Chapter 3
Integration of Cost-Efficient Carbon Electrodes into the Development of Microbial Fuel Cells

Jiseon You, Iwona Gajda, John Greenman, and Ioannis A. Ieropoulos

Abstract Microbial fuel cells are a renewable energy technology that can generate electricity from organic fuel such as wastewater, whilst simultaneously treating it. In order to implement this technology at larger scale, a major challenge is the choice of suitable electrode material that determines system performance and cost. This chapter discusses carbon-based electrodes and characteristics of carbonaceous materials that are both high performing and cost-effective in light of the technology implementation at larger scale for practical applications. The focus is on the most recent findings and incorporation of lightweight, robust and biocompatible carbon fibre electrodes in the form of carbon veil both as the anode and cathode counterparts and their suitability in larger scale designs. The chapter also presents modification strategies of this substratum with microparticles such as activated carbon as another cost-effective approach that may assist in bringing this technology closer to market.

Keywords Microbial fuel cell · Carbon veil · Activated carbon · Electrode modification · Cost-effective · Electrode material

3.1   Introduction

Research on sustainable energy is now focusing on organic waste material as a renewable energy source. Waste products both organic (wastewater) and inorganic (CO_2, pollutants) are abundant, easily accessible and yet still require energy and resources to treat. Recent advances in treatment and processing of waste are shifting towards more sustainable methods of resource recovery that require novel,

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innovative ways of preserving natural resources with simultaneous biorefining of already polluted soil, air and water.

Bioelectrochemical energy conversion within the microbial fuel cell (MFC) technology brings new alternatives and novel opportunities to convert organic substrates in waste-streams into green electricity using living microorganisms as active biocatalysts. Hence, the use of MFCs in water quality improvement, which is related to wastewater treatment, has attracted attention over recent years. More importantly, MFCs can operate as self-powered wastewater treatment systems.

Microorganisms respire and capture energy for the production of ATP (adenosine triphosphate) through the oxidation of organic and inorganic matter and reduction of a terminal electron acceptor. The application of MFCs for wastewater treatment has become increasingly important due to the multiple benefits of recovering electrical energy from wastewater, reducing the organic content and recovering nutrient resources. Organic molecules can be continuously broken down by the bio-electrochemically active microbes leading to continuous operation of the MFC. These benefits open up opportunities for technology scale-up and with the advancement in material science, power output levels increase and come closer to those desired for practical applications.

3.2   Microbial Fuel Cells

Microbial fuel cell (MFC) is an energy transducer that coverts chemical energy into electrical energy using microbial metabolism. The earliest discovery of the link between metabolic processes of living organisms and electrical energy was made by Luigi Galvani who observed the bioelectrical properties of animal tissue in 1791. More than a century later, in 1911 an English botanist Michael C. Potter published the first MFC report on the ability of microorganisms to transform organic substrates (chemical energy) into electricity by demonstrating production of electrical energy from living cultures of either *Escherichia coli* and *Saccharomyces cerevisiae* [1]. Although there were several remarkable milestones in the MFC development history, such as Cohen's stack of multiple MFCs and the USA National Aeronautics and Space Administration (NASA)'s attempt to recycle human waste in space missions [2, 3], it was only in the last two decades that significant advancements in terms of power performance have been made.

Initially researchers started paying attention to the two main aspects of the MFC technology; electricity generation and organics degradation. Since almost any type of organic matter and some inorganic compounds can be used as an MFC substrate, the vast majority of MFC studies have been carried out with a focus on sustainable and effective waste/wastewater treatment [4, 5]. However, the range of applications of MFCs go beyond that; in the early 2000s, two robots, Chew-Chew and EcoBot I, both inspired by the MFC technology were developed [6, 7]. MFC powered robots can be goes truly autonomous with no maintenance requirements unlike most of the miniaturised mobile robots that are powered from standard primary or secondary

chemical batteries. They can also be less affected by latitude, location, time of day and weather, in comparison with (e.g.) photovoltaic panels. Moreover MFC powered biodegradable robots can be realised, so that they may decompose once the task at hand has been completed in 'release and forget' missions [8]. Sensing is another promising application of the MFC technology. Since the current flow of an MFC is affected by the surrounding environmental cues, such as concentration of organic substrate or toxicants, pH or temperature, the number of studies into MFC biosensors has been growing significantly [9, 10]. The potential use of MFC as a power source in implantable medical devices is a more recent interesting research avenue that is being explored [11]. Soft MFCs made of flexible materials for low-power electronics such as wireless communication system have also been suggested [12, 13].

