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Electrochemical Characterization of Stainless-Steel Mesh and carbon-felt Electrodes for Enhanced Power Generation in Terrestrial Microbial Fuel Cells

[a]Imologie M. Simeon*, [b]Amarachi C. Alaka, P. Daniel[a], [C]Olalekan D. Adeniyi.

[a]Department of Agricultural and Bioresources Engineering, PMB 65, School of Infrastructure, Process Engineering and Technology, Federal University of Technology, Minna, Nigeria. Email: Email: *Correspondence email: s.imologie@futminna.edu.ng

[b]Department of Agricultural and Bio-environmental Engineering, Imo State Polytechnic, Omuma, P.M.B. 1472, Owerri, Imo State, Nigeria.

[c]Department of Chemical Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology, Minna, Nigeria.

Abstract

Terrestrial Microbial Fuel Cells (MFCs) represent a promising avenue for sustainable energy production, leveraging microbial metabolism to convert organic matter in the soil into electricity. Crucial to MFC performance is selecting electrode materials, which directly interface with electroactive microbes for electron transfer. This study conducts a comparative analysis of surface-modified stainless-steel mesh (SMS) and carbon felt (CF) electrodes in terrestrial MFCs, evaluating their performance metrics and impedance spectroscopy. The SMS electrode, fabricated using the pasting and reinforcement process, demonstrated superior performance with a maximum power of 859 µW compared to the 234 µW power of the commercially available CF electrode. This better performance of the SMS electrode was attributed to its pseudocapacitive behavior, enhancing internal charge storage capacity and overall MFC efficiency. Electrochemical impedance spectroscopy revealed a substantially higher charge transfer resistance (R_{ct}) in the CF electrode, impeding electron transfer processes. Conversely, the SMS electrode exhibited lower R_{ct} and improved diffusion characteristics, facilitating efficient electron transfer and mass transport. Notably, the R_{ct} of the CF electrode was over 40 times higher, while its diffusion coefficient was approximately six times greater compared to the SMS electrode. These findings underscore the significance of tailored electrode materials in optimizing MFC performance and emphasize the utility of impedance spectroscopy in elucidating complex electrochemical processes within MFC systems, thus guiding future advancements in sustainable power production in terrestrial MFCs.

Keywords: Impedance, microbial fuel cell, modified stainless-steel mesh, electrode, carbon-felt, performance

1. Introduction

The microbial fuel cell (MFC) is, generally, a bioelectrochemical system (BES) that generates electrical power through the metabolism of electroactive microorganisms. MFCs use microbes as catalysts to oxidize organic and inorganic materials, generating electricity (Logan *et al*., 2006; Simeon and Raji, 2016). While the nomenclature of MFCs is usually based on the architecture, configuration, and substrate type, terrestrial MFCs (TMFCs) are usually inoculated with biologically active soil (Simeon *et al*., 2016). In its simplest configuration, the TMFC comprises two electrodes separated by a layer of soil (the electrolyte) and connected through an external electrical load (Garbini *et al*., 2023). The electrode material is a crucial factor for power generation in MFCs, as it directly links electron transfer and electroactive microbes (Schröder, 2007). The electrodes (anode and cathode) used in MFCs must possess desirable properties. They should be electrically conductive, non-corrosive, non-fouling, porous, inexpensive, easy to manufacture, and suitable for larger systems. In particular, the anode should have a large surface area for microbial settlement (Rahimnejad *et al*., 2015). Conductivity is a crucial property of these materials as electrons must flow through them from transmission by the microorganism to the collection site. It is important to note that any material that serves as an anode can also function as a cathode material by adding a catalyst. An effective cathode for microbial fuel cell (MFC) research should be made of a conductive material containing a catalyst and be in contact with the anode substrate and air. Carbon and metal-based electrodes are commonly used as they meet these criteria (Logan 2007; Santoro *et al*. 2017).

