Carbon NanoStructures/Metal Oxide Modified Anodes in Microbial Fuel Cells for Wastewater Treatment/Power Production

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First, I thank Allah the Almighty for supporting me during the preparation of this work. Second, I would like to appreciate ENI's support for my study in Itlay. I'd like to deeply thank my supervisor Prof Antonio Marzocchella for his unconditional support and guidance, and many of my professors at UNINA Including Prof Stefano Marrone, Marco Moracci and Prof Pierro Salatino. I would also like to extend my deep gratitude to Prof Marielle di Lorenzo at the University of Bath for the precious experience at her Lab under her guidance, and deep thanks to Prof. Ehab Elsawy for his support and guidance at his lab at the American University in Cairo.

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Riassunto

L’attività svolta nell’ambito del programma di Dottorato di Ricerca, finanziato da ENI Energy Corporate nell'ambito del programma Young Talent from Africa, è focalizzato sul progresso della tecnologia delle celle microbiche a combustibile (MFC), con particolare attenzione alle Soil Microbial Fuelle Cell (SMFC) alimentate da acque reflue. Le SMFC rappresentano un'opzione promettente per la generazione di energia pulita grazie alle loro semplici condizioni operative, ai bassi costi di esercizio e ai requisiti infrastrutturali minimi. Lo studio mira a ottimizzare i materiali anodici per aumentare le interazioni microbiche e le prestazioni generali delle MFC, consentendone l'utilizzo in applicazioni concrete.

La tesi si compone di tre parti principali: la rassegna della letteratura, la realizzazione degli anodi e la simulazione di processo basata sull'apprendimento automatico per la previsione delle prestazioni. La rassegna della letteratura riassume l'attuale tecnologia MFC, le sfide nella preparazione dei materiali degli elettrodi e gli sviluppi per una migliore efficienza energetica e stabilità.

Il Capitolo 3 descrive la costruzione di anodi supportati da nanobastoncini di ossido di cobalto-molibdato ($CoMoO_4$), selezionati per la loro elevata conduttività. I dati sperimentali rivelano un notevole miglioramento dell'attività elettrochimica e dell'adesione microbica, ad esempio densità di potenza doppie rispetto agli anodi a base di carbonio. Le prestazioni migliorate illustrano il potenziale dell'utilizzo di materiali avanzati per migliorare le prestazioni delle celle a combustibile microbiche (MFC). Il Capitolo 4 esamina gli elettrodi in acciaio inossidabile trattati con ossidazione a fiamma con altre modifiche superficiali. Mentre i test di laboratorio hanno indicato che gli elettrodi modificati hanno mostrato prestazioni migliori rispetto ai loro equivalenti non modificati, i test sul campo hanno indicato che l'acciaio inossidabile non modificato ha prestazioni migliori rispetto alle versioni modificate. Questa osservazione evidenzia l'importanza della colonizzazione microbica su superfici più semplici, il che implica che materiali economici e facilmente conformabili potrebbero rivelarsi più efficaci nell'uso reale.

Il Capitolo 5 descrive tecniche di apprendimento automatico e reti neurali per prevedere le prestazioni delle celle a combustibile a media densità (MFC) a partire da dati ottenuti sperimentalmente per diverse configurazioni. Sebbene si siano osservati alcuni trend positivi nelle previsioni, l'accuratezza è stata limitata dall'elevata dispersione dei valori di produzione di energia nei dati. La potenza erogata è influenzata dalle variazioni delle condizioni del sistema e, pertanto, necessita di set di dati ampi e continui per catturare questo ampio intervallo. Il capitolo discute le sfide dell'applicazione dell'apprendimento automatico alle celle a combustibile a media densità (MFC) e sottolinea l'importanza di condizioni sperimentali standardizzate e di dati di qualità per migliorare la robustezza delle previsioni.

I risultati della tesi di dottorato contribuiscono alla comprensione della tecnologia MFC studiando nuovi materiali anodici e il loro impatto sulle prestazioni in laboratorio e su scala pilota. Dimostra il potenziale di materiali a basso costo e abbondanti e la sfida di modellare i sistemi MFC tenendo conto del futuro sviluppo della tecnologia sostenibile delle celle a combustibile microbiche.

Summary

This PhD study, supported by ENI Energy Corporate within the Young Talent from Africa program, is dedicated to advancing microbial fuel cell (MFC) technology with special attention to soil microbial fuel cells (SMFCs) powered by wastewater. SMFCs represent a promising option for generating clean energy due to their simple operating conditions, low-cost inputs, and minimal infrastructure requirements. The study seeks to optimize anode materials to increase microbial interactions and general MFC performance and enable real-world applications.

The thesis has three main parts: a literature review, anode fabrication, and a process simulation based on machine learning for predicting performance. The literature review summarises current MFC technology, challenges in preparing electrode materials, and developments for better energy efficiency and stability.

Chapter 3 outlines the construction of cobalt-molybdate oxide ($CoMoO_4$) nanorod-supported anodes selected due to their high conductivity. Experimental data reveal considerable improvement in electrochemical activity and microbial attachment, e.g., power densities twice that of plain carbon-based anodes. The improved performance illustrates the potential for utilizing advanced materials to improve microbial fuel cell (MFC) performance. Chapter 4 examines flame oxidation-treated stainless steel electrodes with other surface modifications. While laboratory tests indicated that the modified electrodes showed improved performance over their unmodified equivalents, field tests in actual practice indicated that unmodified stainless steel performed better than the modified versions. This observation highlights the importance of microbial colonization on simpler surfaces, implying that low-cost and easily conformable materials might prove more effective in actual use.

Chapter 5 describes machine learning techniques and neural networks to predict MFC performance from experimentally obtained data for various configurations. Although there were some positive trends in the predictions, accuracy was limited by the high scatter in the power production values in the data. The power output is influenced by variations in system conditions and, as such, needs large and ongoing datasets to capture this wide range. The chapter discusses the challenges of applying machine learning to MFCs and emphasizes the importance of standardized experimental conditions and quality data for improving prediction robustness.

This PhD thesis assists in understanding MFC technology by investigating new anode materials and their impact on performance at the laboratory and practical scales. It demonstrates the potential for low-cost, abundant materials and the challenge of modelling MFC systems with the future development of sustainable microbial fuel cell technology in mind.

1 Introduction.

1.1 Scientific background.

Resource depletion due to increasing human needs has been a rising concern. Water and energy are two of the most critical assets that may hinder human development. Finding sustainable energy resources and ensuring water sustainability has become a must in modern society. From this point of view, renewable energy resources and innovative wastewater treatment are required to secure the future of modern society. One solution to both issues is to couple energy production and wastewater treatment. Few systems offer this opportunity: they include biofuel cells such as microbial fuel cells (MFCs). MFCs are fuel cells that use organic matter and bacteria as catalysts. Microbial fuel cells proved promising when investigated in treating domestic wastewater (Logan et al., 2006a), animal waste (Min et al., 2005), and food waste (Jia et al., 2013).

