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AN EASY-TO-FABRICATE, SUBMERGED CARBON-BASED AIR CATHODE FOR BIOFUEL CELLS

ASIAH SUKRI[1], RAIHAN OTHMAN[1*], NORSHAHIDA SARIFUDDIN[2]

[1]Department of Science in Engineering, Kulliyyah of Engineering, International Islamic University Malaysia, Kuala Lumpur, Malaysia
[2]Department of Manufacturing and Materials Engineering, Kulliyyah of Engineering, International Islamic University Malaysia, Kuala Lumpur, Malaysia

**Corresponding author:* raihan@iium.edu.my

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ABSTRACT: An air cathode (AC) is one of the main components in an AC-based biofuel cell (BFC). Its cost accounts for nearly half of the total cost of the cell as specific requirements must be met for it to perform as an effective site for the oxygen (O_2) reduction reaction (ORR) to occur. In most applications where the AC is totally submerged in the electrolyte, air or O_2 is bubbled throughout the electrolyte during the entire discharge operation to enhance the cell performance. This is because the dissolved O_2 (DO) concentration is merely one-third of the O_2 concentration in ambient air. Unfortunately, this approach increases the overall complexity and cost of the system. Therefore, this present study developed an effective, easy-to-fabricate AC for use under totally submerged and unaerated conditions. The design principle of the proposed AC is a balance between the hydrophobicity and hydrophilicity of the components used, i.e., the combination of a carbon felt, an interwoven carbon fiber sheet, and a nickel mesh. All the cathode components were snugly fitted merely using the mechanical pressure of the cylindrical BFC holders. The fabricated AC was assembled in a zinc-air BFC employing fungal microbes *Phanerochaete chrysosporium* (*P. chrysosporium*). When tested at a constant current of 1.0 mA under unaerated, uncontrolled ambition conditions, the zinc-air BFC discharge lasted 42 days with an average operating voltage of 200 mV. Under these conditions and even without the inclusion of any catalytic material, the cell performance met the operating requirements of the low-powered remote sensing devices. Therefore, the proposed easy-to-fabricate submerged air electrode has demonstrated its viability for use in BFCs.

ABSTRAK: Katod udara (AC) merupakan salah satu komponen utama bagi sel fuel bio (BFC) berasaskan AC. Hampir separuh daripada keseluruhan kos sel berpunca dari katod udara (AC). Ini kerana beberapa keperluan khusus perlu dipenuhi bagi menyediakan tapak tindak balas penguraian (ORR) oksigen berlaku. Bagi kebanyakan aplikasi biasa di mana AC terendam sepenuhnya dalam larutan elektrolit, udara atau oksigen dialirkan secara berterusan sepanjang operasi discaj bertujuan menggandakan prestasi sel. Ini disebabkan kepekatan oksigen terlarut adalah hanya sepertiga daripada kepekatan oksigen di dalam udara sekitar. Malangnya, kaedah ini akan menambah kompleksiti dan kos. Oleh itu, kajian ini bertujuan membina AC yang efektif, bersifat mudah-pasang bagi aplikasi terendam sepenuhnya dan tanpa pengudaraan. Prinsip asas pada cadangan rekabentuk katod udara AC ini adalah bagi mengimbangi ciri hidrofobik dan hidrofilik komponen yang digunakan, iaitu kombinasi fabrik karbon, lapisan serat karbon terjalin dan jaringan nikel. Kesemua komponen katod ini terangkum kemas menggunakan tekanan mekanikal pada selinder pemegang BFC. AC ini kemudiannya diguna pakai di dalam BFC zink-udara yang terkandung mikrob kulat *Phanerochaete chrysosporium* (*P. chrysosporium*). Apabila diuji dengan arus tetap 1.0 mA dalam keadaan tanpa udara dan sekitaran tanpa kawalan, sel BFC zink-udara mampu bertahan selama 42 hari dengan purata voltan operasi sebanyak 200 mV. Dalam kondisi ini dan walau

tanpa sebarang unsur pemangkin, prestasi sel memenuhi keperluan operasi peranti penderiaan jauh bertenaga rendah. Oleh itu, katod udara yang dibangunkan bagi aplikasi elektrod terendam penuh dan bersifat mudah-pasang ini telah berhasil memenuhi keperluan bagi kegunaan BFC.

