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Application of electrodeposition for Nickel Nanoparticle-Modified Electrodes in Biochemical Systems ☆,☆☆

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ABSTRACT

Microbial fuel cells (MFCs) have garnered significant attention from researchers as an innovative and environmentally friendly method for the treatment of urban and industrial wastewater. The type and material of the electrode are critical factors affecting the efficiency and energy production of this process. The electrodeposition method was employed to dope nickel (Ni) and modify the surface of graphite plates (GP) and carbon felt (CF). The maximum voltage, current density, and power generated by the MFC were evaluated under consistent temperature conditions. Field-emission scanning electron microscopy (FE-SEM) results confirmed successful Ni doping and adequate microorganism attachment to the Ni-deposited electrodes, leading to enhanced electron transfer and increased power generation. Additionally, the highest average voltage output was observed using modified bio-Ni@CF (468.0 mV) and bio-Ni@GP (422.0 mV) electrodes, compared to bare CF (382.0 mV) and GP (301.0 mV) electrodes. Monitoring the open-circuit voltage (OCV) data for four loadings of the MFC with different anodes indicated the suitable resistance and stability of the Ni film over time. Therefore, the electrodeposition method can be considered a suitable technique for modifying the anode electrodes using Ni in MFCs.

Specifications table

Subject area:	Environmental Science
More specific subject area:	Bioelectrochemical
Name of your method:	Electrodeposition
Name of your protocol:	Application of Electrodeposition for Nickel Nanoparticle-Modified Electrodes in Biochemical Systems
Reagents/tools:	Dual-chambered plexiglass homemade Microbial Fuel Cell (DC-MFC) reactor, Proton exchange membrane (Nafion 117, Sigma-Aldrich, USA), Carbon felt, Graphite plate, Nickel sulfate, Ammonium chloride, Boric acid (Merck Company)
Experimental design:	After the preparation of modified electrodes, we investigated the performance of bioanode electrodes modified with nickel nanoparticles in the generation of bioelectricity by microbial fuel cells (MFCs).
Trial registration:	–
Ethics:	–

☆ **Related research article:** Investigation of microbial fuel cell performance based on the nickel thin film modified electrodes.
☆☆ **For a published article:** Mahmoodzadeh F, Navidjouy N, Alizadeh S, Rahimnejad M. Investigation of microbial fuel cell performance based on the nickel thin film modified electrodes. Scientific Reports. 2023 Nov 25;13(1):20755. (https://doi.org/10.1038/s41598-023-48290-3).

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(continued on next page)

Value of the Protocol:	MFCs represent an innovative and environmentally sustainable technology for the concurrent treatment of wastewater and generation of bioelectricity. Modifying the physical and chemical properties of bare electrodes can enhance MFC efficiency by improving electron transfer and facilitating microbial attachment. The electrodeposition method can be utilized as a green, versatile, and effective approach for the resistant and stable deposition of nanoparticles on bio-anode electrodes. Furthermore, the fabrication of bio-Ni@CF modified electrodes via electroplating techniques can serve as a novel, efficient, and versatile bio-anode in various MFC systems, resulting in increased conductivity, improved electron transfer kinetics, enhanced growth and stabilization of microorganisms, higher power density, and superior wastewater treatment.

Background

Microbial fuel cells (MFCs) represent an innovative and environmentally friendly technology for the simultaneous treatment of wastewater and generation of bioelectricity. A dual-chamber MFC reactor consists of a cathode and an anode, separated by a proton exchange membrane [1]. In the anaerobic anodic chamber, electrogenic microbes serve as active biocatalysts for the oxidation of organic materials, resulting in the production of protons, electrons, and carbon dioxide (Eq. 1) [2]. Electrons and protons are transferred from the anodic chamber to the cathodic chamber via an external circuit and membrane, generating an electric current through electrochemical reactions with electron acceptors (Eq. 2) [2].

