3,4]. It was also shown that urine can be successfully treated in MFCs [5], and in some other examples, H_2 production has been reported [6]. Treating urine in MFCs was recently scaled up to pilot scale [7]. Our studies showed that power was sufficient to power LED lights and that urea was not contributing to the generation of power. So far, successful transformation of urea into electrical energy was only demonstrated in conventional fuel cells, including solid oxide fuel cells [8]. An interesting example of a hybrid technique using MFCs was described by Chen et al. who reported an electrodialysis system, consisting of an MFC used for alkali production and demonstrated its use for biogas upgrading [9]. Bioelectrochemical systems (BESs) can also be employed to techniques for sustainable synthesis of chemicals in a process known as microbial electrosynthesis [10], as well as for the recovery of valuable metals by reduction on the anode surface as demonstrated by Wang et al. [11]. The cathodic reactions may also lead to the removal of toxic hexavalent chromium as reported by Xafenias et al. [12].

Regardless of the design and application, the essential parts of every MFC are the anode and the cathode. These elements determine the overall performance of the MFC and both are susceptible to dynamic changes taking place during their operation. These changes include increasing the biofilm thickness as well as precipitation and adsorption of chemical compounds on the electrode surface [13,14]. The biofilm activity on the cathode was widely used as an approach for increasing the oxygen reduction rates, when pure phototrophic cultures or aerobic activated sludge were used as the biocatalyst [15,16]. Similarly, the utilisation of the laccase enzyme, an enzyme synthesised by several fungal species, which reduces O_2 to H_2O, leads to improved performance and extends the lifetime of the cathode [17,18]. On the other hand, unwanted biofilm growth on the surface of MFC components such as the proton exchange membrane or the cathode, may deteriorate the performance due to biofouling [19,20].

In recent studies workers have indicated that a power reduction during long-term MFC operation may be caused by biofouling or fouling of the membrane or the cathode. Behera et al. [21] observed a power drop just after 35 days of operation in ceramic MFCs. Pasternak et al. [22] reported decrease of performance after 32 days of operation of air–cathode MFCs built from four different types of ceramic materials. Similar observations were also reported by other authors, even when other types of material have been used to construct MFCs [23,24]. However, none of these studies have scientifically proven that it is the biofouling of the cathode that causes the power drop [21,22,24–27]. Chung et al. [23] reported that deterioration of the cathode was accompanied by the biofilm formation. However, following the cleaning procedure (10% HCl for one hour), the performance of the cathode decreased further. This phenomenon was also investigated by Yuan et al. [28] and it was found, that the presence of a biofilm on the inner (anode facing) layer of the cathode may decrease the performance of the MFC. Nevertheless, a solution for combating the undesirable biofilm growth has not yet been proposed.

Although the presence of a biofilm on the outer layer of the cathode can be inspected at a macroscopic scale, it is important to determine whether this biofilm may cause the power drop in MFCs. In the field of conventional fuel cells science, R&D has led to determining the role of cathode deterioration on power performance and appropriate strategies are currently being developed to address this problem [29,30].

In the field of MFCs, several long-term studies have reported deteriorating performance levels, primarily due to challenges with setup [31], environmental/experimental conditions and material degradation [24,32]. In particular, cathode biofouling has been reported as one of the main factors for decreased performance [23,24]. Investigating the impact of the undesirable biofilm growth on the cathodic performance, preventing it, or combating it once it occurs, is essential for the design and long-term operation of MFC-based scaled-up systems and their implementation into real world problems.

In this paper we investigated the characteristics of a serious performance deterioration observed for open-to-air cathode MFCs, operated for 131 days. The aim of this study, in addition to identifying the reasons for the significant power reduction, was to find a fast and simple method for *in-situ* regeneration of the conductive graphite-painted cathodes and recover their performance to the original levels.

2. Materials and methods

2.1. MFC construction and operation

Small scale, single chamber continuous flow MFCs (SCMFCs) were built using ceramic earthenware cylinders as both the chassis and the proton exchange membrane (Scientific & Chemical Supplies Ltd, UK). The ceramic tubes were cut to maintain the internal volume of empty MFCs equal to 11.4 mL. The anodes were made of carbon fibre veil and the cathodes were applied using conductive paint. For the carbon veil anode, the carbon loading was 20 g/m^2 (PRF Composite Materials, Dorset, UK). Carbon veil was cut into rectangles and pierced with a plain NiCr wire (Ø0.45 mm, Scientific Wire Company, UK). The total surface area of the anode was 252 cm^2. For the cathode, the outer ceramic membrane surface was covered with a conductive graphite paint prepared as described by Winfield et al. [33]. In brief the polyurethane rubber coating (PlastiDip, Petersfield, UK) was dissolved in petroleum spirit and mixed with graphite (Fisher Chemicals, UK) in a 1:3 (plastidip:graphite) ratio. After covering the ceramics with the first layer (carbon loading of 21.12 mgC/cm^2) a stainless steel mesh was used as the current collector (20 × 20, 0.18 mm). The mesh was then covered with an additional layer corresponding to a carbon loading of 14.08 mgC/cm^2. The projected surface area was 24.18 cm^2 and the total carbon loading for the whole electrode was 0.851 gC. The two layers of the cathode were separated by the current collector to decrease the internal resistance of the electrode. The stainless steel mesh allowed better integrity of the MFC as a whole and the thin, outer layer of the cathode was crucial to improve the contact between the current collector and the conductive carbon material.

The MFCs comprised a 3D printed Nanocure® RCP30-resin lid with inlet and outlet tubes, and a transparent acrylic lid (3 mm thick). SolidWorks 2013 software was used to design the RCP30 lids, which were manufactured with Perfactory4 3D printer (Envisiontec, Germany). Silicon gaskets were used to seal the space between the acrylic and RCP30 lids. Both lids were attached to the ceramic membranes by a single plain nylon screw (Ø3 mm, RS, UK). The MFC design is shown in Fig. 1.

A cascade of 9 MFCs was operated in continuous flow conditions. To avoid a liquid electrically conductive bridge between the units, the cells were fluidically isolated by gas gap fluid drip mechanisms – a physical air gap between the MFCs. The MFCs

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