KEYWORDS: *Air-cathode biofuel cell; carbon-based air cathode; zinc-air biofuel cell; microbial fuel cell; Phanerochaete chrysosporium (P. chrysosporium)*

1. INTRODUCTION

Biofuel cells (BFC) are a potential source of green energy as they are clean, renewable, and can be derived from waste, such as municipal wastewater and agricultural waste [1,2]. However, the amount of energy discharged by BFCs is low and far from providing reasonably priced and energy-balanced operations. Multiple studies on increasing the energy balance of BFCs have yielded system designs that are manifold, complex, and costly. For instance, the capital cost of an air cathode (AC)-based BFC for treating municipal wastewater is estimated to be 30 times higher than that of existing activated-sludge systems [3].

The AC is an essential component in an AC-based BFC and accounts for nearly 45% of the overall capital cost of the entire system [4]. Carbon-based ACs, such as carbon felt, carbon cloth, carbon rods, and carbon fiber sheets, are commonly used as they have a large surface area and good electrochemical properties [5]. Catalysts are often used in an AC to increase oxygen (O_2) affinity and decrease the activation energy required for the O_2 reduction reaction (ORR) to occur, which, in turn, increases the amount of energy discharged by the AC [6]. Although platinum-based ACs enable BFCs to discharge energy at significantly higher densities [7], they are very expensive. Cobalt oxides and manganese oxides-based ACs are cheaper alternatives [8,9]. An effective AC also requires a stable triple interface, which is the electrolyte (liquid), the O_2 (gas), and the catalyst/current collector (solid) interface at which the ORR occurs. However, although this feature is unique to ACs, it is rarely discussed when reporting the performance of an AC. An AC's airside is usually porous and coated with a semipermeable, hydrophobic membrane, such as Teflon™, while the electrolyte side is denser and often dispersed with a catalyst. This ensures a good balance between hydrophobicity and hydrophilicity in an AC and, more crucially, prevents flooding, which occurs when an electrolyte accumulates in the porous airside of the AC and hinders the transportation of O_2 to the active sites of the catalyst or conductor [10,11]. Commercially available ACs are expensive due to the distinctive features mentioned above.

This present study designed and fabricated an effective and easily produced carbon-based AC for use in BFCs operating under submerged and unaerated conditions. The proposed AC comprised layers of CF and CFS fortified by a nickel mesh (NM) without the use of a catalyst. All the cathode components were cut into the desired sizes and snugly assembled using the mechanical pressure of the cylindrical holders in the BFC. The proposed AC was tested in a zinc-air microbial fuel cell (MFC) under a submerged condition [12]. Unlike an open-air AC, a submerged AC relies solely on the dissolved O_2 (DO) in the electrolyte. The DO concentration is one-third of the O_2 concentration in ambient air. Therefore, in most BFC applications with a submerged AC, the electrolyte is saturated with O_2 by bubbling air throughout the cell discharge operation. However, this approach decreases the existing low energy gain yields (output/cost) of BFCs.

2. MATERIALS AND METHODS

2.1. Design considerations and fabrication of the air cathode

Submerged aquatic plants rely on DO and dissolved inorganic carbon (DIC) for respiration and photosynthesis [13]. There are two major challenges concerning submerged aquatic plants as compared to leafy plants exposed to atmospheric air, namely (i) significantly lower DO and DIC concentrations and (ii) profound underwater gas exchange resistance [13]. Hydrophobic leaves are a special adaptation feature or mechanism for submerged aquatic plants. A thin gas film forms on the surface of these leaves due to the hydrophobic nature of its surface. This gas film reduces gas exchange resistance caused by the diffusion boundary layer (DBL) [14,15]. For a similar transport distance across a DBL and the same concentration gradient, the gas exchange under water is 104-fold slower than in air [13]. Due to the large water-gas interfaces between DBL and thin gas film, more O_2 can be collected inside the thin gas film before diffusing into the leaves' stomata [14].