$$C_6H_{12}O_6+6H_2O \rightarrow 6CO_2+24H^++24e^- \quad (1)$$

$$H^++24e^-+6O_2 \rightarrow 12H_2O \quad (2)$$

Various parameters can impact the functionality and efficiency of MFCs and their power generation capabilities. These factors include the type of inoculum (biocatalyst), the concentration and types of substrates, fuel cell configuration, the nature of the proton exchange membrane, the internal and external resistance of the cell, ion concentration, and the nature and type of electrodes [3]. Researchers are focusing on the modification and development of new materials for anodes and cathodes as an effective strategy to enhance MFC performance [4]. Ideal electrode materials should exhibit properties such as high electronic conductivity, large surface area, environmental friendliness, suitable chemical and mechanical stability, and economic affordability [5]. Materials such as carbon fibers, carbon cloth, carbon paper, carbon brushes, carbon felt (CF), graphite, and graphene have been utilized as electrodes in MFCs [4]. Altering the physical and chemical characteristics of bare electrodes can significantly enhance the efficiency of MFCs by improving electron transfer and microbial attachment. Consequently, researchers have focused on the use of inexpensive and readily available catalysts with simple and eco-friendly modification techniques [6]. Anodic and cathodic electrodeposition [7–9], paired electrodeposition [10], electrochemically assisted self-assembly [11], electro-grafting [12] and electro-polymerization [13,14] are green, versatile, and eco-friendly procedures for fabricating and modifying electrodes with different electroactive thin films for targeted applications. Electroplating is a general procedure for preparing metallic thin layer films on inert substrates through the cathodic electrodeposition of dissolved or anodically released metal cations under a direct electric current density [15,16]. Among the types of nickel (Ni) electrodeposition, black Ni and hard Ni are prevalent. In this study, the hard Ni method was selected due to its tensile strength and increased resistance to abrasion and corrosion [15,16]. The aim of this study was to employ electroplating or cathodic electrodeposition as a simple, one-step, green, and in-situ method for modifying CF and graphite plates (GP) with a Ni thin film, resulting in low-cost, highly conductive, and highly resistant electrodes. Subsequently, the Ni-doped electrodes were post-modified with microorganisms to create novel and efficient bio-anode materials through the growth of biofilms. In this comprehensive and comparative study, various parameters such as maximum voltage, current density, power generation, and electrode stability were investigated using a dual-chamber microbial fuel cell equipped with bio-Ni@CF and bio-Ni@GP electrodes under constant temperature conditions for bioelectricity generation.

Description of protocol

Preparation of modified electrodes

The electroplating or cathodic electrodeposition method was employed to coat the surfaces of CF and graphite electrodes with a Ni metallic thin film. Initially, the electrodes were pretreated by soaking them in an acetone solution for 20 minutes, boiling in 0.1 M HCl for 15 minutes, and subsequently washing them with water to remove any oils and foreign particles from their surfaces. The electroplating process was conducted in an undivided homemade reactor, consisting of a metallic Ni plate as the anode and GP and CF as the cathode, with nickel sulfate ($NiSO_4{\cdot}6H_2O$) serving as the supporting electrolyte in an aqueous solution of boric acid (H_3BO_3) at room temperature (Table 1).

As mentioned in the introduction, electroplating is a general procedure for preparing metallic thin-layer films on inert substrates through the cathodic electrodeposition of dissolved or anodically released metal cations under a direct electric current density [15,16]. Among the types of Ni electrodeposition, black Ni and hard Ni are prevalent. In this study, the hard Ni method was selected due to its tensile strength and increased resistance to abrasion and corrosion [15,16]. By applying a suitable current density for a specific

Table 1
The concentration of electrodeposition elements.