The MFC is promising in decentralizing wastewater treatment and providing accessibility to wastewater treatment, especially in developing countries such as Africa. Efficient, commercially available, durable, and scalable MFC units – and cell components - shall facilitate using the MFC in rural areas without central treatment systems. Easy access to wastewater treatment shall decrease the amount of untreated wastewater discharged to water bodies worldwide. In developing countries such as Africa, it is estimated that more than 80 % of wastewater is discharged into water bodies without adequate treatment, increasing the health risks humans face in these areas(UNESCO), 2009). Unfortunately, there are 842000 deaths yearly resulting from inadequate sanitation and lack of safe water. 54% of these deaths could be prevented by sanitation and safe drinking water(*Sanitation*, n.d.).

1.1.1 MFC scale-up and soil microbial fuel cells (SMFCs)

Several factors currently limit the widespread implementation of microbial fuel cells (MFCs). Soil microbial fuel cells (SMFCs) are a promising variant of MFC technology, which can be readily integrated into soils and wetland environments. They offer significant cost advantages by utilizing the soil as a natural separator rather than relying on synthetic membranes, as shown in Figure 1-1. Moreover, SMFCs typically employ air cathodes - usually composed of plain graphite felt – suppressing the need for expensive catalysts. This approach makes SMFCs appealing as a sustainable, economical, and environmentally friendly solution for energy generation and carbon-neutral bioremediation of contaminated sites(Chakma et al., 2025)

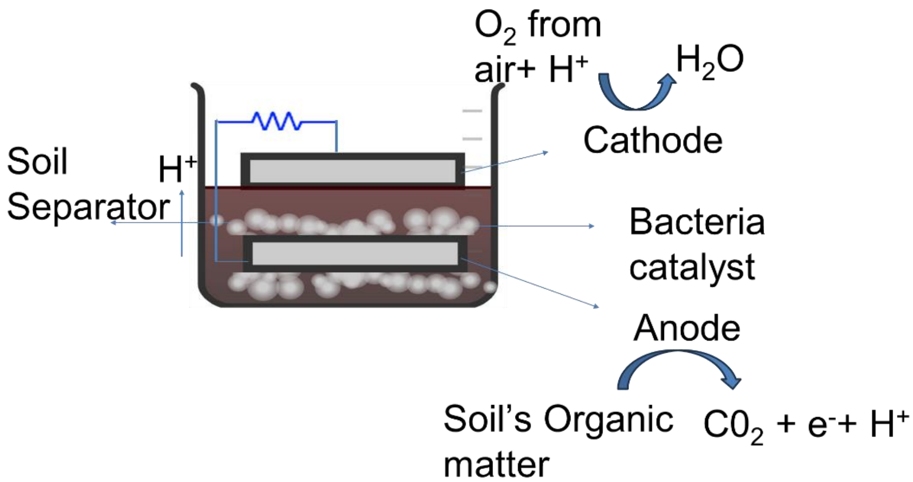

Figure 1-1:Schematic diagram of a Soil Microbial Fuel Cell (SMFC)

The attention in the present PhD thesis is focused on cobalt molybdate oxide (CoMoO4)-modified graphite felt (GF) electrodes in SMFCs. These modified electrodes may enhance the formation of robust electroactive biofilms, thereby significantly improving electrogenic performance. The focus is on (CoMoO4)-modified graphite felt (GF) because of the promising performance proved for cobalt-based anodes, particularly when coated with polymers(Dhillon et al., 2023). A bimetallic oxide is anticipated to provide enhanced stability and conductivity without additional polymer coatings. SMFCs offer numerous practical advantages, including a straightforward design, long-term operational stability, and minimal maintenance requirements, making them particularly suitable for remote applications. Moreover, the technology provides attractive prospects for self-sustaining bioremediation processes in contaminated soils.

Despite these advantages of SMFCs, one major challenge affecting the scalability of MFC/SMFC technology is the efficiency of power generation, which is often reduced by energy losses within the system. Specifically, these losses are greatly influenced by the characteristics of the anode, which serves as the critical interface facilitating electron transfer from bacteria to the external circuit of the MFC/SMFCs. Thus, identifying and developing efficient anode materials is essential for optimizing electrochemical performance and maximizing power output.

Furthermore, the bacterial communities within the microbial fuel cell significantly affect its overall performance. The composition and structure of these microbial communities are crucial, as they directly mediate the oxidation of organic matter in wastewater and electron transfer to the anode. Consequently, a deeper understanding of microbial community dynamics and optimizing operational parameters are vital for advancing the efficiency and practical performance of SMFC systems.

1.1.2 Anode state-of-the-art

1.1.3 Biofilm

A biofilm is an extracellular polymeric substance (EPS) encased, surface-adhering microbial community (Toutain et al., 2004). Conventional theory categorizes biofilm structure around three primary stages of development: initial attachment, maturation, and detachment (Read et al., 2010) (Greenman et al., 2021). The EPS physically immobilize the bacteria while simultaneously providing them the opportunity for cell-to-cell contact and communication(Read et al., 2010).

Forces govern the initial attachment of cells to a solid substratum include advective flow and three types of non-covalent forces: (1) Lifshitz–van der Waals (L.W.), (2) Lewis acid-base (A.B.) and (3) electrical double layer (E.L.). Furthermore, many interacting variables are thought to be important in cell attachment and biofilm formation (Donlan, 2002). These include the types of surfaces, their texture, roughness and hydrophobicity. The cell surface properties include flagella, pili, capsules and/or slime formation (extracellular polymeric substances) and the fluid feedstock. The latter provides nutrient types and concentrations, pH, redox, presence of divalent metal ions, oxygen, inhibitory compounds and temperature.

1.1.4 Characteristics of efficient anode.

The MFC is based on the extracellular activity of bacteria to transfer electrons to the anode. Bacteria are attached to the anode, and electrons are transferred to the anode at the surface. For enhanced MFC performance, an anode should have the main features listed hereinafter.

Biocompatibility: The anode material should be favourable for bacterial growth, requiring a toxins-free surface. The most used anode is carbon-based in the form of carbon, graphite, or graphene, as well as metal and metal oxides, and conductive polymers such as polyaniline and polypropylene.

Surface structure: The anode's surface should provide a high specific surface area to transfer electrons from the bacteria to the electrode. Most developments on anode use more rough surfaces and porous material. Also, the development of material allowed the emergence of microporous and nonporous materials, microfibers, and nanocomposite materials.

Conductive Anode: The anode receives the electron from the bacteria at its surface and transfers it through the cathode's external circuit. Surface electron transfer affects the resistance of the MFC and the voltage losses. The surface electron transfer is tested in the material before use in MFCs.

Chemical and mechanical stability: The anode should be corrosion-resistant and mechanically stable for long-term running and scaling.

1.1.5 Materials used as anode

1.1.6 Carbon materials

Carbon-based materials are the primary material used as anode in an MFC because they are free of toxins, chemically stable, and characterized by good conductivity. Many improvements have been proposed for carbon. Carbon materials are used as amorphous Carbon and other well-defined Carbon.