Extensive research has been conducted on using metals and metal coatings as electrode materials to enhance MFC performance. Stainless steel (plate, mesh, foam, or scrubber) has been preferred due to its conductive, robust, and cost-effective properties (Ketep *et al*. 2014). Ouitrakul *et al*. (2007) investigated the impact of various electrode materials, including silver (Ag), aluminum (Al), nickel (Ni), stainless steel (SS), and carbon-fiber mesh, on MFC performance. According to Ouitrakul *et al*. (2007), the weak adhesion of the inoculated microorganisms to stainless steel limits its application. Although many other metals possess these important properties, their corrosive properties and lack of a suitable surface for bacterial adhesion limit their suitability. Recent studies have shown that metals such as copper, nickel, silver, and titanium can be successfully used as anode electrode materials (Santoro *et al*., 2017). However, copper is not commonly used because even trace amounts of copper ions are toxic to bacteria (Logan *et al*., 2006).

Stainless steel mesh (SSM) is a commonly used metal-based electrode due to its desirable characteristics, including excellent mechanical strength and corrosion resistance (Papillon *et al*. 2021). It is highly conductive and can be easily incorporated into SMFCs as an electrode (Umar *et al*. 2021). However, due to the small electrochemically active surface area of SSM and its tendency to corrode in the presence of oxygen or even in an oxygen-free environment (Li and Yu, 2015), surface modification is usually necessary to improve its properties.

The most common surface modification strategy is to use polymeric binders to bond nano carbon powder or carbon granules such as activated carbon (AC) and carbon black (CB) to the surface of the SSM electrode as a catalyst (Simeon and Freitag, 2020). Polymeric adhesives commonly used to bond catalysts to SSM mechanically include polytetrafluoroethylene (PTFE), Nafion, polyvinyl alcohol (PVA), polyvinylidene fluoride (PVDF), and commercial epoxy adhesives (Dong et al. 2012; Wei et al. 2012; Chen et al. 2015; Zafar et al. 2021). The use of modified SSM (SM) as an electrode and polymer binder in SMFCs has been extensively reviewed and reported (Simeon et al., 2022a). However, not much is known about the impedance kinetic of the SMS electrode vis-à-vis the commonly used CF electrode in the TMFC

Understanding the principles and behavior of electrochemical reactions at the interface between biotic entities in the biofilm, the substrate, and the electrode surface is crucial for the further development and improvement of the SMS mesh electrode and its practical implementation in TMFCs (Yasri et al., 2019). Linear sweep voltammetry (LSV) is a polarization technique that provides valuable information about the losses and performance indices of the MFC. It does not, however, provide information about its impedance kinetics. Electrochemical impedance spectroscopy (EIS), on the other hand, is a non-destructive technique for studying electrochemical reactions at metal-biofilm interfaces (Chandrasatheesh and Jayapriya, 2019) and provides a more accurate measurement of the internal resistance and capacitive component of a fuel cell in terms of impedance (He and Mansfeld, 2009; Simeon, 2023).

Major losses in microbial fuel cells (MFCs) are associated with electrode kinetics, including mass transfer, charge transfer, and activation. Despite their critical role in power generation, electrode kinetics are often ignored in studies related to TMFCs. It is important to consider these factors to improve power generation efficiency. The study aims to investigate the impedance spectroscopy and performance metrics of the modified stainless-steel electrode in a terrestrial or soil microbial fuel cell (MFC) using the SMS mesh. The major performance indices considered include theoretical current, maximum performance characteristics, fill factor, and internal resistance. This study compares the newly developed SMS mesh electrode with a commercially available electrode to identify potential strategies for improving the performance of the SMS electrode in a TMFC for enhanced biopower production.