   MFCs usually consist of two compartments; an anode and a cathode separated by a membrane which facilitates ion flow. In the anode, electrochemically active microorganisms oxidise organic matter (fuel) and release CO_2, electrons and cations. Electrons produced in the anode flow towards the cathode via an external circuit as the result of electrophilic attraction from the cathode electrode, whilst protons migrate from the anode to the cathode through the separator between the two compartments. The electrons and protons subsequently combine with oxygen (final electron acceptor) and this reduction reaction completes the circuit. The electron flow through the external circuit results in current flow.

3.3   Cost-Effective Carbon Electrodes

For every MFC, regardless of its design or application, both the anode and cathode electrodes are essential components that determine performance and initial material cost [14]. For the successful choice of MFC electrodes, certain criteria need to be met such as high conductivity, large surface area, high porosity, good stability and durability as well as low cost. Considering that MFC anodes and some cathodes (i.e. bio-cathodes) act as hosts to microorganisms, biocompatibility is imperative. Moreover, minimising the environmental impact throughout the life cycle should be considered when selecting suitable electrode materials as well as easy accessibility and fabrication methods for the purpose of commercial applications.

   A wide variety of MFC electrode including carbon-based and metal/metal oxide-based materials have been tested [14, 15]. Carbon-based materials such as carbon cloth [16], carbon paper [17], carbon veil [18] and granular activated carbon (GAC) [19] are mostly used as both anode and cathode due to the aforementioned properties. Carbon fibre brushes are also widely used as anodes due to their good surface area, however their cost has been reported as double that of carbon cloth [20]. Anodic carbon materials can significantly promote interfacial microbial colonisation and accelerate the formation of electroactive biofilms, which eventually improves power by providing a conductive microenvironment for extracellular electron transfer [21]. On the other hand, despite the excellent electrical conduc-

tivity, metals tend to suffer from corrosion and biocompatibility issues (for biotic electrodes) with a few exceptions [22, 23]. The high cost of metals also cannot be overlooked for system scale-up. Recent advances in the design and fabrication of MFC electrodes based on bio-derived carbon such as biochar and activated carbon as well as pyrolised natural raw materials such as corncob [24], sugarcane [25], coconut [26] or even bread [27] is worth noting. Utilisation of these bio-derived carbon substrata is an attractive strategy due to their sustainability, low cost and satisfactory performance. However, the stability and longevity are rarely reported to assess how the materials would behave in real environments over prolonged periods.

The benefits of the larger surface area therefore higher surface activity than that of solid materials are faster biochemical and electrochemical reaction rates and higher catalytic activity leading to improved system performance. Therefore, nanocomposite electrodes containing carbon nanotubes, graphene or polyaniline (PANI) have gained a great deal of attention in recent years. These nanomaterials have shown promising results, although poor stability, cellular toxicity, complex fabrication process and high cost prevents their use in commercial products [28–31].

One of the biggest hurdles that hinders commercialisation of the MFC technology is the high cost of materials [32, 33]. Therefore, continuous efforts should be made in searching for cost-effective but well performing and robust electrode materials for long-term operation.

This chapter presents one of the most cost-effective carbonaceous materials such as carbon fibre veil and activated carbon powder used as electrode material in both the anode and the cathode within various microbial fuel cell settings.