Chemical Name	Formula	Bath Concentration (g)	Metric (g/L)
Nickel Sulfate	$NiSO_4{\cdot}6H_2O$	8.90	179.70
Ammonium Chloride	NH_4Cl	1.50	24.70
Boric Acid	H_3BO_3	1.50	29.96

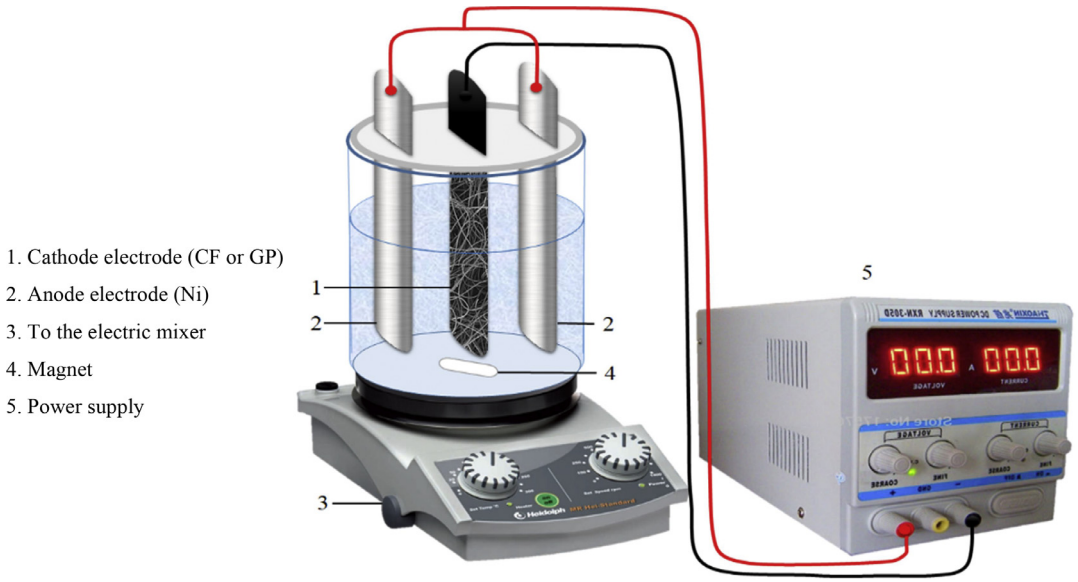

Fig. 1. Schematic representation of nickel electrodeposition process on electrode surfaces.

duration, the metallic anode (positive pole) oxidizes and releases the desired metallic cations ($Ni^{2+)}$ into the solution. Simultaneously, the dissolved metallic cations undergo cathodic electrodeposition ($Ni^{2+} + 2e^- \rightarrow Ni$) onto the GP and CF electrodes (negative pole) as Ni metal. The nucleation and growth mechanism of the Ni film in this study can be delineated in three steps:

1- Nucleation Step:
2- Island-like growth step
3- Intergrowth Step:

According to this mechanism, the nucleation step begins when the amount of Ni^{2+} ions near the cathodic surface reaches a critical concentration. The Ni-formed crystals can act as nucleation centres for the growth of the subsequent Ni crystals, as the islands-like growth. These conditions cause the connection and integration of the Ni islands and the formation of a dense and continuous Ni film. The Ni metallic ions can be penetrated from the created porosity in the film to the electrical doubled layer, for integration growth of the Ni film [17]. Finally, the Ni-coated GP and CF electrodes were removed from the solution and rinsed with water (Figs. 1–3) [18].

Microbial fuel cell set-up and operation

This study was conducted using a dual-chambered plexiglass homemade Microbial Fuel Cell (DC-MFC) reactor, featuring anodic and cathodic chambers, each with a volume of 350.0 mL (Fig. 4). The anodic and cathodic chambers were separated by a proton exchange membrane (Nafion 117, Sigma-Aldrich, USA) to facilitate the migration of H^+ ions from the anodic to the cathodic chamber.