2. Materials and methods

2.1 Fabrication of the SMS electrode

The metal base of the SMS electrode was fabricated using a stainless-steel mesh (SSM: type 1.4301, Edalshop, Germany) with a mesh size of 0.63 mm and a wire thickness of 0.25 mm. The mesh was cut to a diameter of 6.5

cm, pre-cleaned with abrasive paper, and then cleaned in an ultrasonic bath (DA-968, China) for 20 min before drying at room temperature. A bonding paste was prepared by mixing approximately 0.26 g of CB (Vulca-72) with a two-component epoxy adhesive (UHU plus ENDfest, Germany). The two epoxy components were mixed in a 1:1 ratio so that the paste contained approximately 5 g of epoxy. The paste was applied evenly to both sides of the SSM by manual stirring in a Petri dish. Additional CB was applied to both sides of the electrode and clamped between two smooth surfaces using a mechanical clamp. The prepared SSM electrode was clamped overnight at room temperature (20°C $\pm$ 0.5°C) to allow proper formation before use. The dry SM electrode had a thickness of approximately 1.7 mm. Twisted wire strips, left as extensions of the SSM, were used as current collectors to connect the electrode to the external load and the MFCs to a data acquisition system. All exposed parts of the current collectors were insulated with heat shrink tubing (ID 1.6-0.8 mm) and heated to 120°C using a heat gun (Simeon and Freitag 2021; Simeon, 2023). Figure 2.1 A is a pictorial representation of the SMS electrode that was completely fabricated. A commercial CF electrode (AVcarb soft carbon felt C100) was prepared as a control. The CF electrodes (anode and cathode) were cut into a round shape with a diameter of 6.5 cm (corresponding to a circular surface area of 33.18 cm^2) and pretreated by heating in an oven at 400 °C for 4 h to remove impurities that could interfere with charge transfer (Wang et al. 2009). Titanium wire (0.5 mm diameter, 99.9% purity) was inserted into each of the CF electrodes and secured on both sides with an adhesive paste of epoxy and highly conductive CB (Cabot Vulcan XC 72, Quitech, Germany) to ensure proper electrical contact between the CF and the current collector (Simeon et al., 2021a, b).