3.3.1   Carbon Veil

Carbon veil (CV) is a thin ply of continuous strands of fibres that are looped randomly throughout the material. The fibres are held together with a binder such as poly vinyl alcohol (PVA). The nonwoven structure allows excellent resin uptake that increases chemical stability and reduces the risk of micro-cracks forming on the composite surface. Therefore, it is widely used in a variety of industries worldwide, including aerospace, defence, automotive, construction, consumer electronics and energy. Carbon veil can also be used for grounding a composite structure, minimising the build-up of static electricity that could prove dangerous when in contact with explosive liquids and gases.

As an MFC electrode material, carbon veil has many advantages. It possesses a non-woven structure of smooth, long, cylindrical fibres (Fig. 3.1) forming a network with large (macro-size) gaps between fibres that determine the porosity of this type of carbonaceous substratum. Carbon veil structure even when convoluted, allows for permeation and interpenetration of liquid media within the structure due to macro-porosity [18], and as such has been explored as the material for mL- and L-scale MFCs including large scale applications. It has good electrical conductivity,
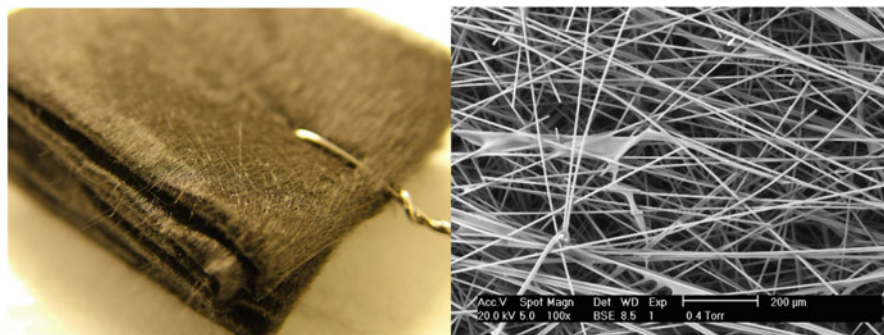
Fig. 3.1 A photo of folded 3D carbon veil material electrode (carbon loading: 20 g/m^2) (; left) and a scanning electron microscopy (SEM) image of the single layer at ×100 magnification (; right)

chemical resistance and higher porosity than woven carbon cloths. This lightweight, carbon-based nonwoven fabric, can be easily manipulated, folded, convoluted in any shape forming three dimensional (3D) structures for the bacterial attachment and formation of the electroactive biofilm. Also compared to other carbon-based fabric materials such as carbon cloth, the cost of carbon veil is much lower per unit area. Thus, it has been widely used as a 3D MFC electrode material.

As mentioned earlier, modification of the plain carbon veil is essential for enhanced performance, longevity and easy of scale-up. Thanks to the aforementioned properties (cost-effective and nonwoven structure that enables high chemical uptake) and high thermal stability, carbon veil is an ideal base material for electrode modification.

3.3.2 Activated Carbon

One of the most low-cost carbon-based materials are activated carbon and graphite which both show the lowest cost per mass [21]. Activated carbon (AC), also known as activated charcoal, is a carbonaceous, porous solid material with high specific surface area, relatively even pore size distribution and high-degree of surface reactivity. Due to its excellent adsorption properties, it has been widely used as an absorbent in many fields including hydrogen storage [34], medicine [35], water purification [36] and air filtration [37].

AC is produced from various carbonaceous materials such as wood, nutshells, fruit stones, peat, lignite, coal and petroleum pitch [38]. The process of AC synthesis usually consists of two steps, carbonisation and activation. Carbonisation is to reduce the volatile content of raw materials via pyrolysis of the carbon precursors in the temperature range of 300–900 °C. The following activation step is to improve the specific surface area or porosity through opening new pores and improving the existing pores. Properties of synthesised AC such as porosity, pore size, pore size

distribution and surface reactivity are affected by several factors including carbon precursors, activation pathways and activating agents [39].