CF was used as the cathode in all reactors, while the anodes consisted of bare GP, bare CF, nickel-coated carbon felt (Ni@CF), and nickel-coated graphite plate (Ni@GP), each with a surface area of 12.0 cm^2 (40.0 mm × 30.0 mm × 3.0). The MFC reactor operated in batch mode at a constant temperature of 30 ± 1°C and atmospheric pressure. The anodic chamber of the reactor was inoculated with active microorganisms sourced from the anaerobic sludge of the municipal sewage treatment plant of Urmia City and fed with glucose at a concentration of 5.0 g/L as the carbon substrate. Additionally, synthetic wastewater containing a mineral solution (g/L) composed of K_2HPO_4 (1.4), KH_2PO_4 (0.25), NH_4Cl (0.31), $MgSO_4$ (0.1), KCl (0.13), and NaCl (0.1) was used [4]. The anolyte was adjusted to a pH of 7.2 using a phosphate buffer to support microbial activity in the seed sludge. Prior to each run and the addition of inoculum, the anolyte was purged with N_2 gas for 5 minutes to create an anaerobic environment and remove any oxygen. Additionally, a continuous air pump was employed in the cathodic chamber to supply dissolved oxygen.

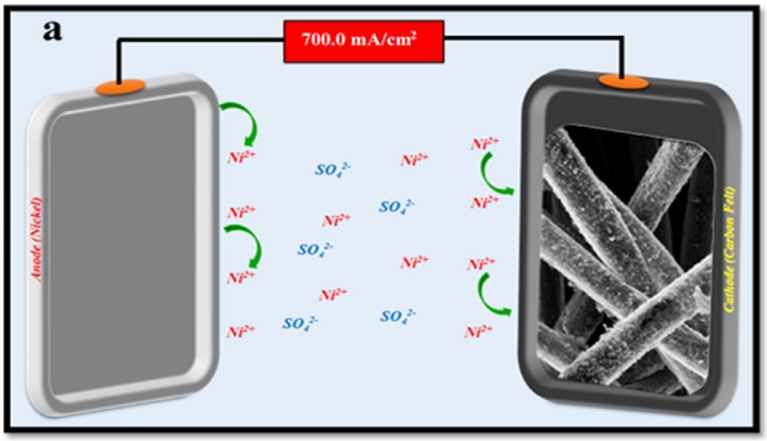

Fig. 2. Nickel film electrodeposition on carbon felt [18].

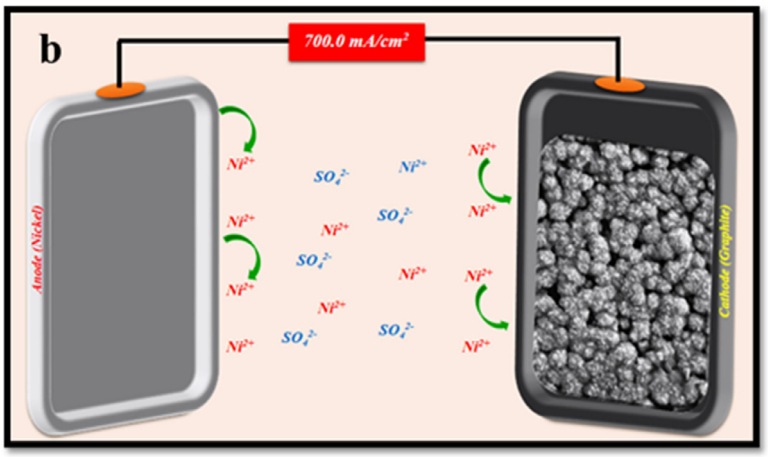

Fig. 3. Nickel film electrodeposition on graphite plate [18].