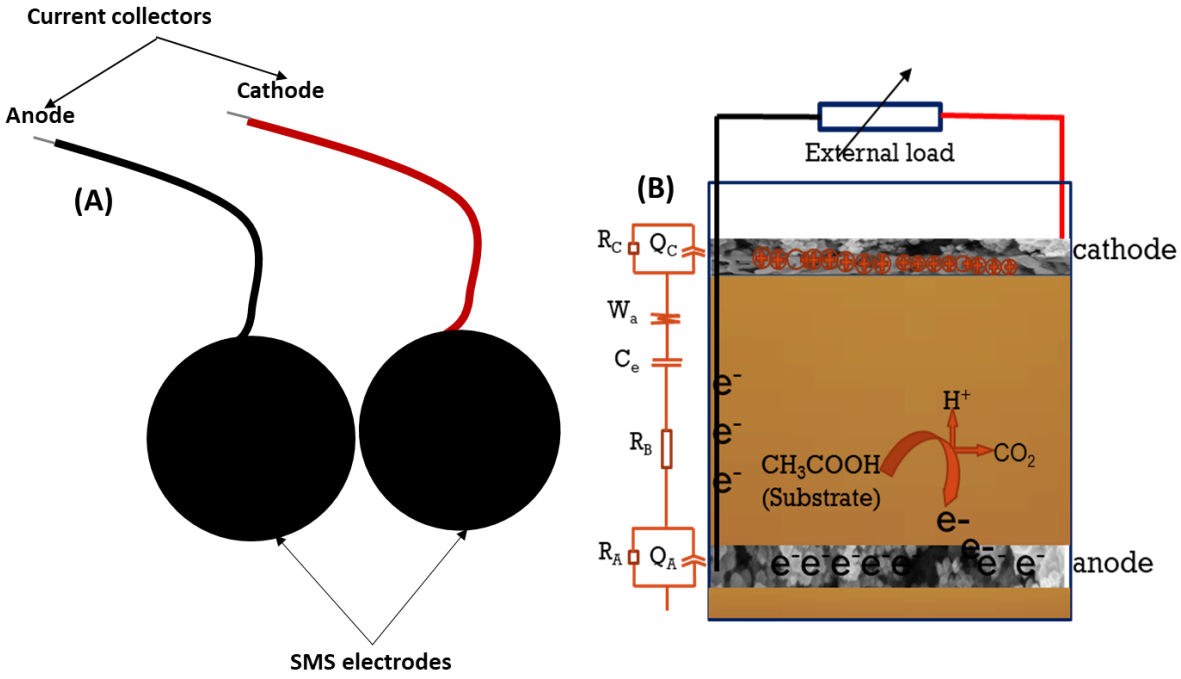

Figure 2.1. (A) A pictorial display of the fabricated SMS electrode; (B) A schematic view of the TMFC set-up, mode of operation, and equivalent electrical circuit for impedance spectroscopy. RA and RB are the anode and cathode R_{ct}, Q_A and Q_B are constant phase elements that represent the anode and cathode double-layer capacitance, C_e is the system's equivalent capacitance, and R_B is the Ohmic resistance.

2.2 TMFC Assembly and operation

The assembly of the TMFC was carried out following the procedure previously described (Simeon, 2023). Figure 2.1 (B) presents a schematic representation of the setup. Before anode installation, a layer of biologically active soil sludge, approximately 1 cm thick, was applied to the bottom of the MFC. Additional sludge was then added to achieve an anode-to-cathode spacing of 4. The mass of sludge between the anode and cathode was

approximately 306.72 g. Finally, the cathode was installed. The vessels were designed to leave a 4 cm space between the cathode and the lid after installation to ensure sufficient ventilation of the cathode. The TMFCs were operated at an ambient temperature of 20 ± 0.5°C.

2.3 Performance evaluation and Impedance spectroscopy

For continuous data collection, the MFCs were connected to the ADC-24 data acquisition system (Pico Technology) via a terminal board (176-74-252) to record data hourly. Continuous data recording was required to monitor the voltage trends of the SMFCs up to their maximum value before evaluating their electrochemical performance using linear sweep voltammetry (LSV) and electrochemical impedance spectroscopy (EIS), following the methodology of Simeon and Freitag (2021). A Potentiostat (Biologic VMP3, France) was used for all electrochemical measurements during the experiment. When fully polarized, other performance indices such as theoretical power, short-circuit current, cell voltage, and fill factor were measured in addition to maximum power and cumulative charge. The measured power was normalized to the geometric surface area of the cathode to obtain the power density (P_d) using Equation 2.1.

$$P_d(mWm^{-2}) = \frac{P(mW)}{A(m^2)} \qquad 1$$

Using the cell voltage, current, and resistance at MPP, the total internal resistance of the MFCs was calculated using Equation 3.3.

$$r = \left(\frac{E_{ocv}}{E_{cell}} - 1\right) * R_{ex} \qquad 2$$

The impedance characteristics and reaction kinetics of the MFCs were determined using electrochemical impedance spectroscopy (EIS). A small AC signal of 10 mV (or 7.07 mV_{eff}) was applied to the terminals of the MFCs in a frequency range of 100 kHz and 10 mV at 10 points per decade. The physical parameters of interest were determined by fitting the experimental data to the measured data using the electrical equivalent circuit in Figure 2.1. The impedance spectra were represented in Nyquist plots.