Activated carbon electrodes have been widely used in bioelectrochemical systems, electrical energy storage as well as capacitive deionisation technologies due to the high storage capacity on the available large surface area, cost-effectiveness, availability and chemical stability. In MFCs, activated carbon is usually employed in granular, powder or woven form in either anodic or cathodic half-cell. However, it is primarily the powdered form that is used for in-house fabrication, modification and incorporation with other carbonaceous or metallic substrates. While the use of binding agents such as polyvinylidene difluoride (PVDF) or polytetrafluoroethylene (PTFE) is needed for AC powder attachment to the surface of the backbone, these binders can hinder ion access to the pore [40]. Currently, the choice of electrode core material and the method of surface modification is one of the main challenges undertaken in MFC research as it determines the system performance as well as technology cost for the purpose of future commercialisation.

3.3.3 *Activated Carbon Doped Carbon Veil Anode*

In order to enhance the bacterial adhesion as well as the electron transfer for efficient current collection, a strategy including the combination of carbonaceous scaffold decorated with a microporous agent can be successfully pursued [40], which can be followed utilising low-cost materials such as activated carbon as one of the most affordable and widely available forms of carbon [21]. Given the MFC anode is biotic, it is essential to give priority on pore size as well as the large surface area and high porosity. Surface topology has a direct influence on the formation and performance of electroactive biofilms [41]. This is important from the perspective of the mass transfer and diffusion limitations with regard to the substrate availability for the microbial biofilm grown on the surface of the electrode.

The clogging of pores in 3D porous carbon materials by the entrapment of bacterial cells can ultimately result in fast accumulation of inactive cells [42]. This may lead to a significant reduction of the power output levels. It has been reported that clogging most likely will happen if the diameter of the pore is smaller than 200 μm, and as a result mass transfer limitations would affect the bioanode performance [43].

Increasing the electrode macro area and electrode thickness by introducing multiple layers of carbon fibres provides a larger surface area for the electroactive biofilm to inhabit, but also a longer electron transfer path. Therefore the combination of macro- and micro-porosity has been introduced as a means of increasing the specific surface area in order to decrease the total (bulk) geometry [44].

The following sections describe some of the material parameters (Table 3.1) and show SEM images as well as data from real MFC experiments in which these materials were rigorously investigated; the methodology for experiments has been previously reported [44]. In summary, the MFCs were of cylindrical shape, made

Table 3.1 Material surface area and resistivity

Material	Surface area (m^a/g)[b]	Resistivity ($\Omega \bullet m$)[a]
Carbon veil	0.3183	9.2×10^{-4}
Activated carbon	672.1842	n.d.[c]
Modified anode	498.4422	7.7×10^{-4}

[a]measured using the four-wire resistance measurement
[b]measured by the Brunauer–Emmett–Teller (BET) method
[c]not determined
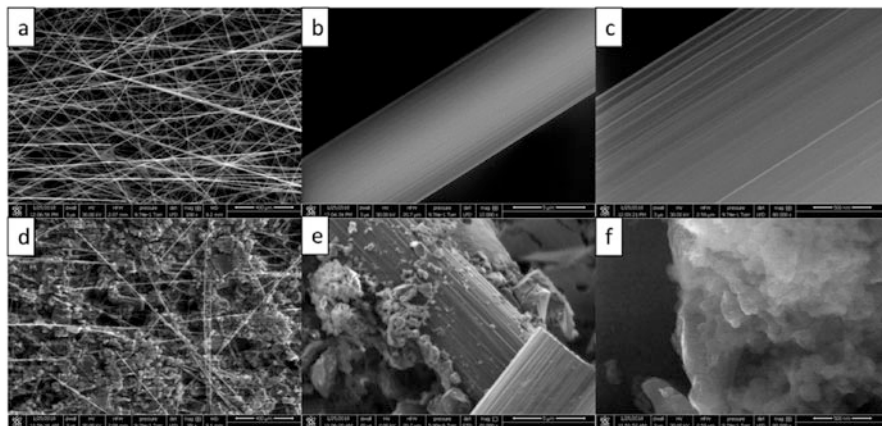
Fig. 3.2 Scanning electron microscopy observing plain carbon veil at (**a**) 100× (**b**) 10,000× (**c**) 80,000× and activated carbon doped carbon veil at (**d**) 99× (**e**) 20,000× (**f**) 80,000×. (Adapted from [44], Elsevier, under licence CC BY 4.0)

in house with terracotta clay and had an internal volume of 5 mL. The anode was a modified carbon veil with 5 mg/cm^2 of activated carbon powder and the cathodes were also modified carbon veil electrodes with 60 mg/cm^2 of activated carbon powder loading. In both cases, PTFE was used as the binding agent and it was 5% w/v for the anode and 20% w/v for the cathode. During the main parts of the experiment and for those data shown herewith, the external load connected to the MFCs was 100 Ω.