1.1.6.1 Graphite:

Graphite is used as an anode for its high conductivity. Researchers used graphite in different forms to enhance power production in MFCs. It was shown that porous graphite electrodes such as graphite felt and graphite foam have higher power production than graphite rods. Graphite felt generated a threefold electric current than the rod electrode, while graphite foam produced 2.4 times higher than the rod. (Chaudhuri & Lovley, 2003).

The graphite brush was used as an anode and provided 10 times higher power generation than the graphite rod (Bruce Logan et al., 2007). Figure 1-2 shows the different forms of graphite used as an anode in MFCs and the effect of the shape and surface structure on the performance of the MFCs.

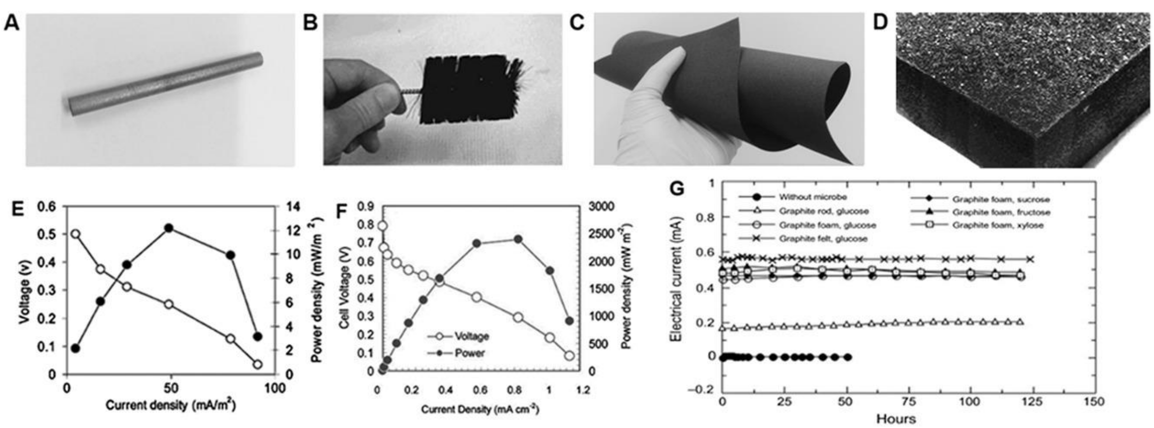

Figure 1-2: Photographs of: graphite rod (A), graphite brush (B), graphite felt (C), and graphite foam (D). E) Power generation related to the graphite rod. F) The power density and cell potentials of a device equipped with a graphite brush anode. G) Effect of different graphite electrode structure and sugar on electricity generation of MFCs (S. Li et al., 2017)

Graphite surface's functionalization to enhance surface hydrophilicity and surface charge has been investigated (Picot et al., 2011) in less defined carbon materials such as carbon veil. Surface heating decreases the oxygen-to-carbon (O/C) ratio and increases the surface electron transfer. Moreover, ammonia treatment is done to functionalize the carbon with nitrogen doping and increase the nitrogen-to-carbon ratio (N/C), enhancing the surface charge and improving MFC's performance. For example, after a carbon mesh was treated with ammonia gas, the output power increased significantly, from 893 mW m^{-2} (45 W m^{-3}) to 1015 mW m^{-2} (51 W m^{-3})(X. Wang et al., 2009).

1.1.6.2 Graphene coatings of carbon cloth/stainless steel mesh

Liu et al. (2012) proved that the graphene coating on carbon cloth improved power density and energy conversion efficiency by 2.7 and 3 times. The improvement is attributed to the high biocompatibility of graphene, which promotes bacteria growth on the electrode surface, results in more direct electron transfer activation centres, and stimulates the excretion of mediating molecules for a higher electron transfer rate. (J. Liu et al., 2012)

Call et al. (2017) presented pristine graphene-based electrodes at an MFC's anode and cathode. Using graphene-based aerogel anodes, they achieved a volumetric peak power output (P.V.) of up to 3.51 ± 0.50 W m^{-3}, a relatively high power production. They attributed the enhanced MFC output to the graphene-based electrode's improved surface area, enhanced conductivity, and catalytic surface groups. In addition, they achieved a 500-fold increase in $P_{.V.}$ to 1.3 ± 0.23 W m^{-3} when using a graphene-coated stainless steel (S.S.) air cathode, compared to an uncoated S.S. cathode, demonstrating the feasibility of a platinum-free, graphene catalyzed MFCs.(Call et al., 2017).

3D graphene was recently proposed as the anode in MFC. The MFCs equipped with anodes were characterized by less internal and ohmic resistance than the MFCs equipped with graphene oxide. Figure 1-3 shows the surface of the 3D structure.(Pareek et al., 2019)

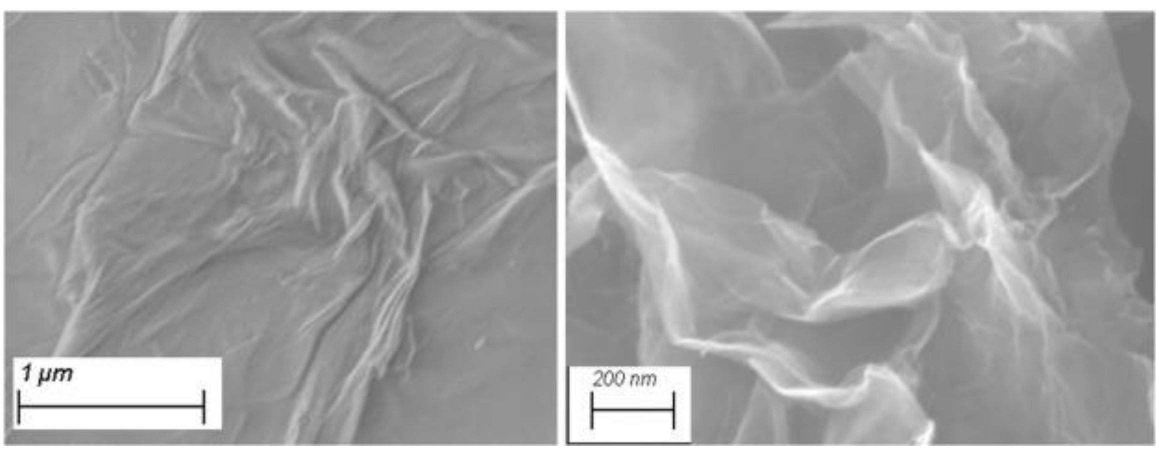

Figure 1-3:FESEM images of (a) Graphene Oxide (G.O) . and (b) 3D graphene on carbon cloth.(Pareek et al., 2019)

Carbon black, activated carbon, and carbon particles: the deposition of these carbon materials on various substrates increases the specific surface area of the active anode and can also increase the interaction between the electrode surface and the microorganisms (Yuan et al., 2009). Ieropoulos and co-workers coated a carbon veil and cloth with carbon black and poly(tetrafluoroethylene) (PTFE) (You et al., 2014). They measured an output power density of 60.7 mW m^2 and 50.6 mW m^2, respectively, almost two times higher than the power measured for the unmodified carbon veil anodes.