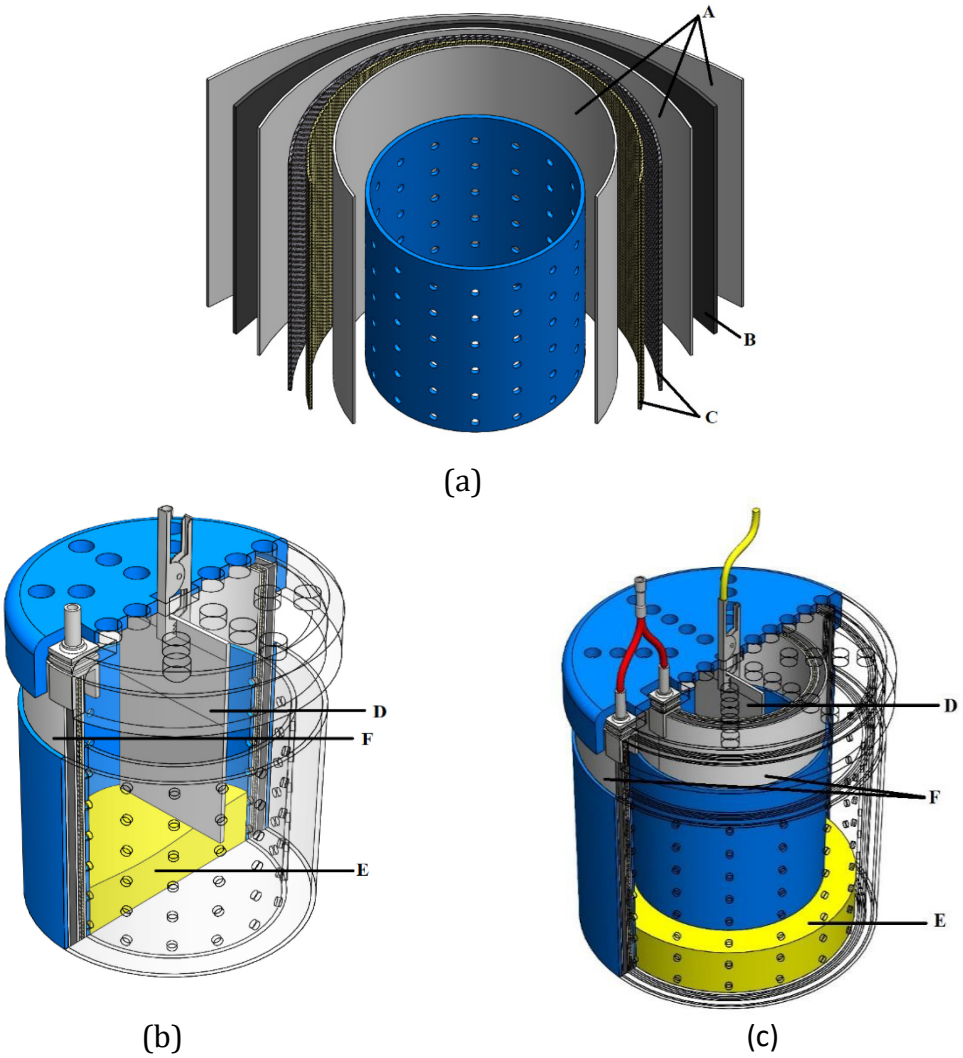

Fig. 1. Schematic illustrations of the (a) components of the AC, (b) the Zn-air MFC, and (c) the bipolar Zn-air MFC, which uses two ACs.

Legend: A – NM; B – CF; C – 2-ply CFS; D - Zn anode; E – Fungus-cultivated EFB; F – AC

Therefore, hydrophobicity must be considered when designing a totally submerged AC to ensure its effective functioning. The AC that this present study proposed comprises NM, CF, and CFS. The NM served as both structural support and the current collector for the external circuit, the sponge-like nature of the CF served as an air trap while its intrinsic hydrophobicity enabled the formation of a gas film, and the CFS provided electron carriers for the ORR to the external circuit as it is compact, conductive, and has a large surface area.

The holder of the AC comprised two polyvinyl chloride (PVC) tubes of different diameters (height = 45 mm, ϕ_1 = 58 mm, ϕ_2 = 50 mm) with a perforated inner insert. All the cathode components were cut into the desired sizes and wedged between the two cylindrical PVC tubes. This ensured that a uniform and uniaxial pressure was placed on the multiple layers of the AC, thereby ensuring that the exact same properties were maintained for each test run. An inner insert (ϕ_2) with a different diameter could be used to apply a different uniaxial pressure. Fig. 1a illustrates the overall arrangement of the cathode, which was CF and 2-ply carbon sheets inserted between sheets of NM. As seen in Fig. 1c, a bipolar configuration was later used to increase the surface area of the AC. For this configuration, two concentric cylindrical holders of different diameters were used. The performance of the proposed AC was benchmarked against that of commercially available E4/E4A EFL AC strips that had been cut into identical 3 cm by 3 cm pieces.