As shown in Table 3.1 and Fig. 3.2, AC particles attached on the smooth long fibres of plain carbon veil through the modification, created much higher surface area (1566 times) without losing the large void spaces. The additional micro- and nano-porous structures enhanced the surface roughness (Fig. 3.2e and f). The rough surface as a result of combination of macro, micro and nano pores should provide easier attachment for electroactive microorganisms compared to the smooth carbon fibres, hence higher cell population [45]. For bacterial attachment, the effect of electrode surface topology is predominant in macro-pores and grooves [46, 47], while the actual microbe-electrode interaction is determined by the microscopic roughness [47].
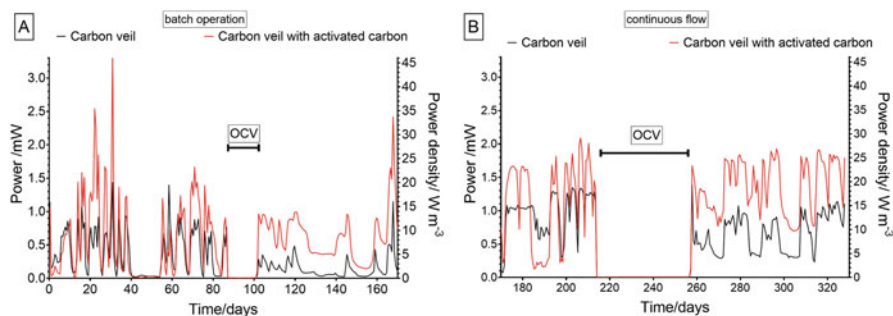
Fig. 3.3 Performance of activated carbon doped carbon veil anode in comparison to plain carbon veil both in batch operation (**a**) and under continuous flow feeding mode (**b**), OCV (open circuit voltage) depicts the period of time when the MFCs were operated under open circuit condition

Figure 3.3 illustrates 1-year performance of MFCs equipped with modified or unmodified, plain carbon veil electrodes. In Fig. 3.3a, the MFCs were operated in batch-fed mode and the maximum power performance of 3.2 mW was achieved by the modified anode MFCs, while the plain carbon veil showed up to 1.4 mW of power. Under continuous flow shown in Fig. 3.3b, MFCs with modified anodes stabilised at 1.8–2.0 mW, while MFCs with unmodified anodes showed output levels of 0.9–1.3 mW. The power fluctuations in this case were due to the cathode flooding by the continuously formed catholyte inside the cathode half-cells. The modification showed improved performance throughout the prolonged period of time and under various operating conditions including periodical feeding regime and long-term open circuit conditions (Fig. 3.3b). This shows both stability and resilience in the long term, which is sought after when implementing the technology in practical demonstrations. This is in line with the results obtained in the stack configuration [26].

These results show the enhanced surface area, surface topology, and electrical conductivity contribute to anode performance improvement. The results also demonstrate that the modified anode is suitable for long-term operation, showing no decrease in power generation performance for nearly one year of operation. In the literature, longevity is rarely reported which is connected to the performance stability within the lab, field trials and prototypes tested with real wastewater. The majority of materials and methodologies are unsuitable for practical implementations due to complex manufacturing processes, their scalability or high costs; moreover, the long-term stability of most tested materials is not given or is still unknown. Apart from the materials, scaling-up to multi-litre level brings about various challenges relating to design, stacking, fluidic and electrical complexities such as fuel starvation and voltage reversal. In this regard, the choice of materials for MFC assembly apart from electrodes as well as the choice of the semi-permeable membrane is of primary concern as it dictates technology performance and overall cost.

