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Performance evaluation and impedance spectroscopy of carbon-felt and reinforced stainless-steel mesh electrodes in terrestrial microbial fuel cells for biopower generation

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ABSTRACT

Terrestrial Microbial Fuel Cells (TMFCs) offer promising potential for renewable energy by harnessing microbial metabolism to generate electricity from soil-based organic matter. Electrode materials are key to TMFC performance, facilitating electron transfer between microbes and the circuit. However, the effect of electrode impedance on TMFC efficiency is not well understood. This study fills that gap by comparing surface-modified stainless-steel mesh (SMS) and carbon felt (CF) electrodes, focusing on performance metrics and impedance spectroscopy to optimize electrode design for improved power generation from TMFCs. 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 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 in the CF electrode, resulting in a 190.8 % difference between the two electrodes. Conversely, the SMS electrode exhibited lower resistance and improved diffusion characteristics, facilitating efficient electron transfer and mass transport. These findings underscore the significance of tailored electrode materials in optimizing MFC performance and emphasize the utility of electrochemical impedance spectroscopy in elucidating complex electrochemical processes within MFC systems, thus guiding future advancements in sustainable power production in terrestrial MFCs.

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 [1,2]. While the nomenclature of MFCs is usually based on the architecture, configuration, and substrate type, terrestrial MFCs (TMFCs) are generally inoculated with biologically active soil [3]. In its simplest configuration, the TMFC comprises two electrodes separated by a layer of soil (the electrolyte) and connected through an external electrical load [4]. The electrode material is a crucial factor for power generation in MFCs, as it directly links electron transfer and electroactive microbes [5]. The electrode (anode and cathode) materials used in MFCs must possess specific 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 [6]. 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 with the addition of a catalyst. An effective cathode for MFCs 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 [1,7].

Extensive research has been conducted on the potential use of metals and metal coatings as electrode materials to enhance MFC performance. Stainless steel (plate, mesh, foam, or scrubber) has been preferred due to

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its conductive, robust, and cost-effective properties [8]. Ouitrakul et al. [9] 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 Ref. [9], 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 [7]. However, copper is not commonly used because even trace amounts of its ions are toxic to bacteria [1].

Stainless steel mesh (SSM) is a commonly used metal-based electrode due to its desirable characteristics, including excellent mechanical strength and corrosion resistance [10]. It is highly conductive and can be easily incorporated into TMFCs as an electrode [11]. 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 [12], surface modification [13] is usually necessary to improve its properties [14,15].

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 [16]. Polymeric adhesives commonly used to mechanically bond the catalyst to the SSM include polytetrafluoroethylene (PTFE), Nafion, polyvinyl alcohol (PVA), polyvinylidene fluoride (PVDF), and commercial epoxy adhesives [17–20]. The use of modified SSM as an electrode and different polymer binders in SMFCs has been extensively reviewed and reported [21]. However, not much is known about the impedance and capacitive characteristics of the surface-modified SSM (SMS) electrode vis-à-vis the commonly used CF electrode in the TMFC. While many studies on SMS electrodes focus on their morphology and the enrichment of electroactive microbes [14,22], the impedance spectroscopy of such electrodes used in TMFCs is often not reported [23–25], likely due to the challenges posed by the unique configurations of TMFCs [26], which make the insertion of reference electrodes for the study of the half-cell reactions of the cathode and anode difficult.

However, 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 electrode [27] and its practical implementation in TMFCs [28]. Linear sweep voltammetry (LSV) is a polarization technique that provides useful information about the losses and performance indices of the MFC. It does not, however, provide information about its impedance characteristics. Electrochemical impedance spectroscopy (EIS), on the other hand, is a non-destructive technique for studying electrochemical reactions at metal-biofilm interfaces [29] and provides a more accurate measurement of the internal resistance and capacitive component of a fuel cell [30,31].

Major losses in microbial fuel cells (MFCs) are associated with electrode kinetics, including mass transfer, charge transfer [32], and activation [33]. Despite their critical role in power generation, electrode kinetics are often ignored in studies related to TMFCs. It is important to consider these factors in TMFC configurations to improve their power generation efficiency. The study aims to investigate the impedance spectroscopy and performance metrics of the SMS and CF electrodes in a terrestrial or soil MFC. 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 CF electrode to identify potential strategies for improving the performance of the SMS electrode in TMFCs for enhanced biopower production.