Carbon nanotubes: carbon nanotubes have a large specific surface area, good mechanical properties, excellent stability, and high conductivity, which help enhance surface electron transfer (P. Cheng et al., 2024; S. Zheng et al., 2015).

Polymers: conductive polymer chains can adsorb proteins and other biomolecules in culture media, stimulating biofilm formation and guiding microorganism growth. Polyaniline (PANI) and pyrrole (PPy) are characterized by good biofilm adhesion, electrical conductivity, and environmental stability. Hence, they have been widely studied for surface modification of anodes based on carbon materials in MFCs(C. Li et al., 2011).

1.1.7 Metal, metal oxides, and metal composites.

1.1.7.1 Metals/metal oxides

Metals have two advantages over carbon materials. They have the mechanical strength for scaling up. Their electric conductivity lies two to three orders of magnitude higher than graphite. Any increasing electrode resistivity in an electrochemical system like a (microbial) fuel cell leads to a decreasing cell voltage and power. The power loss may be negligible for small, lab-based systems, but it may result in the complete collapse of the electrochemical performance in an up-scaled system.

In general, the metal should be electrochemically inert in the potential operational window of the bioelectrochemical system. This feature may be accomplished with the use of either electrochemically noble or electrochemically passivated metals. Noble metals - such as gold and platinum – are characterized by high electrochemical reversibility but they are expensive. Hence, inexpensive base metals are used as anode material, provided that a compact oxide layer (passivation layer) protects the metal from further oxidation.

Baudler et al(Baudler et al., 2015) studied various metals and found that noble metals gave current as follows: gold (1175 μA cm^{-2}), silver (1119 μA cm^{-2}), and copper (1515 μA cm^{-2}), which is similar or even slightly higher than that of graphite (984 μA cm^{-2}). Moreover, they proved that passivated stainless steel was characterized by the highest performance for a passivated metal (674 μA cm^{-2}), followed by nickel (384 μA cm−2). The current densities at cobalt and titanium were negligible compared to the other electrode materials.

Other metals, such as molybdenum, were used as nanoporous Mo_2C functionalized anodes. The Mo_2C improved microbial electrocatalysis in MFCs with a 5-fold higher power density and long-term stability of electricity production. The significant enhancement is attributed to the introduction of a rough Mo_2C nanostructural interface into macroporous carbon architecture for promoting microbial growth with significant excretion of endogenous electron shuttles (flavins) and rich available nanopores for enlarging the electrochemically active surface area. (Zou et al., 2017)

1.1.7.2 Metal composites

Metal can be used in composites with graphene (G) or polymers. Song et al.(Song et al., 2017) reported tests with iron oxides used in G/Fe_3O_4 composite on carbon paper (CP). The microbial electricity generation was tested with the carbon paper (CP), Carbon paper/Graphene (CP/G), CP/Fe_3O_4, and CP/G/Fe_3O_4 anodes at a poised potential of 0.2 V (vs SCE). The results pointed out that the current and power densities produced by CP/G/Fe_3O_4 anode were much higher than those produced by CP, CP/G, and CP/Fe_3O_4 anodes. The enhanced performance was due to the increased surface area by adding Fe_3O_4, which facilitated bacterial growth on the anode. Moreover, the graphene increased the electric conductivity at the surface, enhancing the performance.

Mohamed et al. (Mohamed et al., 2021) in 2021 used tungsten carbide (W.C.) and tungsten carbide on reduced graphene oxide (W.C. + rGO) nanolayers as anode catalysts in MFC for the simultaneous generation of power and treatment of wastewater. They found that the unique features of WC/CF and W.C.+rGO/C.F. anodes - surface area, biocompatibility, structure morphology, and catalytic activity - resulted in significant performance improvements. In particular, W.C. + rGO/C.F. exhibited a 4.4-, 7.6-, and 2.1-fold power density, current density, and coulombic efficiency relative to the benchmark C.F. anode.

1.1.8 Stainless steel - a durable and commercially available anode.

Many studies focused on stainless steel (S.S.) as a commercially available electrode, that was characterized by enhanced performances with respect to carbon-based materials, especially at high potentials (Pocaznoi et al., 2012). However, some drawbacks must be addressed to enhance the S.S. performance as an anode for MFCs. Two critical disadvantages are the S.S. smooth surface that hinders biofilm adherence and the S.S. passive semiconducting surface film that reduces the surface conductivity and biocompatibility (Pocaznoi et al., 2012). To this end, the current research on S.S. anodes focuses on increasing the surface roughness and the electrochemically active surface area according to various methods. Some solutions include acid treatment (Peng et al., 2016; S. Zheng et al., 2015), mechanical alteration(Pocaznoi et al., 2012), carbon modification(Peng et al., 2016; S. Zheng et al., 2015), flame oxidation (Ledezma et al., 2015; Yamashita et al., 2016), composite modification (Liang et al., 2017), and using stainless steel foam or fibre felt(Hou et al., 2014; Ketep et al., 2014). For example, flame-oxidized S.S. increased the MFC power by 184% and 24% more than the bare S.S. and carbon cloth anodes, respectively. The enhancement was related to the formation of hematite nodes that caused surface roughening and enhanced the surface's electrochemical activity and biocompatibility (Yamashita et al., 2016). Lamp et al. [2](Lamp et al., 2011) reported that the surface roughness was increased by depositing carbon nanotubes on the surface of bare S.S., They used flame deposition that increased the produced power from 2.9 to 187 mW/m(Lamp et al., 2011). Acid-treated SS mesh coated with carbon black (C.B.) has also been tested as an MFC anode and showed a high activity enhancement(S. Zheng et al., 2015).

The analysis of the presented literature suggests that many advances have been made in the anode material of Microbial fuel cells. Much research has been done on every material, including metals, carbon, biomass, etc. Also, the process of anode fabrication has provided many trials and improvements. Many materials have been used and many modifications. However, Many of the Anodes were only tested at the lab with synthetic wastewater outside the real conditions where they should be operated.

1.2 AI modeling

The real literature gap in MFCs anode and MFCs, generally, is that the data about the system performance in literature are not reported to reference either in environmental conditions or standardized setups. Many researchers are focused on developing system parts, including anodes, cathodes, membranes or operating parameters. Researchers focus on fabricating new anodes for the MFCs, claiming they have the best performance or are more sustainable or economical. These claims could never be supported because every lab works in different conditions, with different systems and even different inoculum (bacteria strains). These allowed us to consider addressing these gaps.

A total of 11,397 articles were published on the topic till 2020(Naseer et al., 2021). The anode has been mentioned as a keyword in 2.13% of those papers as seen in Table 1-1, which are nearly more than 242 articles. From the amount of literature published on anode fabrication, the number of research papers on the anode is expected to be much higher than that. For example, many papers were published on anode modification without having anode as a keyword. Table 1-1 shows the distribution of the most common keywords used in microbial fuel cells.

Table 1-1: *Most frequent keywords used in the MFCs field*(Naseer et al., 2021).