3.3.4   Cathode Electrodes: Incorporation of Carbon Veil and Activated Carbon

Carbon veil has also been a material of choice for the cathode in the biotic [48], synthetic [49], open to air cathode configurations [50], where the progress in material science and modification strategies are leading towards functionalisation of the carbon fibre matrix as a scaffold for the incorporation of microporous layers [51]. Cathode development is focusing on the improvement of air-breathing (open to air) cathode electrodes for the oxygen reduction reaction (ORR). The backbone of the carbon veil becomes a gas diffusion layer (GDL) and current collector where the active layer is directly attached to the scaffold and made of various carbon powder blends. This includes the carbon black and activated carbon. Activated carbon as the most affordable option was chosen for several lab-based studies as well as multiple prototypes tested in field trials of the technology. The powder of AC particles show micro-nano structure that promote electro-activity and enhances ORR while the carbon veil treated with hydrophobic agent such as PTFE allowing the air to permeate into the active layer (Fig. 3.2). Performance of the air-cathode that can be improved by developing an ORR catalyst with a high electrocatalytic activity, ensuring efficient transport, and enhancing oxygen diffusion.

The physico-chemical stability, robustness and cost of ceramics makes it particularly suitable for the technology advancement into larger scale and real-world applications. Optimum cathode modification results in better levels of performance, which can directly be used to power practical applications, as reported in [52]. As part of previous work, the modification was investigated under microscopy, which is now shown below in Fig. 3.4. Briefly, the MFCs were made of terracotta clay with 10 cm height, 3.6 cm inner diameter and a volume of 100 mL. Plain carbon veil (carbon loading: 20 g/m^2) was used as the anode and for the cathode, activated carbon/PTFE blend (20% w/w) was applied onto PTFE-treated carbon fibre veil. The MFCs were supplemented with a mixture of sludge and 0.1 M sodium acetate and tested in triplicates in batch-fed mode under 100 Ω external load.

In a different unpublished study, four types of carbon veil-based cathodes (10 cm^2) were tested as follows: i) plain carbon veil (20 g/m^2), ii) PTFE treated carbon veil, iii) thin layer of activated carbon paste with 20% PTFE content applied onto and iv) thick layer of AC paste applied onto the PTFE treated carbon veil backbone. The thickness of the thick AC was 3.0–3.5 mm, while the thin one was 1.5–1.6 mm. Carbon loading was 0.693 $\pm$ 0.03 g for the thin layer and 1.304 $\pm$ 0.05 g for the thick layer. The total of twelve MFCs were assembled as previously described [50].