2. Materials and methods

2.1. Fabrication of the SMS electrode

The SMS electrode was fabricated using the pasting and reinforcement method [21], which involves the application of a conductive carbon layer onto a stainless-steel mesh (type 1.4301, Edalshop, Germany) with a mesh size of 0.63 mm and a wire thickness of 0.25 mm. This fabrication method was chosen due to its simplicity, cost-effectiveness, and compatibility with resource-constrained environments, particularly in rural settings where access to specialized equipment may be limited.

The stainless-steel mesh was cut into a diameter of 6.5 cm, precleaned with abrasive paper, and then subjected to ultrasonic cleaning in a bath (DA-968, China) for 20 min before air drying at room temperature. A bonding paste was prepared by mixing approximately 0.26 g of carbon black (CB, Vulca-72) with a two-component epoxy adhesive (UHU plus ENDfest, Germany). The epoxy components were mixed in a 1:1 ratio, resulting in a paste containing approximately 5 g of epoxy. The choice of epoxy adhesive over alternatives like PVA, PTFE, and PVDF was driven by its superior adhesive strength and durability [34]. Epoxy's exceptional bonding properties ensure uniform and robust deposition of the conductive carbon black layer onto the stainless-steel mesh substrate, minimizing the risk of delamination during extended operation [21], The paste was applied evenly to both sides of the SSM by manual stirring on 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 SMS electrode was clamped overnight at room temperature (20 °C ± 0.5 °C) to allow proper formation before use. The dry SMS 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 to connect 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 [31,35]. Fig. 1 (A) is a pictorial representation of the SMS electrodes that were completely fabricated. As a control, a commercial CF electrode (AVcarb soft carbon felt C100) was also prepared. 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 [36]. 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 [37].

2.2. TMFC assembly and operation

The Assembly of the TMFC was carried out following the previously described procedure [31]. Fig. 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 until the maximum open-circuit voltage was reached before polarization and EIS were performed.

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-

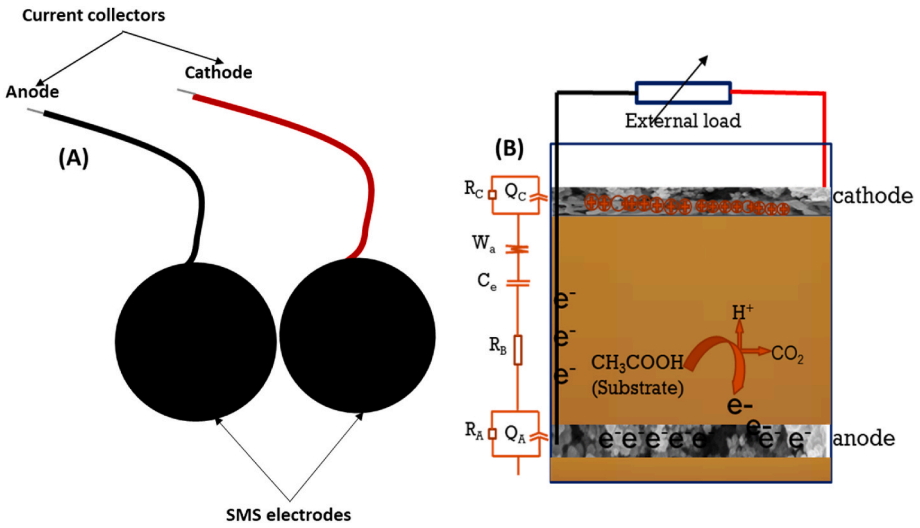

Fig. 1. (A) A pictorial display of the fabricated SMS electrode; (B) A schematic view of the TMFC set-up, mode of operation, and equivalent electrical circuit for impedance spectroscopy. R_A and R_B are anode and cathode charge transfer resistance (R_{ct}), Q_A and Q_B are constant phase elements used to represent the anode and cathode double-layer capacitance, C_e is the system's equivalent capacitance, R_B is the Ohmic resistance, and W_a is the Warburg element to determine the oxygen diffusion characteristic of the MFC.

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 LSV and EIS, following the methodology previously described [35,38]. 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 (1).

$$P_d\left(mWm^{-2}\right) = \frac{P(mW)}{A(m^2)}\,. \tag{1}$$

Using the cell voltage, current, and resistance at MPP, the total internal resistance of the MFCs was calculated using Equation (2) [39].