3. Results and discussions

3.1. Performance indices of the electrodes

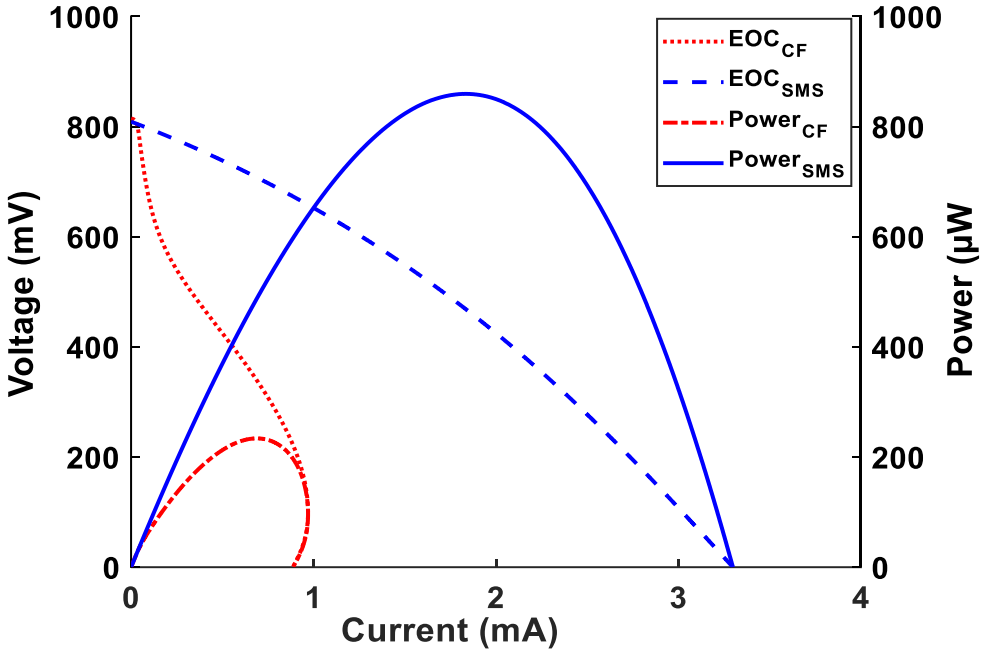

Figure 3.1. Polarization and performance curves of an SMS and a CF electrode in a TMFC

Figure 3.1 presents the polarization and power curves for CF and SMS electrodes plotted against cell current. Analysis of the polarization curves reveals the presence of activation, ohmic, and concentration losses in the CF electrode. Conversely, the SMS electrode primarily exhibits ohmic losses. The higher concentration losses in the CF electrode translate to its inferior performance compared to the SMS electrode. The power curve further emphasizes this disparity, indicating a roughly threefold increase in power output for the SMS electrode compared to the CF electrode. Specific performance metrics are presented in Table 3.1. Notably, while the CF electrode exhibits a slightly higher open circuit voltage (Eoc), the SMS electrode delivers a superior current density and cell voltage, enhancing overall performance. This can be attributed to the higher overpotential experienced by the CF electrode, resulting in a significant "lost voltage," as described by Equation 3.