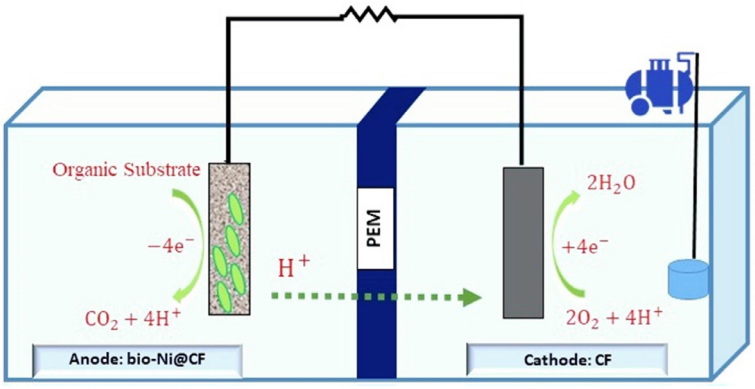

Fig. 4. Schematic representation of the dual-chamber Microbial Fuel Cell (MFC) reactor.

Protocol validation

Fig. 5 presents field-emission scanning electron microscopy (FE-SEM) images of both bare and Ni-coated CF and GP electrodes [18]. As observed in both the large and close-up views of the recorded images, Ni nanoparticles are continuously deposited onto the CF and GP electrode surfaces, in contrast to the bare electrodes. The morphological images reveal that the size and dimensions of the

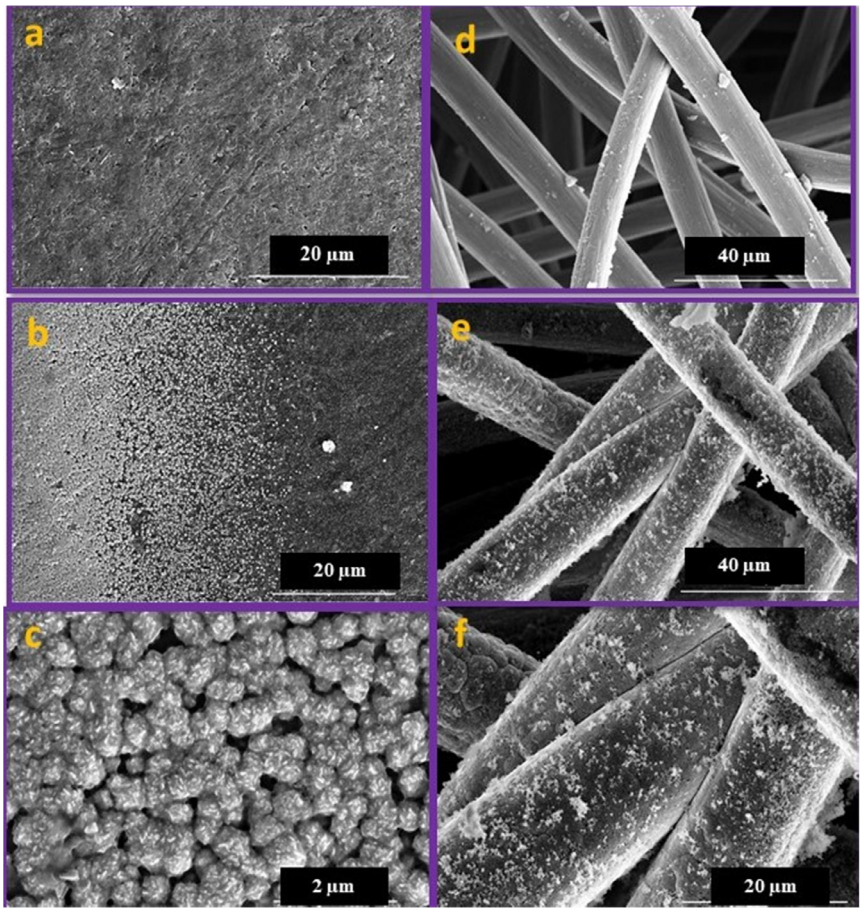

Fig. 5. FE-SEM images of (a-c) Ni@GP and (d–f) Ni@CF electrodes at different magnifications [18].

Ni nanoparticles on the CF are smaller than those on the GP. This disparity is attributed to the larger surface area of the stranded CF compared to the flat GP, given a constant applied current density for a fixed duration.