2.2. Characterisation of the air cathode structure

A JEOL® JSM-6700F field emission scanning electron microscope (FESEM; Tokyo, Japan) was used to observe the CF and CFS surface morphologies of the proposed AC and the E4 AC. ImageJ was used to analyze the hydrophobic contact angle of the CF.

2.3. Fabrication and the operating principles of the Zn-air MFC

The membrane-less, single-chamber cylindrical BFC was filled with 250 ml of 24 g/L unbuffered potato dextrose broth (PDB) electrolyte. The microbes were cultured from 5 g of dried *Phanerochaete chrysosporium* (*P. chrysosporium*) in an organic substrate of 2 g of oil palm-derived empty fruit bunches (EFBs). The fungus-cultivated EFBs resided at the bottom of the BFC enclosure. Further details may be obtained from [15]. A 40 mm by 100 mm Zn anode strip was placed in the middle of the MFC enclosure.

The ORR that occurs on an AC is biocatalytic. When an AC is supplied with lignin-rich EFBs, the *P. chrysosporium*-derived fungal microbes secrete ligninolytic enzymes that predominantly contain laccase, which has a specific affinity for O_2 molecules as its electron acceptor. Therefore, while the fungal microbes degrade the lignocellulosic wall, the laccase catalyzes the reduction of molecular O_2 [16].

$$O_2 + 4H^+ + 4e^- \xrightarrow[laccase]{} 2H_2O \quad (1)$$

As such, a bio-catalyzed electrochemical BFC can be produced by pairing a Zn anode with an AC in a medium rich with *P. chrysosporium* hyphae.

2.4. Electrochemical analysis

A Neware® BTS 4000 battery tester (Shenzhen, China) was used to test the power density and discharge capacity of the Zn-air MFC containing the proposed AC under a 1 mA constant current.

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3. RESULTS AND DISCUSSION

A galvanostatic discharge test has been used to evaluate the bioelectrochemical system's capacity to produce electricity utilizing the proposed submerged air electrode. It is a direct method to assess the system's ability to maintain the charge transfer rate between the electroactive species and the conducting electrode, as well as the stability of the charge transfer processes [12]. Fig. 2 shows the discharge capacity of the proposed AC at a constant current of 1 mA. The Zn-air MFC containing the proposed AC discharged an average operating voltage of 200 mV for 42 days. The total output energy estimated based on the area under the discharge plot was 211 mWh. The performance of the proposed AC was benchmarked against that of a commercially available E4 AC. The E4 AC was able to discharge an average operating voltage of 280 mV for 53 days for a total output energy of 403 mWh. Obviously, being incorporated with a manganese-based catalyst, the E4 air electrode could discharge two times more energy than the proposed non-catalytic AC.

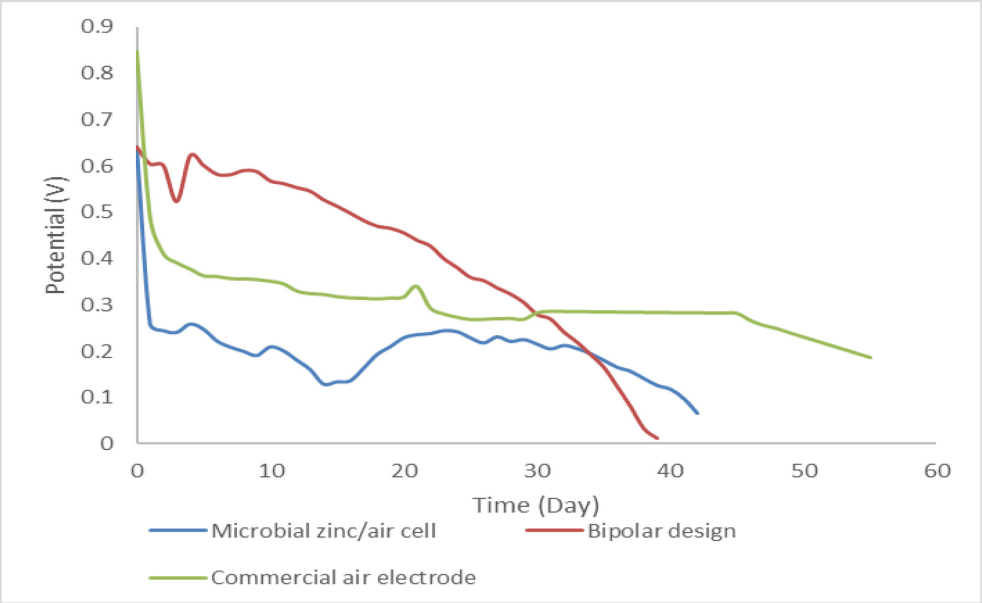

Fig. 2. An evaluation of the AC based on the Zn-air MFC discharge profile at 1 mA