Anode half-cells contained well matured anodes adapted from previous long-term MFC experiments operated for over 18 months. MFCs were assembled using 25 mL anode and 25 mL cathode chambers separated by a cation exchange membrane (CMI-7000, Membranes International Inc., New Jersey, USA) as previously described [48]. Anode electrodes were made of plain carbon fibre veil (20 g/m^2). MFC anodes were connected to the reservoir to recirculate the feedstock
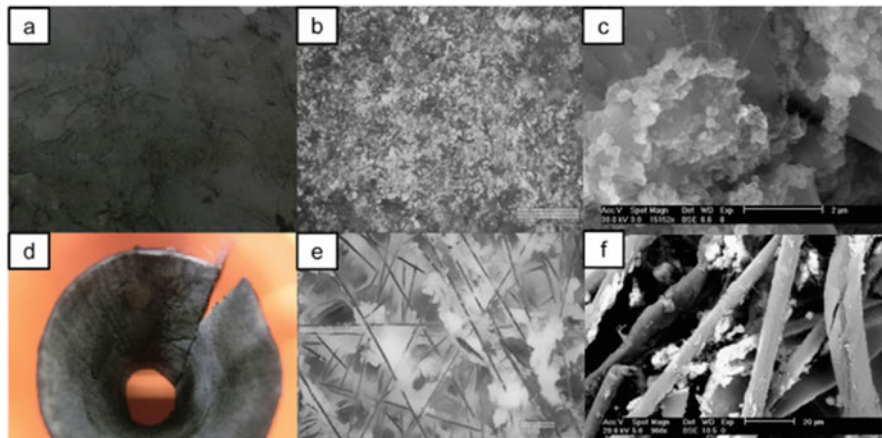
Fig. 3.4 Cathode electrode morphology. (**a**) Photograph illustrating AC layer; (**b**) digital microscope image using 400× magnification of AC; (**c**) SEM image of AC under high (15,152×) magnification; (**d**) photograph illustrating GDL on the air side; (**e**) digital microscope image at 400× magnification of GDL; (**f**) GDL under 960× magnification. (Adapted from [53], Elsevier, under licence CC BY 4.0)
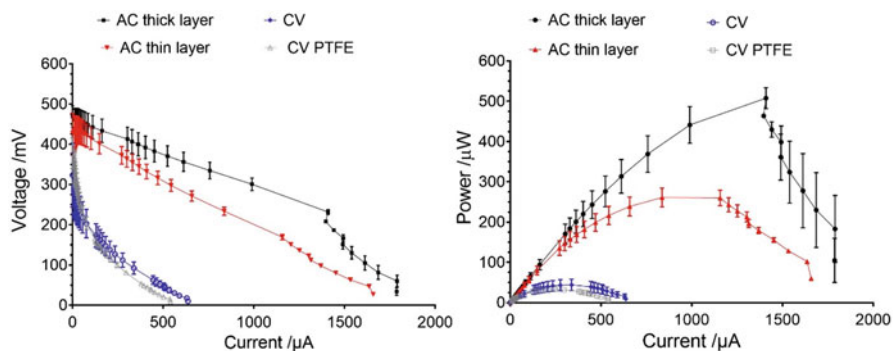

Fig. 3.5 Performance of thin and thick layer of activated carbon applied onto carbon veil cathode in comparison to plain carbon veil and PTFE treated carbon veil

(acetate/wastewater and urine) at a constant flow rate, while the external load connected was 300 Ω.

As can be seen in Fig. 3.5, the performance of the both AC-based cathodes was significantly higher than the unmodified plain CV both in PTFE treated and untreated versions. The maximum power achieved by the thick AC cathode was up to 500 μW, while the thin AC reached up to 260 μW, while the CV PTFE and CV produced only 32 μW and 43 μW respectively.

Figure 3.6 illustrates all MFC groups when tested under external load and two different feedstocks, namely wastewater supplemented with acetate and human
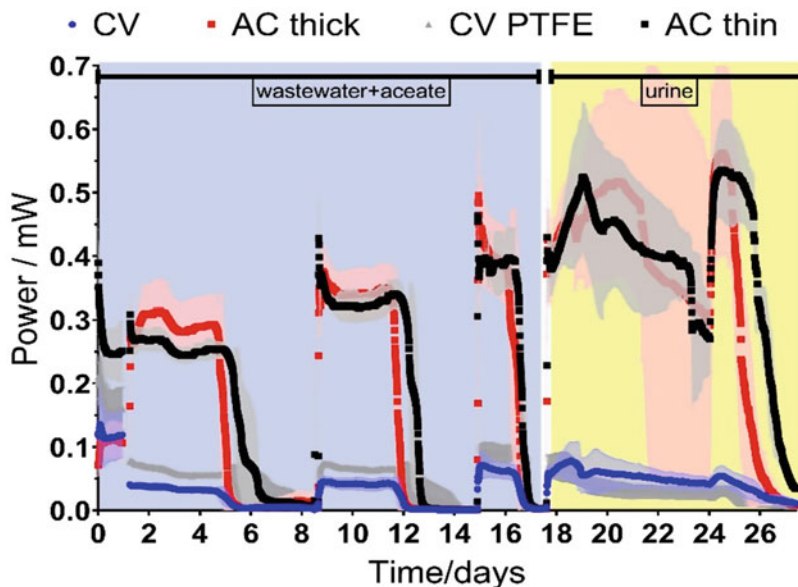
Fig. 3.6 Temporal behaviour of all four types of MFCs (n = 3, shaded areas illustrate error bars) when fed with sodium acetate/wastewater and when fed with neat human urine. Drop in performance shows feedstock depletion while the increase in power indicates fresh feedstock supply