Bio-anode synthesis procedure

The nickel-coated carbon felt (Ni@CF) and nickel-coated graphite plate (Ni@GP) electrodes were immersed in a microorganism solution for one month through several loadings to facilitate the growth and stabilization of a biofilm on the Ni-coated electrodes, resulting in the fabrication of bio-Ni@CF and bio-Ni@GP electrodes.

Fig. 6 illustrates the presence of microorganisms as a biofilm on the Ni-coated GP and CF electrodes [18]. To supply the microbial communities and form a biofilm layer, mixed active microorganisms from anaerobic sludge were used. Recognized species included Klebsiella pneumoniae, Bacillus subtilis, Shewanella, Enterobacter, Geobacter, Pseudomonas, and Aeromonas. These electrogenic bacteria are capable of forming a biofilm layer and performing direct and mediated extracellular electron transfer (EET) [1]. A comparison of the images of the bio-Ni@CF and bio-Ni@GP electrodes, as shown in Fig. 6, reveals that the characteristics of the anode surface significantly impact the colonization of exoelectrogenic bacteria. CF exhibits a larger and more suitable surface area than GP for the penetration and attachment of microorganisms due to its high porosity [19]. Conversely, GP may exhibit poor microbial attachment due to its uniform and smooth surface [20].

Furthermore, the utilization of nanomaterials as catalysts with high biocompatibility can enhance electron transfer, reduce internal resistance, increase energy generation, and improve the efficiency of MFCs due to increased interaction between the electrode surface and microorganisms [21,22]. A study conducted by Zhong et al. demonstrated that modification of the CF surface affected the microbial community and increased biofilm formation, leading to an increase in voltage, current density, and power output [23]. Additionally, findings from a study by Ouis et al. confirmed that modification of the graphite electrode with conductive substances enhanced electron transfer and power density, which was attributed to the high adhesion of bacterial species on the anode and the formation of a more stable biofilm [24].

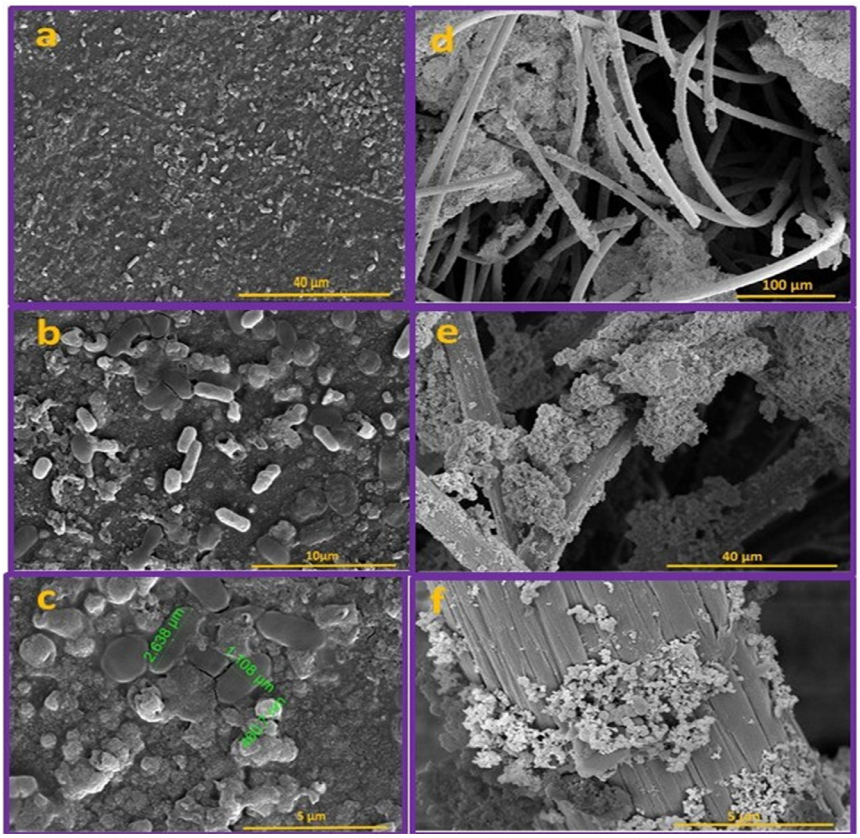

Fig. 6. FE-SEM images of (a-c) Bio-Ni@GP and (d–f) Bio-Ni@CF electrodes at different magnifications [18].