Enhancing the AC surface area has been identified as one of the most critical factors to increase the volumetric power density of an AC-based MFC [17]. It was reported that doubling the AC surface could increase the output by 62%. However, this option is very costly and infeasible for a catalyst-based AC. A bipolar configuration was then used to increase the proposed non-catalytic AC's surface area, significantly improving its discharge profile (Fig. 2). More specifically, increasing the surface area of the proposed AC by 2.5 times enabled it to discharge more energy than the E4 AC for the first 31 days. Furthermore, although the enhanced proposed AC stopped discharging energy on Day 40, its total energy output was 396 mWh, which was only 1.7% lower than that of the E4 AC.

The proposed AC was tested under the following conditions: (i) totally submerged in an unaerated electrolyte so as to solely rely on DO, which constitutes one-third of the O_2 concentration in ambient air [18]; (ii) solely rely on the biocatalyst secreted by the freely suspended fungal *P. chrysosporium*, while the E4 AC was incorporated with a manganese catalyst, and (iii) discharged at a higher amount of current (1 mA) as compared to the commonly reported discharge load of 0.6 to 0.7 mA [12]. In the abovementioned test conditions, the Zn-air MFC containing the proposed submerged, non-catalytic AC

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continuously discharged 1 mA of current for over a month under unaerated and uncontrolled ambient surroundings. These conditions are essential for a bio-electrochemical cell to serve as a stand-alone energy source for low-powered remote sensing devices. First, an open-air AC would induce electrolyte evaporation from the cell as air access must be allowed, whereas for a submerged air electrode, it solely depends on DO, and only a tiny hole is sufficient to maintain the air/electrolyte gas exchange. Second, the cell must be able to operate in a self-sustaining mode for an extended duration without the input of external systems/energy, such as electrolyte aeration or intermittent substrate replenishment [17]. Third, a typical low-powered sensor requires about 2 mA at start-up and hundreds of μA in steady state [19]. Fig. 3 shows the power density profile of the Zn-air MFC containing the proposed submerged AC. The cell registered a maximum power output (1.17 mW) at 4.2 mA, which is way beyond the operating requirements of the low-powered sensors. Fig. 3 also compares the power density profile of the commercial AC. The maximum power density was 1.61 mW at a discharge current of 2.7 mA, which is much higher than the proposed submerged AC. However, the proposed submerged AC possessed a much higher limiting current density.

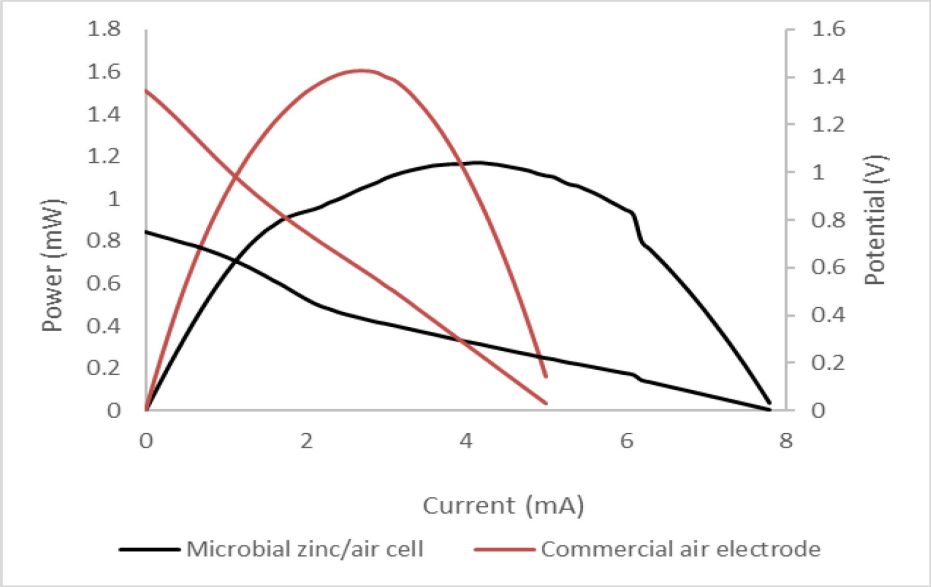

Fig. 3. A comparison of the power and polarization profiles of the Zn-air MFC using the proposed submerged AC and a commercial AC.