Most frequent keywords used in the MFC field.

Keywords	Frequency of occurrence (%)
Performance	9.92
Electricity generation	8.19
Microbial fuel cell	4.67
Waste water treatment	4.31
Waste reduction	3.13
Anode	2.13
Cathode	2.10
Bacteria	2.09
Electron transfer	2.05
Degradation	1.85
Membrane	1.71
Oxidation reaction	1.66

However, the anode performance can not be judged without considering all the other factors. The effect of the various factors and their correlation can hardly be estimated. Recently, machine learning models solved these problems by training a model on the data and then letting the model predict the output and present an equation. The factors affecting the anode performance are scattered - as reported in Figure 1-4 - and we do not know how they impact each other. In such cases, artificial intelligence (AI) can mimic the human mind to process the data and produce a model after training.

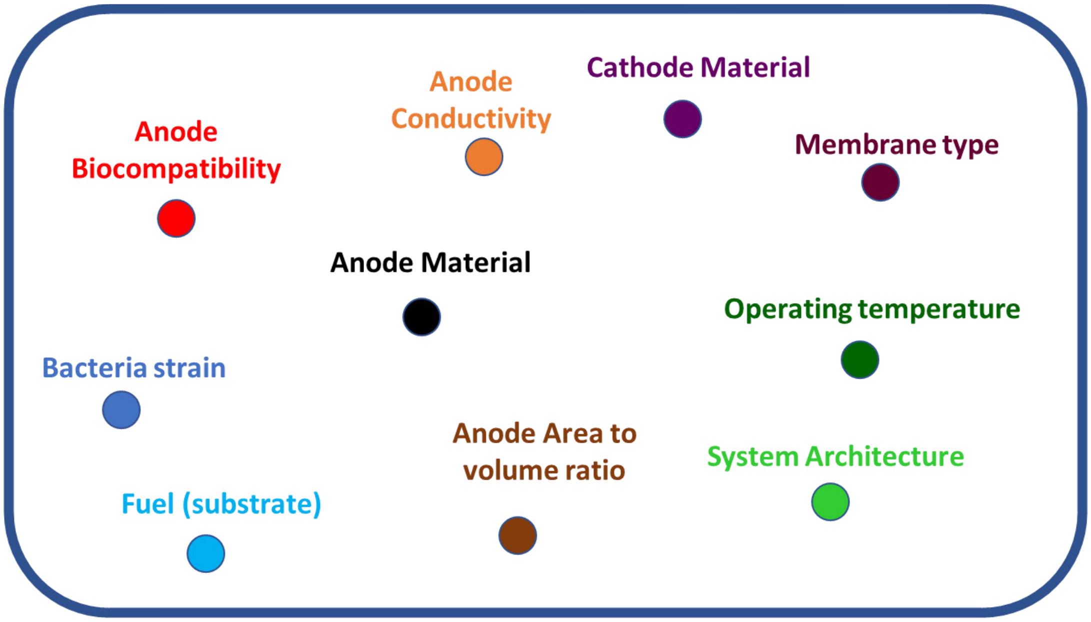

Figure 1-4:Factors that impact the performance of MFCs.

For instance, machine learning on other wastewater treatment systems, such as wetlands, where artificial neural networks are used to model the factors of the wetlands that treat wastewater, which also have many interacting factors(Martins et al., 2020) (Albalasmeh et al., 2022). AI has also been used to model many industrial biotechnological applications and many other applications. Hence, MFCs can follow this lead as a biological wastewater treatment system. (Albalasmeh et al., 2022; Martins et al., 2020; Naseer et al., 2021).

Generally, machine learning is the future of all sciences and is currently accessible in many resources. The deeper and broader analysis and modelling incorporating literature shall provide future researchers and funding institutions with knowledge that can guide them better in experimental design before going to experimental work. This kind of guiding model is valuable.

1.2.1 The Algorithms to be used

Various machine learning algorithms can be used in bioelectrochemistry applications including MFCs. However, the accuracy of such algorithms to predict the performance depends on the datasets, the dependency of the features on each other and their correlation, and the amount of consistent data entries available. The algorithms can have various prediction accuracies based on the various aforementioned factors. Generally, the suitability of the algorithm for prediction depends on the suitability of the Algorithm's built-in equations, constraints, and concepts to the features, dataset and required model. Below is a brief explanation of some most commonly used algorithms in regression.

1.2.1.1 Decision tree.

The decision tree is a model that consists of a base node split into a series of decision nodes to increase the homogeneity of the node to the target. The splitting happens according to a set of logical if-then conditions based on the training data. The end nodes (Leaf nodes) represent all the possible outcomes of the training, as shown in the schematic diagram in Figure 1-5. (Dupont et al., 2020)

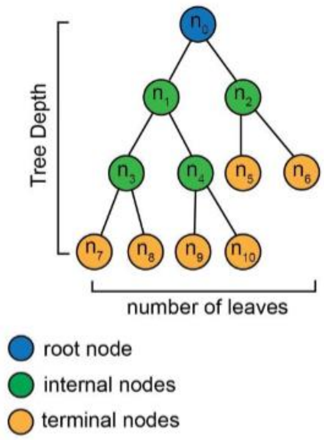

Figure 1-5: Schematic depiction of a decision tree. (Dupont et al., 2020)

1.2.1.2 Support vector machines (SVM)

A support vector machine (SVM) is a binary linear classifier. SVM aims to classify data and find the optimal boundary, *i.e.*, the boundary with the most significant distance between points (margin). The SVM algorithm maps each object as a point in space and finds the linear equation that separates the data with the most significant margin Figure 1-6a. New data is classified based on the region in which it falls. (Dupont et al., 2020)

1.2.1.3 Neural Networks.

The name and structure of the methodology are inspired by the human brain, mimicking how biological neurons signal to one another. They consist of an input layer, hidden layer/s, that contains the neurons that try to find hidden relationships between the input layer and hidden layer and the output layer. A schematic diagram of a neural network is shown in Figure 1-7.