Electrochemical performance of MFC

According to the obtained OCV data, the highest average voltage output was observed using modified bio-Ni@CF (468.0 mV) and bio-Ni@GP (422.0 mV) electrodes, compared to bare CF (382.0 mV) and GP (301.0 mV) electrodes [18]. Polarization and power density curves were obtained for different electrodes after several loading steps and biofilm formation, as well as ensuring a steady state of MFC conditions in terms of voltage generation by applying variable resistances (0.01–100.0 kΩ). As depicted in Fig. 7b, the maximum power density and current density using the bio-Ni@CF electrode were 130.72 mW/m^2 and 760.0 mA/m^2, respectively. power density using bio-Ni@GP, bio-CF, and bio-GP reached 106.58, 55.04, and 27.01 mW/m^2, respectively [18].

Enhanced performance with nanomaterial-modified anodes

Modifying the anodic electrode with nanomaterials to enhance bacterial growth and strengthen microbial attachment significantly improves the performance and efficiency of MFCs. As illustrated in Fig. 7, the bio-Ni@GP and bio-Ni@CF electrodes exhibit the highest energy generation efficiency compared to the bare GP and CF electrodes. Ni acts as a catalyst, increasing conductivity and enhancing the electroactive properties of the electrodes.

The findings from this study, along with previous research, confirm that Ni can be used as a stable catalyst compatible with the conditions of microorganisms in the bio-anode. The stability of doped nanoparticles on the electrode is influenced by the doping method. Previous reports indicate that Ni was used as a catalyst in the cathode to modify activated carbon, but this led to reduced electrode conductivity and electrical performance due to the use of polytetrafluoroethylene (PTFE) [25].

In the current study, the direct and in-situ electroplating or cathodic electrodeposition process was identified as an effective method for achieving resistant and stable nanoparticle doping on the electrode. As depicted in Fig. 7a, the voltage output of four loadings using Ni@CF and Ni@GP electrodes remained stable in the bio-anode.