Among the notable demonstrations of self-sustaining MFC employing the AC is reported by Khaled et al. [19]. In battery mode (i.e., a single supply of sodium acetate substrate, without replenishment), the MFC power stack could operate a low-powered temperature sensor for 20 days. The power stack comprised two MFCs and was incorporated with a power management unit (PMU). The PMU comprised a DC/DC converter, capacitors to filter MFC output voltage fluctuations, and a mechanical switch. Each MFC alternately powered the sensor in a 48-hour cycle. The MFC comprised a single chamber with 700 ml capacity, inoculated with industrial wastewater and fed with 0.7 g of sodium acetate. The anode was a stainless-steel mesh with a projected area of 100 cm^2. The AC was a 120 cm^2 carbon cloth (30% Teflon treated, 0.5 mg/cm^2 platinum (Pt) catalyst loading). The biofilm on the AC was developed separately. With a total Pt catalyst loading of 60 mg, the MFC registered a maximum power point of about 1.1 mW/m^2 at around 3.5 mA and 0.9 V. Conversely, the Zn-air MFC employing the non-catalytic AC produced a maximum power point of 50 mW/m^2 at 4.2 mA and 0.3 V. The immediate consequence of a non-catalytic AC is the huge potential drop due to the high activation energy, which, however, is compensated by its ability to sustain a higher current.

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An AC generally comprises a conductive base and a gas diffusion layer (GDL). If a catalyst is required, it is incorporated between these two components. The conductive base supplies the electrons required for the ORR to occur from the external circuit or anode, while the GDL is where O^2 diffusion occurs between the electrolyte and the active sites [20,21]. As such, GDLs are designed to be hydrophobic to prevent cathode flooding [20]. The GDL is usually called the "airside," while the conductive base is called the "waterside." The interface between the GDL and the conductive base is a triple interface, i.e., the electrolyte (liquid), O_2 (air), and the catalyst/current collector (solid), at which the ORR occurs. Therefore, it is crucial to stabilize the triple interface to maintain the efficacy and functions of an AC. Most metal-air cells contain an open-air AC, where one side is exposed to air. However, in a seawater aluminum/air cell, the AC is fully submerged in the liquid electrolyte. In such cases, cell operation solely depends on the DO in the electrolyte, which is available at significantly lower concentrations than the amount of O_2 in ambient air [18]. A hydrophobic GDL now performs another important function of facilitating the exchange of O_2 between the cathode and the electrolyte at the gas-liquid interface [22,13]. Air cathode (AC)-based BFCs often use submerged ACs [23,24]. Although O_2 is bubbled throughout the operation of most cell configurations to increase the amount of DO available, a well-designed AC could further improve cell performance.