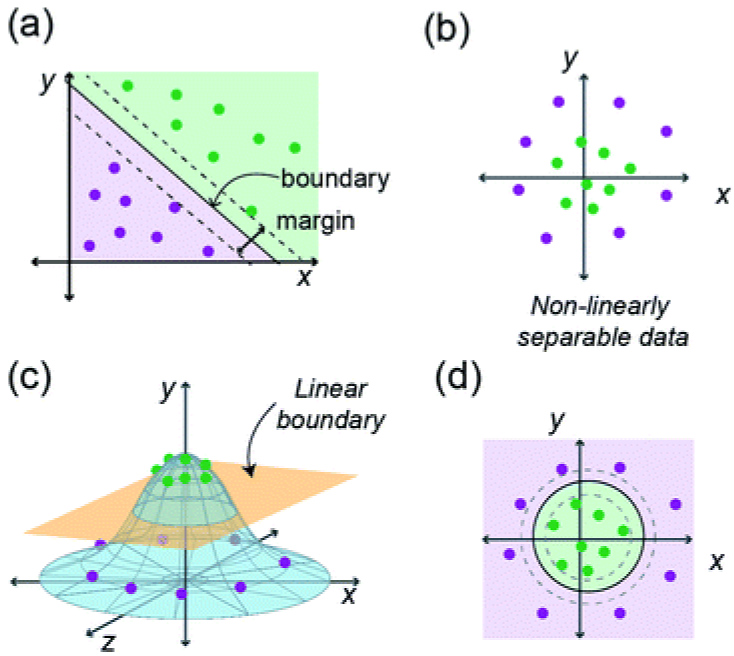

Figure 1-6: (a) Linearly separable data divided by a boundary and margin, (b) non-linearly separable data in 2D, (c) data from (b) transformed into a higher dimension (3D) separated by a linear boundary and (d) boundary in (c) projected onto a 2D plane. (Dupont et al., 2020)

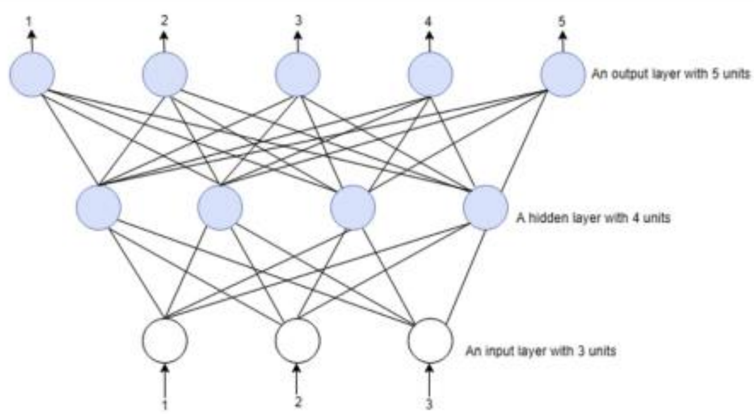

Figure 1-7: Two-layered feedforward neural network.(Isaac Abiodun et al., 2018)

1.2.1.4 Random forest.

It uses the bagging of multiple decision trees as a sub-model to get a highly correlated model. The bagging meta-algorithm employs a combination of bootstrapping and aggregating. Bootstrapping creates many sub-samples of the whole dataset by random selection (with replacement). Each of these sub-samples is trained on the selected model in parallel. The final output is obtained by aggregating the output of each model by either averaging (for regression analysis) or voting (for classification). Figure 1-8. (Dupont et al., 2020)

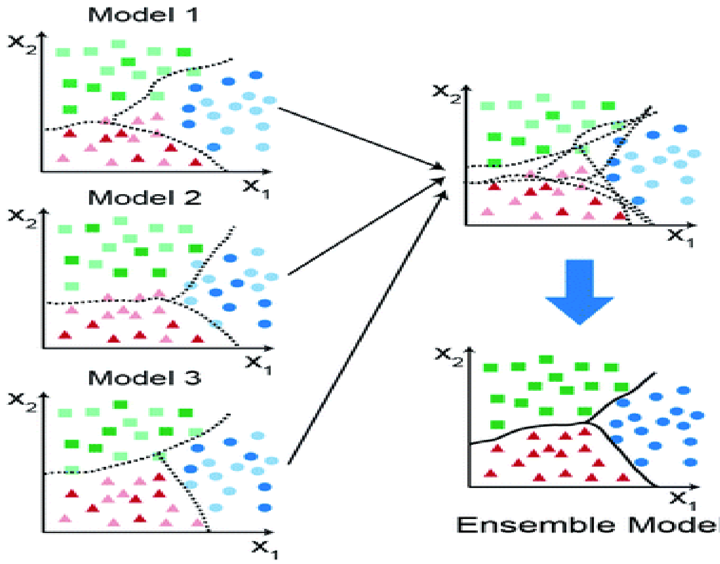

Figure 1-8: Schematic of a bagging ensemble method. (Isaac Abiodun et al., 2018)

1.2.2 VALIDATION METHODS

- The data was split into 80% train sets, and the other 20% was used to test the data.
- In the 5k fold validation method, the model is split into 80% training and 20 % test set. Then, the train set is split into five different folds, each set to be the test set once, and the other four are the train set. The model can be validated on each fold of the trainset and then tested on the test set, as shown in Figure 1-9.

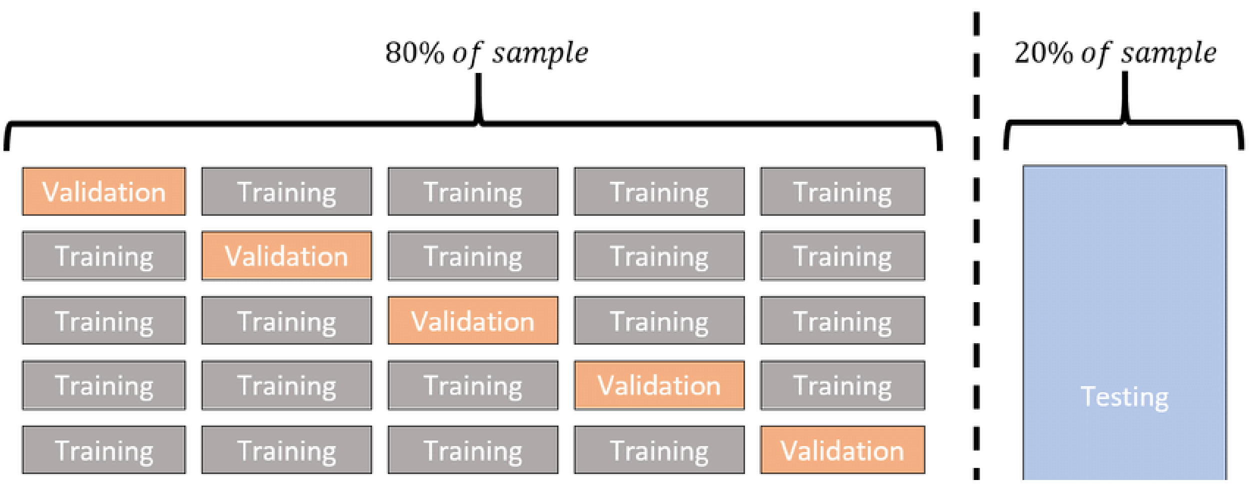

*Figure 1-9: K fold validation and testing. (*3.1. Cross-Validation: Evaluating Estimator Performance — Scikit-Learn 1.6.1 Documentation*, n.d.)*

2 Aim of the PhD thesis

ENI Energy Corporate funded this thesis as the award of the Young Talent from Africa's initiative to fund talented African students in Italy to expand their skills in the renewable Energy research domain.

The aim of the PhD thesis has been the development of a strategy/procedure to produce an advanced anode for microbial fuel cells fueled with wastewater. The investigated case was on soil microbial fuel cells because SMFCs are a practical application characterised by: i) no sophisticated requirements and simple operating conditions; ii) low-cost economic components such as graphite felt anodes and cathodes and soil as the separator membrane. The study has been structured into three sections. The first is the literature analysis. The second regarded anode fabrication. The third process simulation regarded the integration of machine learning models in MFCs 's power generation prediction.