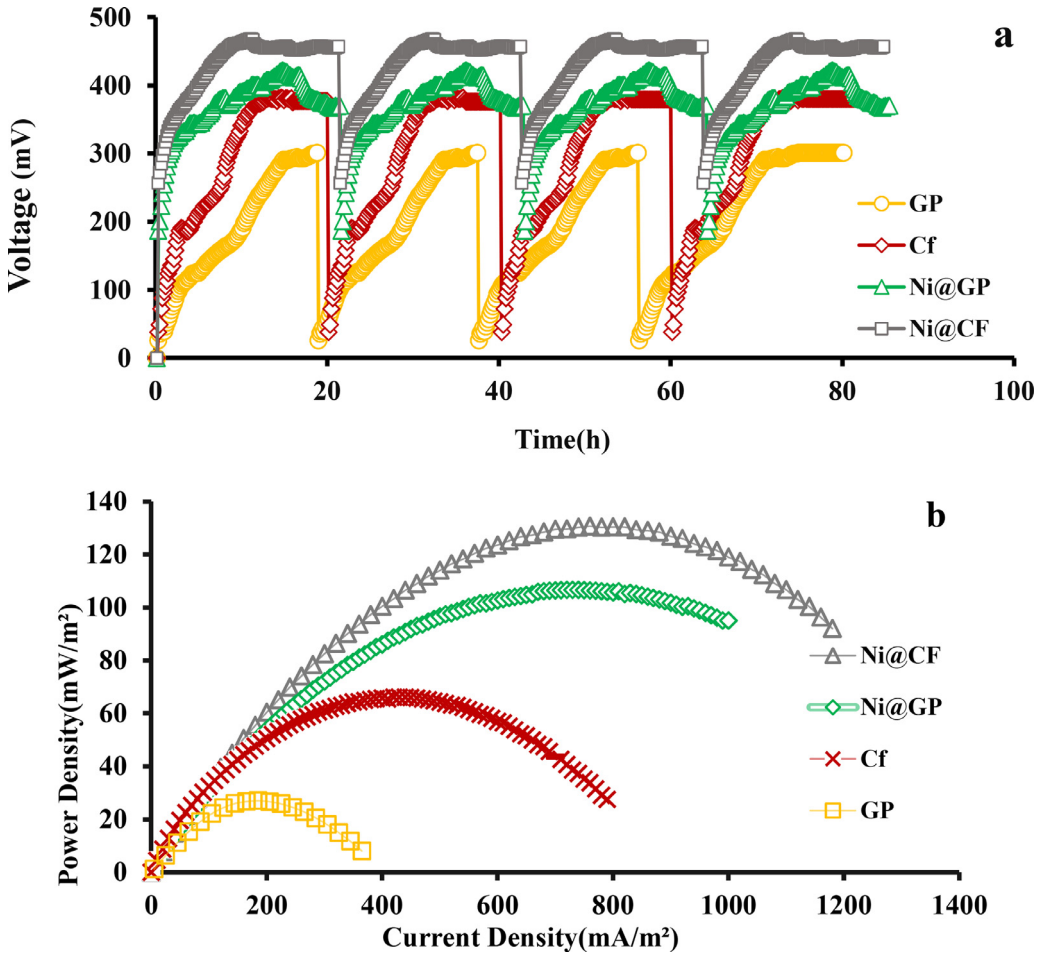

Fig. 7. Open Circuit Voltage (OCV) and (b) Power density as a function of current density [18].

Conclusion

This study aimed to employ electroplating or cathodic electrodeposition as a simple, one-step, green, and in-situ method for modifying carbon felt and graphite plates with a nickel thin film, resulting in low-cost, highly conductive, and highly resistant electrodes. Subsequently, the nickel-doped electrodes were post-modified with microorganisms to create novel and efficient bio-anode materials through the growth of biofilms. In the current study, the direct and in-situ electroplating or cathodic electrodeposition process was identified as an effective method for achieving resistant and stable nanoparticle doping on the electrode. This simple and eco-friendly technique can be employed in preparing various modified electrodes using different substrate electrodes and metallic thin films for various targeted analytes in microbial fuel cells. However, using concentrations of nickel nanoparticles that exceed the threshold limit may reduce the biocompatibility of the bio-anode surface. When the surfaces of the bio-anode electrodes become saturated, these nanoparticles may detach from the electrode surfaces and, due to the increased levels of toxic substances in the anode chamber, negatively affect the growth and activity of microorganisms and biofilm formation. Therefore, optimizing the catalyst concentration is essential as a key factor in enhancing the surface properties of electrodes.

Limitations

Environmental Conditions: The experiments were conducted under controlled laboratory conditions. maintaining optimal conditions for microbial activity such as temperature, pH can affect performance.

The accumulation of Particles and microbial biofilms on the membrane surface can reduce its effectiveness and increase resistanc. Thereforee selecting a membrane material that can resist fouling while ensuring efficient ion transport is essential.

CRediT author statement

The authors confirm contribution to the paper as follows:

Study conception and design: N.N., F.M., S.A., M.R.; Data collection: F.M., N.N.; Analysis and interpretation of results: F.M., N.N., S.A.; Draft manuscript preparation: F.M., N.N., S.A., M.R. All authors reviewed the results and approved the final version of the manuscript.

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

Data will be made available on request.

Acknowledgments

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