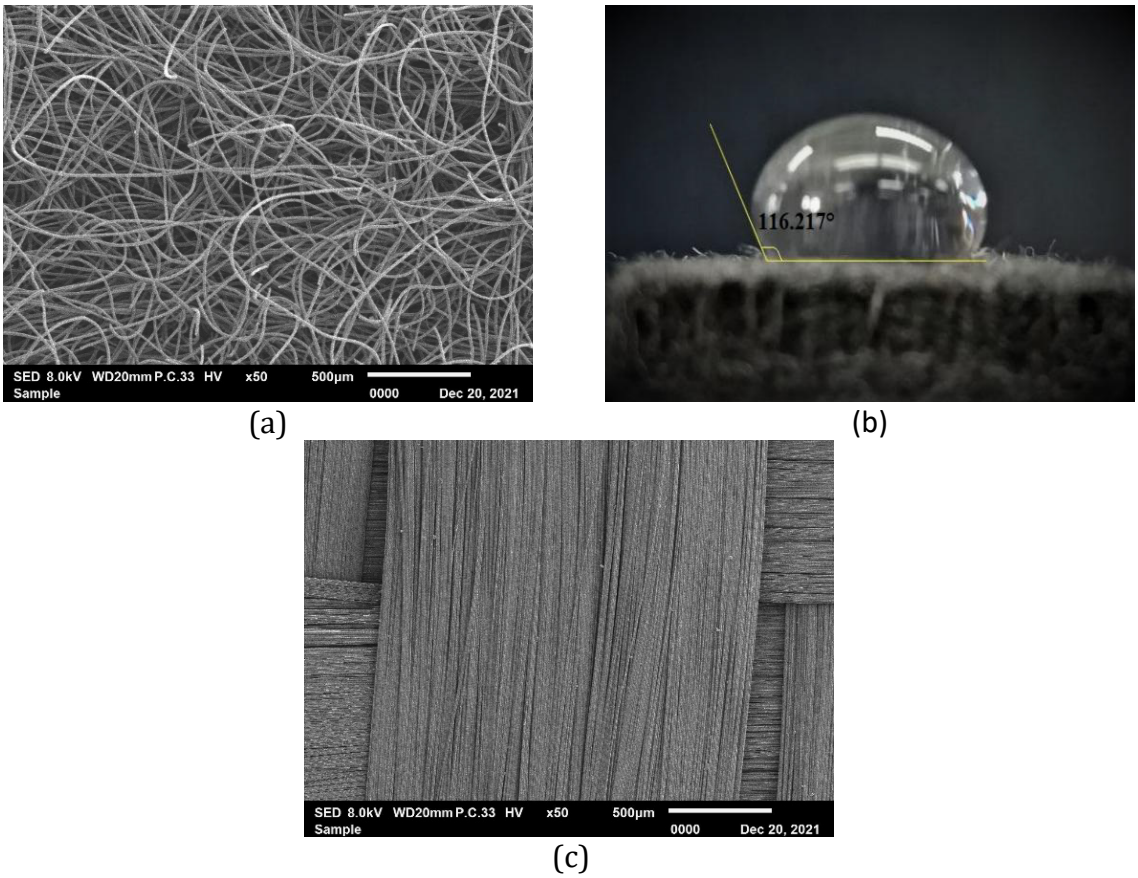

Fig. 4. (a) An SEM image of the carbon fiber depicting extended CF forming the spongy and porous matrix, (b) a digital image depicting the hydrophobic nature of CF; and (c) an SEM image of the CFS depicting the interwound bundles of carbon fiber forming a compact structure.

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The proposed AC functioned effectively and supported the design estimations. The SEM results indicated that CF contains loose and long carbon fibers that form a porous and spongy texture (Fig. 4a), which enables it to entrap air. Fig. 4b depicts the hydrophobic nature of CF, where an H_2O droplet is placed on it. Therefore, when the CF is fully submerged in the electrolyte, its hydrophobicity should enhance O_2 exchange with the electrolyte. Carbon fiber sheets (CFS) were used as a conductive base to supply electrons from the external circuit for the ORR to occur. The SEM results indicated that CFS contain interwoven threads of carbon fiber bundles (Fig. 4c). Carbon fiber sheets (CFS) are widely used as a base in cathodes because they are highly conductive and have good chemical and electrochemical stability. Meanwhile, NM provides structural integrity in an AC and acts as a current collector.