urine. Although the MFCs were operated under constant recirculation mode between the anode and 1 L reservoir, the periodic feedstock depletion resulted in a decrease of power and subsequent feedstock replenishment resulted in restored and even improved output levels. Both thin and thick AC-modified cathode MFCs showed up to almost 400 μW when fed with acetate and over 500 μW when fed with urine, while the CV and CV PTFE reached up to 43 μW and 69 μW respectively when fed with acetate and 77 μW and 90 μW when urine was used as feedstock. The effect of AC-modification suggests an almost eightfold improvement. What is interesting to observe, both thin and thick AC modifications resulted in similar power performance, therefore higher carbon loading in this case did not improve the output.

In Fig. 3.7, both anodic and cathodic modifications are analysed in terms of performance and cost. It shows that for both anode and cathode, AC modification of the carbon veil is the most effective in terms of power to cost of material used (expressed in mW/£) reaching up to 7.4 mW/£. This is calculated using materials sourced in the United Kingdom, whereas both carbon veil and activated carbon are widely available and can be sourced worldwide even at more competitive prices when bought in bulk quantities. For the MFC experiments, both modifications of the core carbon veil are tailored for separate environments where the anode electrode is designed to accommodate biotic biofilm whereas the cathode is more compact,
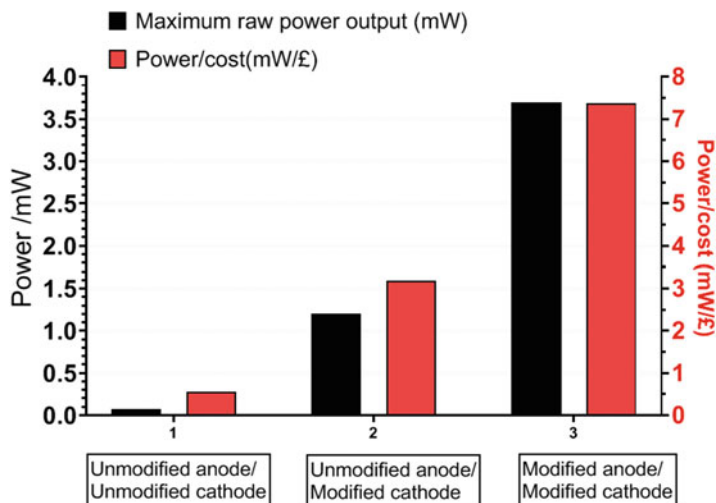
Fig. 3.7 Maximum power output and power/cost (in British pound sterling) comparison of the following anode/cathode configurations: 1) unmodified (plain) carbon veil anode and unmodified (plain) carbon cathode, 2) unmodified (plain) carbon veil anode and modified (with AC powder) carbon veil cathode, 3) modified (with AC powder) carbon veil anode and modified (with AC powder) carbon veil cathode. Please note that the 4th variation of modified anode/unmodified cathode was not tested. Costs of the PTFE binder material and fabrication are not included in power/cost comparison

hydrophobic (higher PTFE content) and abiotic for efficient ORR. Therefore, both methods of material preparation for the purpose of either cathode or anode assembly differ in binder content and in carbon loading because of the specific half-cell requirements. Power/cost ratio is particularly important when developing multi-modular stacks and prototypes for off-grid power production [54]. Therefore, further efforts in optimisation strategy with reducing overall cost and increasing MFC power output at the individual unit scale and from overall multi-unit stack can lead towards commercialisation of this technology.

3.4   Conclusions

The choice of electrode material is crucial for further development of the MFC technology as it determines its market readiness. Recent research had been directed towards anodic and cathodic modifications utilising high performing and affordable materials in order to enable scale-up and implementation. The combination of low-cost materials such as carbon fibre veil and activated carbon powder showed high power output, good longevity and improved power/cost ratio when implemented in both anode and in the cathode half-cells.

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