$$E_{oc} = V + Ir \qquad 3$$

where Ir is the “lost voltage” or overpotentials

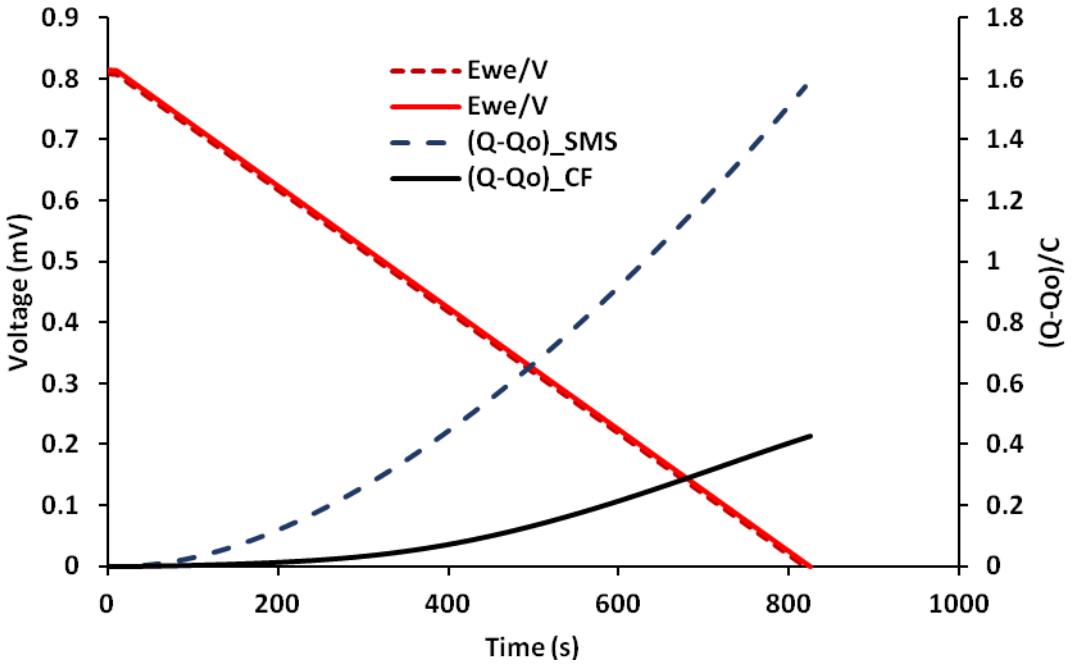

Figure 3,2. Cumulative Charges of the electrodes under polarization

Table 3.1: Performance indices of the SMS and CF controlled electrodes.

parameters	**MFC_c**	**MFC_{nc}**
Isc/mA	3.30	0.89
Eoc/mV	0.81	0.82
PT/mW	2.67	0.722
P_{max}/mW	0.86	0.23
I_{max}/mA	1.83	0.69
V_{max}/mV	0.47	340.60
R_{int}(mV)	256.83	493.62
FF/%	32.20	32.40
Cum. Charge/C	1.57	0.43

Figure 3.2 and Table 3.1 demonstrate that under identical polarization conditions, the charge storability of the SMS electrode surpasses that of the CF electrode. This suggests an enhanced internal charge storage capacity within the MFC equipped with the SMS electrode. The pseudocapacitive behavior of the SMS electrode facilitates charge accumulation, contributing to improved performance. Furthermore, compared to the CF electrode, the SMS electrode offers advantages beyond mitigating the overpotentials and associated losses expected in TMFCs. The CF electrode used in this study has a lower intrinsic capacitive property necessary to store microbially generated charges internally.

It is evident from the performance indices that both electrodes exhibit similar open circuit voltages (OCV), with values overlapping during polarization. Specifically, the maximum voltages recorded for the SMS and CF electrodes were 808 mV and 815 mV, respectively. This close alignment in OCV values suggests the presence of comparable electroactive microbial communities with similar redox potentials in both MFC configurations (Simeon et al., 2022b). Therefore, the disparity in maximum power output cannot be attributed solely to microbial community dynamics but instead points towards dissimilarities in electrode kinetics. Thus, to further elucidate these differences, EIS was employed to evaluate and compare both electrodes' charge transfer kinetics, ohmic resistance, and mass transfer characteristics.

3.2 Electrochemical Impedance Spectroscopy (EIS) Analysis of SMS and CF electrodes

Figure 3.1 presents the impedance spectra of the CF- and SMS-based TMFCs. Table 3.2 presents the electrode kinetics and resistance.