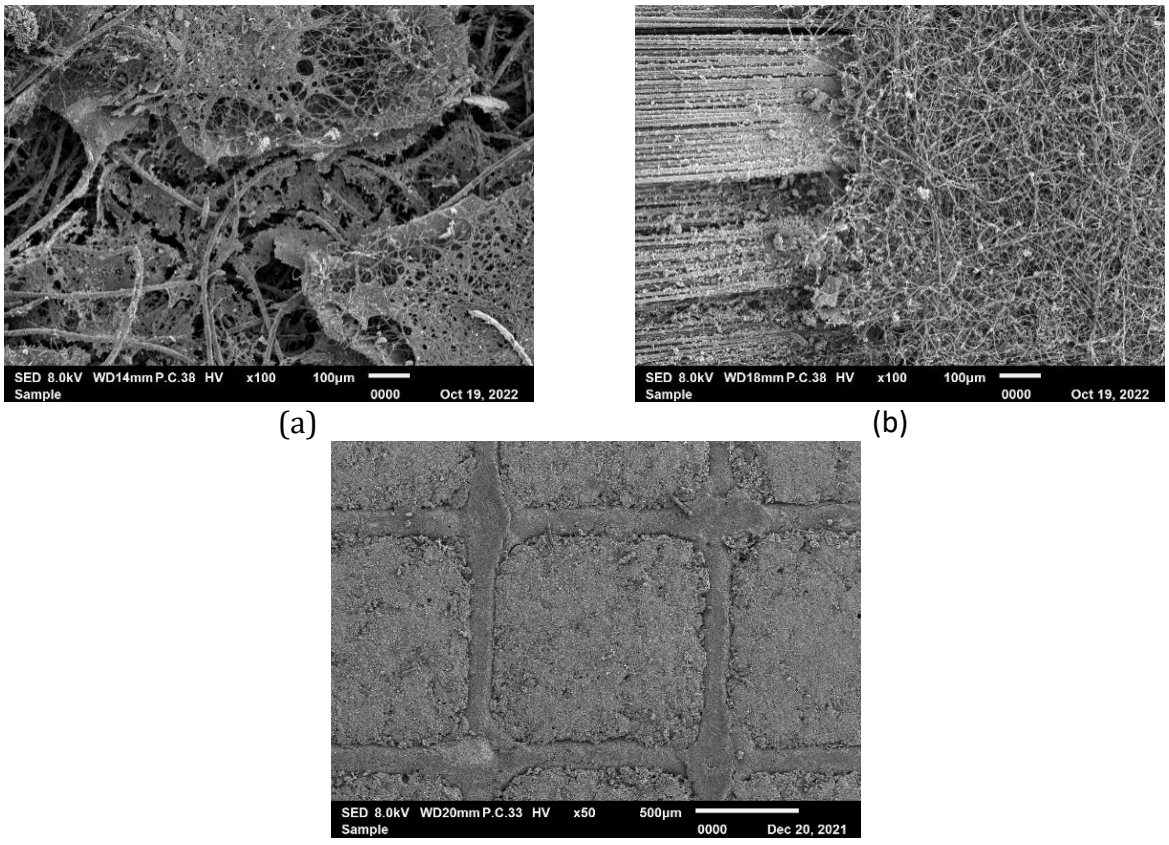

Fig. 5. (a) An SEM image depicting fungal colony growth on the CF, (b) an SEM image depicting the dense mycelia colony of the *P. chrysosporium* on the CFS, (c) an SEM image of E4 AC depicting a compact structure of the waterside with no trace of fungal growth.

When benchmarked against an E4 AC, the discharge duration and capacity of the Zn-air MFC containing the proposed AC were shorter and 48% less than that of the E4 AC. The E4 AC possesses two advantages: first, it is dispersed with an Mn-based catalyst within the AC structure. On the other hand, the proposed AC entirely relied on the freely suspended biocatalyst excreted by the fungal microbes. Second, the electrolyte side of the E4 electrode is dense and prevents the penetration of fungal mycelia colonies. When the proposed AC was configured bipolarly, which is a stack design, it discharged more energy than the E4 AC for the first 31 days. Its discharge duration, however, was still comparable to that of a single AC configuration. Scanning electron microscopy (SEM) was used to examine the proposed

enhanced AC once it had ceased operation. As seen in Fig 5a and 5b, dense *P. chrysosporium* mycelia colonies had formed on the surfaces of both the CF and CFS of the proposed enhanced AC. Conversely, as the surfaces of the carbon components of the E4 AC are dense, they prevented the penetration and proliferation of fungal mycelia colonies (Fig. 5c). The airside of an E4 AC strip is encased in a Teflon™ membrane, which has comparable hydrophobicity to CF. It is possible that the proliferation of fungal mycelia colonies between the CF and CFS layers of the proposed enhanced AC prevented it from functioning effectively and sustaining the desired current output. Therefore, the CFS, or waterside, of BFCs that utilize fungal microbes must be protected or coated with a dense layer to prolong its service life. It is also noteworthy that, although the proposed AC does not contain any catalytic materials, its discharge performance could be tailored simply by increasing its surface area.

4. CONCLUSION

This present study demonstrated a simple method of fabricating an effective, submerged carbon-based AC for use in BFCs. Commercially available CF and CFS were, respectively, chosen as the GDL and conductive base of the proposed AC based on their as-purchased properties and used without any modifications. Therefore, the O_2-reducing performance and efficacy of the proposed AC could be further enhanced by pre-treating the CF and CFS layers using multiple methods. The proposed AC was tested as a fully submerged AC in a Zn-air MFC and found to be able to discharge a comparatively high constant current over a one-month period. Therefore, the proposed AC is viable and effective for use under submerged conditions, whether for non-air-circulated or air-circulated BFCs.

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