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

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 EC-Lab (version 11.32 by Biological Instruments, France) and the electrical equivalent circuit shown in Fig. 1. The impedance spectra were represented in Nyquist plots (Fig. 4).

3. Results and discussions

3.1. Performance indices of the electrodes

Fig. 2 presents the polarization and power curves for both CF and SMS electrodes plotted against cell resistance. 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 of losses in the CF electrode translates 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 1. Notably, while the CF electrode exhibits a slightly higher open circuit voltage (E_{oc}), the SMS electrode delivers a superior current density and cell voltage, leading to enhanced 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).

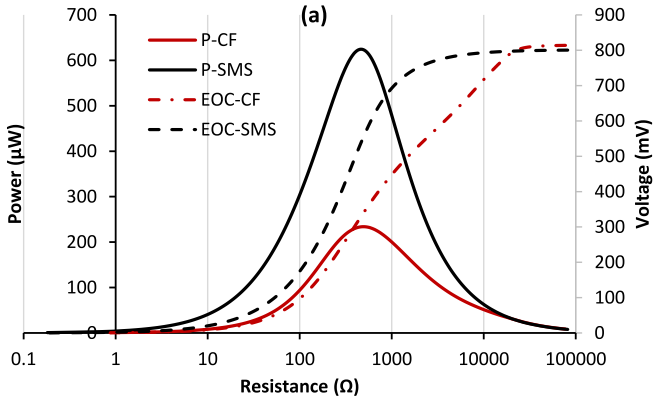

Fig. 2. Polarization and performance curves of an SMS and a CF electrode in a TMFC. The Resistance axis is in logarithmic scale for a better presentation.

$$E_{oc} = V + Ir. \tag{3}$$

where *Ir* is the "lost voltage" or overpotentials

Table 1
Performance indices of the SMS- and CF- based TMFCs.

parameters	SMS-MFC	CF-MFC
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

Fig. 3 and Table 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 common in terrestrial MFCs. 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 E_{oc}, 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 E_{oc} values suggests the presence of comparable electroactive microbial communities with similar redox potentials in both MFC configurations [37]. Therefore, the disparity in maximum power output could not be attributed solely to microbial community dynamics [37] but rather points towards dissimilarities in electrode kinetics. Therefore, to further elucidate these differences, EIS was employed to evaluate and compare the charge transfer kinetics, ohmic resistance, and mass transfer characteristics of both electrodes.

3.2. *Electrochemical impedance spectroscopy (EIS) analysis of SMS and CF electrodes*

Fig. 4 illustrates the impedance spectra of the TMFCs based on SMS and CF electrodes, while Table 2 summarizes the electrode kinetics and resistance parameters. The equivalent double-layer capacitance (C2) of the anode and cathode was estimated from Q_A and Q_B. In contrast, the charge transfer resistance (R_{ct}) corresponds to the series combination of R_A and R_B, since the whole-cell configuration was used for the EIS.

The impedance spectroscopy revealed comparable ohmic resistances (R_{Ohmic}) for both SMS and CF electrodes (Fig. 4 and Table 2). This similarity suggests a minimal influence of contact resistance on the observed performance differences [37]. However, significant discrepancies emerged in the R_{ct} and diffusion coefficient (s3). 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 2) of the SMS electrode is lower 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 Ref. [37].

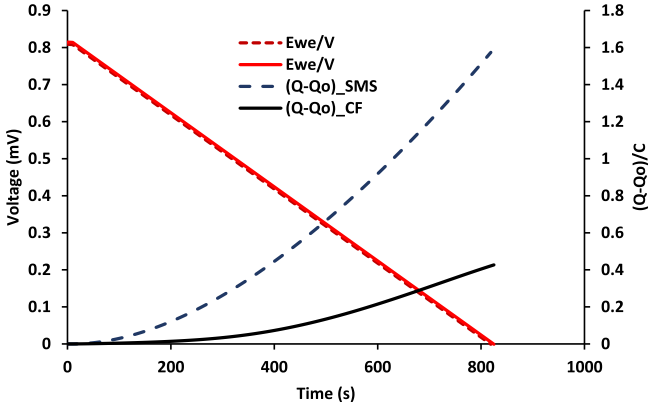

Fig. 3. Cumulative Charges of the electrodes under polarization.