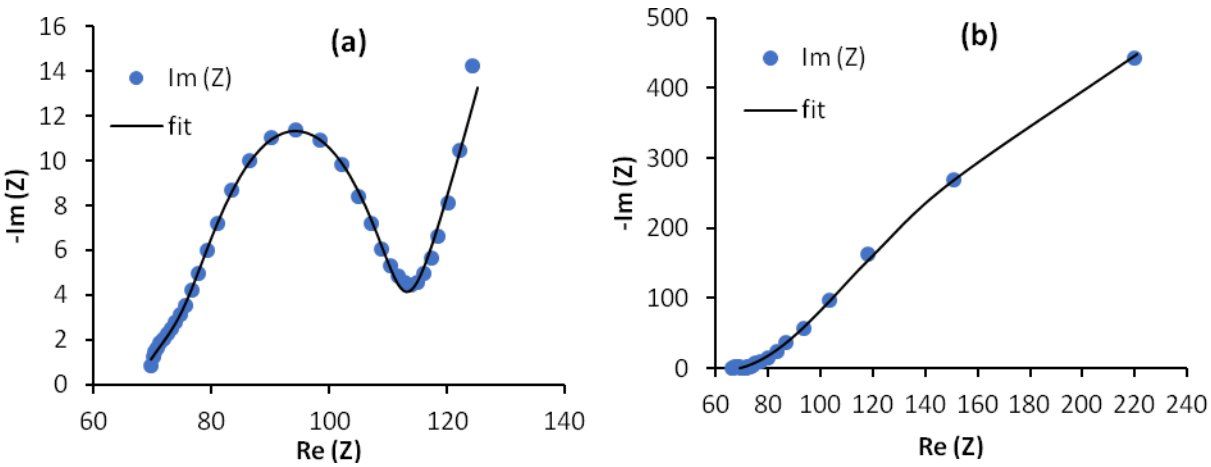

Figure 3.3. Nyquist plots of (a) SMS-based TMFC and (b) CF-based TMFC

Table 3.2. Impedance kinetics of the electrodes

EM	R_{Ohmic} (Ω)	C2 (F)	R_{ct} (Ω)	Q3 ($F.s^{a-1}$)	a	s3 ($Ω.s^{-1/2}$)
SMS	67.23	0.2699E-3	45.79	4.18E-03	3.23E-01	3.45
CF	66.13	0.04146	1953	2.17E-06	0.02686	19.86

The impedance spectroscopy revealed comparable ohmic resistances (R_{Ohmic}) for both SMS and CF electrodes (Figure 3.3 and Table 3.2). This similarity suggests minimal influence of contact resistance on the observed performance differences. However, significant discrepancies emerged in the charge transfer resistance (R_{ct}) and diffusion coefficient. Notably, the R_{ct} of the CF electrode was over 40 times higher, while its diffusion coefficient was approximately 6 times greater compared to the SM electrode. Although the capacitance C2 (Table 3.2) of the SMS electrode is higher than that of the CF electrode, the values of the C2 only represent the equivalent series capacitance of the whole TMFC systems used here. The charge storability of the electrode can be viewed from the double-layer capacitance represented by the CPE (Q3). The actual equivalent double-layer capacitance of the anode and cathode can be estimated as highlighted by Simeon *et al*. (2021b and 2022b).

These EIS findings align perfectly with the LSV results, which indicated the superior performance of the SM electrode due to its lower internal resistance (R_{int}). Importantly, EIS further deconstructs the components of R_{int} that are responsible for the observed performance disparity. The markedly higher R_{ct} of the CF electrode hinders electron transfer processes between the substrate and the microbes, and between the microbes and the electrode itself. Additionally, as Simeon (2023) highlighted, diffusion limitations of reduced or oxidized species within the CF electrode further impede its performance.

4. Conclusions

This study conducted a comparative analysis of performance metrics and impedance spectroscopy of a surface-modified stainless-steel mesh (SMS) and carbon-felt (CF) electrode within a terrestrial microbial fuel cell (MFC) aimed at micro-power production. The SMS electrode displayed superior performance compared to the CF electrode. This can be attributed to two key factors elucidated by this investigation. Firstly, the SMS electrode exhibited a greater charge storage capacity due to its pseudocapacitive behavior, potentially enhancing MFC efficiency and energy output. Secondly, EIS analysis revealed a significantly higher charge transfer resistance (R_{ct}) in the CF electrode, hindering electron transfer between microbes and the electrode. Conversely, the lower R_{ct} and improved diffusion characteristics of the SMS electrode facilitated efficient electron transfer and mass transport, leading to its superior performance. These findings underscore the importance of tailoring electrode materials to promote favorable electrochemical processes within MFCs. Furthermore, the application of EIS offers a valuable tool for dissecting the complex interplay between electrode properties and MFC performance, guiding the development of future more efficient terrestrial MFC systems.

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