Chapter 3's objective: Anodes fabrication for soil microbial fuel cells (SMFCs) cobalt molybdate oxide nanorods.

The analysis of the literature suggests that numerous anode materials have been explored for microbial fuel cells (MFCs). Just a few studies have focused on their application in soil microbial fuel cells (SMFCs). Specifically, materials such as bimetallic oxides and modified stainless steel meshes have seen limited testing in SMFCs. In this work, we selected a bimetallic cobalt-molybdenum oxide ($CoMoO_4$) as an anode material due to the promising performance previously demonstrated by cobalt-based anodes, particularly when coated with polymers. A bimetallic oxide is anticipated to provide enhanced stability and conductivity without additional polymer coatings. Figure 2-1 further explains Chapter 3's objective. Enhancing the anodes is a step towards scaling up MFCs, especially in applicable applications such as SMCs.

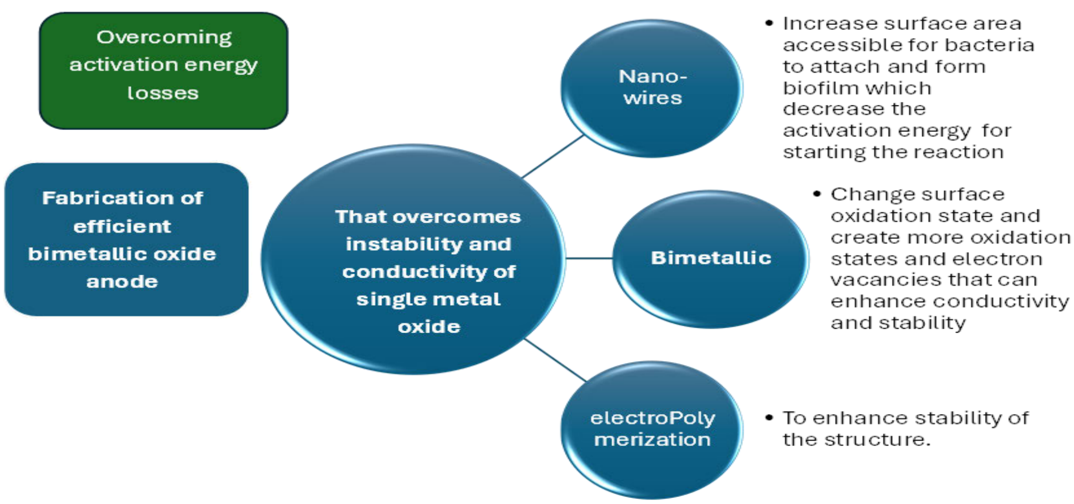

Figure 2-1: Schematic diagram for Chapter 4's objective.

Chapter 4 objective, stainless steel mesh flame oxidized modified and further introduced oxidation states.

Stainless steel electrodes modified through flame oxidation were chosen based on their superior performance to carbon-based and unmodified stainless steel anodes in conventional MFCs. However, their effectiveness in SMFCs remains untested. Given that SMFCs represent a more economically viable and practical configuration of MFC technology, further optimization of the anode material is crucial, mainly since the soil naturally provides the separator function and the cathode material has already been optimized as graphite felt, which performs better than catalyst loaded cathodes. Therefore, this research emphasizes anode development, proposing two novel electrode materials: the $CoMoO_4$ bimetallic oxide anode detailed in Chapter 2 and the flame-oxidized stainless steel electrode described in Chapter 4. Enhancing the anodes is a step towards scaling up of MFCs especially in applicable applications such as SMCs.

Figure 2-2 further explains Chapter 4's objective.

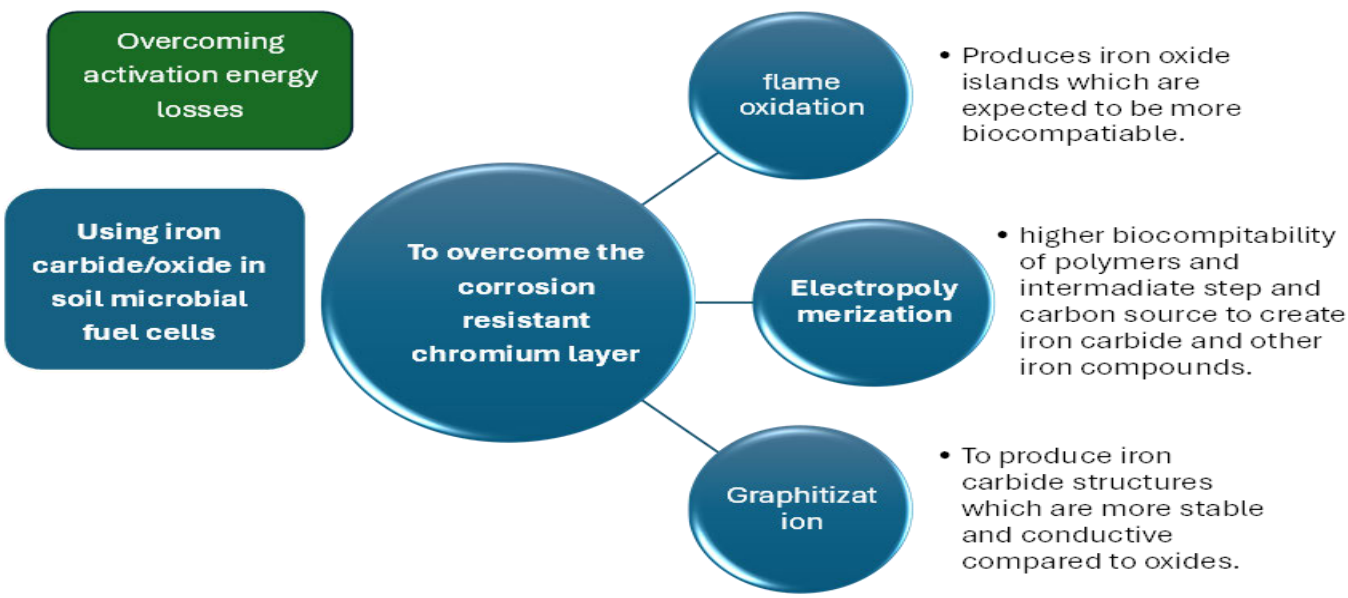

Figure 2-2: Schematic diagram for Chapter 4's objective.

Chapter 5's objective and hypothesis.

The objective of this work is to collect data on the basic features that affect the performance of MFCs by compiling results from a variety of research papers using an incremental approach—where each dataset includes one changed factor. The model is trained to detect the impact of each factor and to serve as a predictive tool for MFC performance under diverse systems and conditions. Additionally, this work aims to explore the potential need for introducing a new variable that reflects handling error or microbial community differences across studies.