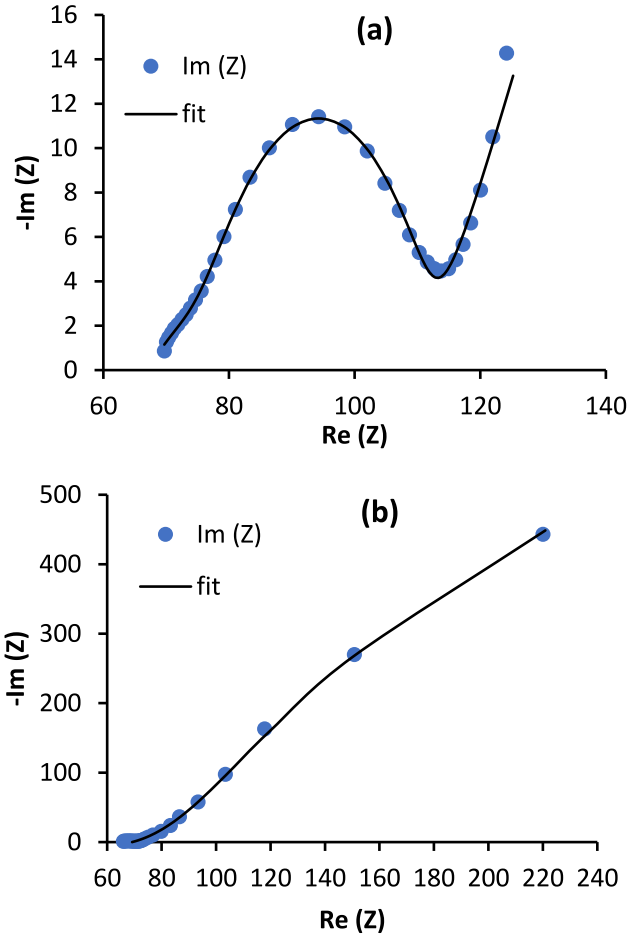

Fig. 4. Nyquist plots of (a) SMS-based TMFC and (b) CF-based TMFC.

Table 2
Impedance kinetics of the electrodes.

EM	R_{Ohmic} (Ω)	C2 (F)	R_{ct} (Ω)	Q3 (F. s^{a-1})	a	s3 ($\Omega.s^{-1/2}$)
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

These EIS findings align perfectly with the LSV results, which indicated the superior performance of the SMS electrode due to its lower internal resistance (R_{int}). Importantly, EIS further deconstructs the components of R_{int} 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. The superior performance of the SMS electrode has been attributed to accelerated electron transfer, which results from its lower resistance and larger surface area. In contrast, the larger voids between the fibers of the CF electrode have been cited as contributing factors to its poorer performance in TMFCs. These voids are often occupied by non-conductive soil particles, leading to increased internal resistance and inefficient electron transfer [37]. Additionally, as highlighted by Ref. [31], diffusion limitations of reduced or oxidized species within the CF electrode further hinder its performance. As presented in Table 2, the lower s3 of the SMS electrode indicates enhanced oxygen diffusion through the electrode, contributing to its superior performance.

4. Conclusions

This study conducted a comparative analysis of the performance and impedance spectroscopy of a surface-modified stainless-steel mesh (SMS) and carbon-felt (CF) electrode within a terrestrial MFC (TMFC) aimed at bio-power production. The SMS electrode displayed superior performance compared to the CF electrode. This was 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 in the CF electrode, hindering electron transfer between microbes and the electrode. Conversely, the lower charge transfer resistance 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 terrestrial MFCs. Furthermore, the application of EIS to the TMFC study offers a valuable tool for dissecting the complex interplay between electrode properties and MFC performance, guiding the development of more efficient TMFC systems in the future.

The long-term operational stability tests and scalability considerations are crucial for the practical implementation of TMFCs. Therefore, future studies should evaluate the long-term stability and scalability of SMS electrodes in TMFCs. Additionally, the performance of SMS electrodes with various surface modifications should be explored alongside their effectiveness in real-world applications, such as wastewater treatment systems and soil pollutant remediation. Such studies would provide deeper insights into the practical utility and versatility of these electrodes in diverse environmental settings.

CRediT authorship contribution statement

Meshack Imologie Simeon: Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Amarachi C. Alaka:** Writing – review & editing, Writing – original draft, Visualization, Data curation. **Peter Daniel:** Writing – review & editing, Writing – original draft, Visualization. **Olalekan David Adeniyi:** Writing – review & editing, Writing – original draft, Visualization, Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

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