In this PhD research, we approached the sustainable system of MFCs from multiple perspectives. Firstly, we focused on enhancing the performance of anodes in SMFCs. This involved exploring methods to increase catalytic activity and improve surface electron transfer by creating more conductive anodes through various surface structuring techniques. Additionally, recognizing that we live in a dynamic world that will increasingly utilize advanced tools and technology, we studied the statistics of MFC experiments conducted under different setups and conditions. Our goal was to assess their suitability for a machine learning modelling tool, which could facilitate more informed research and planning for the development of MFCs. We also evaluated the effectiveness of different algorithms in modeling the performance of MFCs.

The distinct novelty of this research involves not only the development of advanced, bimetallic oxide-polymer composite anodes [redacted], this thesis innovatively employs a machine learning modeling framework trained on diverse, multi-study data, overcoming the limitations of previous single-study predictive approaches

3 Carbon-Felt Coated with Molybdenum-Based Bimetallic Oxides Efficient Anodes for Soil Microbial Fuel Cells

Chapter 3 is organized as a paper submitted for publication in a scientific journal.

Carbon-Felt Coated with Molybdenum-Based Bimetallic Oxides Efficient Anodes for Soil Microbial Fuel Cells

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3.1 Abstract

[redacted]

3.2 Introduction

[redacted]

[redacted]

[redacted]

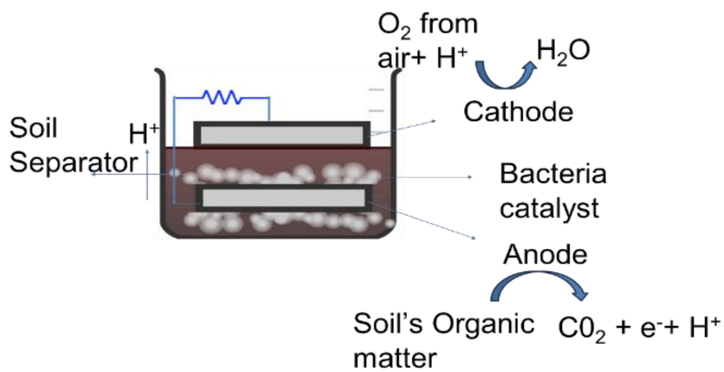

Figure 3-1:A schematic diagram of SMFCs.

[redacted]

4 - Flame-Oxidised and Polymer-Coated Stainless Steel Anodes in SMFCs: When Simplicity Outperforms Complexity

Chapter 4 is organized as a paper submitted for publication in a scientific journal.

Flame-Oxidised and Polymer-Coated Stainless Steel Anodes in SMFCs: When Simplicity Outperforms Complexity

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1 Abstract

[redacted]

4.1 Introduction

[redacted]

5 Artificial intelligence for Prediction of electricity generation in microbial fuel cells under different operating conditions

Chapter 5 is organized as a paper submitted for publication in a scientific journal.

Artificial intelligence for Prediction of electricity generation in microbial fuel cells under different operating conditions

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5.1 Abstract

[redacted]

5.2 Introduction

[redacted]

[redacted]

[redacted]

6 Conclusion

Throughout this thesis, the challenge of driving the technology of microbial fuel cells (MFC) towards real-world sustainability was tackled from several interconnected fronts. These included exploring new electrode materials, enhancing electrochemical characteristics, enhancing microbial interactions, and employing novel machine-learning approaches to improve the prediction of MFC performance. Each chapter contributed new but complementary data, building a complete picture necessary for future real-world deployment of MFC systems.

The introduction and the Aim in the first and second chapters laid the groundwork by highlighting important areas influencing MFC efficiency, namely the design and surface characteristics of anode materials. It also highlighted the growing significance of computational tools and predictive analytics in modern research. Through the integration of empirical experimentation and machine learning, the research aimed at technical improvement and developing a wider and more applied framework for further advancement.

Chapter 3 detailed novel means to enhance carbon-based anodes by surface modification of carbon felt (CF) by cobalt molybdate ($MoCoO_4$) nanowires and further by a conductive polypyrrole (PPy) coating. Thus, The composite material formed in the present work (CMNRs-GF) significantly improved microbial adhesion and electrochemical performance, achieving power outputs nearly two times greater than unmodified carbon felt. Systematic cyclic voltammetry and electrochemical impedance spectroscopy analyses confirmed substantial improvement in microbial electron transfer efficiency and reduced inner resistance. Notably, while the PPy coating decreased the peak power marginally, it raised the electrode's stability considerably under prolonged real-world operation, thus emphasizing a key practical advantage.

Chapter 4, on the other hand, explored some of the surface treatments on stainless steel (SS) anodes, including flame oxidation, polymer coatings, and graphitization. [redacted]

Chapter 5 further developed this integrative approach by employing machine learning, particularly neural networks, to model and predict MFC performance from diverse experimental data. The results demonstrated very good prediction capability, with $R^2 > 0.85$, validating the application of machine learning to manage complex data efficiently. Though, of course, problems ensued inevitably, largely compromised accuracy with tighter training/validation conditions applied. Reduced accuracy highlighted the critical data consistency, quantity, and quality issues. Accordingly, the chapter strongly advocated standardizing experimental conditions, improving data-sharing practice, and integrating comprehensive microbial and electrochemical characterizations to enhance future model reliability and usefulness. [redacted]

[redacted]

Overall, the thesis identifies several underlying issues relevant to the practical production of microbial fuel cells:

- Nanostructured anodes, constructed with great care for excellent performance and microbial interaction, are still confronted with the trade-off of high efficiency and operational stability for extended durations.
- In general, untreated stainless steel and simply treated Stainless steel perform exceptionally well in actual soils, indicating the importance of microbial adaptability and cheap simplicity.
- There is vast potential in machine learning to predict and optimize MFC performance, provided it relies heavily on the quality and availability of data. Thus, there is a need for more stringent standardization and cooperation within the field.

This research provided an integrated solution by merging materials science, microbiology, electrochemistry, and computer analytics expertise. Besides promoting scientific understanding, it provides technical suggestions for stakeholders with a vision of developing sustainable, scalable microbial fuel cell technology. The research facilitates scientific advancement and practical and significant progress in renewable energy technology.

This thesis presents novel contributions by demonstrating the effectiveness of advanced $CoMoO_4$/polymer anode composites and highlighting, for the first time, the superior performance of untreated stainless steel electrodes in practical soil microbial fuel cell environments. Furthermore, the thesis uniquely advances microbial fuel cell modeling through an innovative, multi-source machine learning framework, thereby overcoming significant barriers to predictive accuracy and real-world application posed by earlier research.

Looking ahead, several promising future directions are apparent. First, integrating these advanced microbial fuel cells into hybrid treatment systems—such as constructed wetlands or anaerobic digesters—could potentially enhance overall system performance and operational stability. Second, testing these optimized electrode materials with diverse wastewater types, including industrial effluents or heavily polluted wastewater streams, would broaden the applicability of this technology. Finally, exploring other advanced nanomaterials or composites with potentially enhanced electrochemical and biological properties could open new pathways toward more robust and economically viable microbial fuel cell systems, thus ensuring their real-world scalability and sustainability.

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