

Advancements in Cotton Fabric-Based Air Cathode Electrodes for Membraneless Alkaline Fuel Cell Prototypes

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Abstract

This study examined the development of air cathode electrodes using cotton fabric. Various compositions of PTFE (10, 15, 20 and 25 wt%) were mixed with Nafion and carbon powder to create cotton fabric electrodes that enhance air diffusion into the cathode. The initial analysis focused on the surface properties of the cotton fabric and the assessment of carbon within the fuel cell. The physical characteristics of the cathode were evaluated before the measurements, and the leakage-seepage behavior was examined using a 0.1 M KOH solution. The results revealed that the cotton fabric surface was smooth, which facilitated the adhesion of the ingredients when treated with Nafion, carbon powder and PTFE. Scanning transmission electron microscopy (STEM) revealed spherical catalyst shapes with clustering on the surface. No seepage or leakage of the KOH solution was observed with 20 wt% PTFE. Oxygen meter tests conducted with this composition displayed a high oxygen output of 98.0 %, indicating efficiency. A fuel cell constructed with carbon powder, 20 wt% PTFE and Nafion as the air cathode demonstrated optimal performance, similar to commercial air cathodes. Evaluation of the efficiency using the Pd/C catalyst anode and AgMnO₂/C catalyst revealed a power output comparable to commercial air cathodes.

Keywords: Fuel cells, Alkaline fuel cells, Air cathode, Carbon cloth, Cathode, Membraneless fuel cells, Cotton fabric

Introduction

In the past, fuel cells utilized metal electrodes and an alkaline electrolyte solution. An insulating diaphragm soaked in electrolyte was positioned near the electrodes to prevent the passage of air and hydrogen while allowing for the reaction of fuel and air supplies. Gas-permeable electrodes were developed to enable the reactants to reach the active site of the catalyst and prevent electrolyte solutions from entering the gas routes, resulting in the invention of gas diffusion electrodes [1].

Modern fuel cells typically consist of an air cathode comprising a catalyst layer, a gas diffusion layer

(GDL) and a collector/support layer. Carbon-based compounds such as activated carbon (AC) are preferred over platinum due to their cost-effectiveness and high efficiency [2-5]. Carbon provides mechanical support and reduces electrode resistance during electron transmission [2-6]. However, carbon-based materials can deteriorate over time, leading to decreased performance [7,8]. Carbon fabrics possess corrosion resistance and electrical conductivity, but additional support layers may be required when scaling up microbial fuel cells (MFCs). The current collector is usually integrated with the GDL and serves as an oxygen

diffuser and water barrier. Polymers such as polytetrafluoroethylene (PTFE), polydimethylsiloxane (PDMS) and polyvinylidene fluoride (PVDF) are employed in the production of the cathode GDL [9-13].

Cheng *et al.* [9] investigated the implementation of multiple layers of PTFE on a carbon/PTFE base layer on the air side of the cathode in single-chamber MFCs. This resulted in significant enhancements in coulombic efficiency (CE), maximum power density and a reduction in water loss through the cathode. Zhang *et al.* [10] developed a novel technique for fabricating cathodes in MFCs using metal mesh current collectors and affordable polymer/carbon diffusion layers (DLs). Instead of adding a current collector to a carbon cloth cathode material, a cathode is formed around the metal mesh, eliminating the need for carbon cloth or other supporting materials. Yang *et al.* [11] proposed a cost-effective approach to constructing MFC cathodes through a one-step phase inversion process involving a PVDF binder and activated carbon catalyst. Zhuang *et al.* [12] examined different membrane cathode assemblies (MCAs) and cloth-cathode assemblies (CCAs) in air-chamber MFCs and identified the optimal cathode configuration for scaling up MFCs.

Recent breakthroughs have significantly improved the efficiency and affordability of MFCs for wastewater treatment. The introduction of an activated carbon (AC) air cathode (ACAC) has resulted in increased power output. Rolling carbon powders with PTFE to form catalyst layers (CLs) improved the electron transfer number (n) of the oxygen reduction reaction (ORR) [9,13]. In contrast, brushing with Nafion as the catalyst layer (CL) decreased the n value of Pt/C. These polymers require high-temperature curing processes [9-13]. PTFE emulsions are commonly employed for cathode GDL production and are often coated over a support/current collector layer such as a current collector (CC) or stainless steel mesh (SSM). Multiple layers of PTFE are then formed by heating to 370 °C [5,9]. In some cases, a mixture of carbon black (CB) and

PTFE is extruded onto SSM using a rolling technique to streamline cathode construction. The air cathode has been effectively cultivated on various support materials, such as graphite fiber brush (GFB), carbon cloth, carbon nanotube and stainless steel mesh, to attain impressive performance [14-16]. The air cathode offers excellent flexibility for combining with other technologies, like constructed wetlands and algae, to effectively treat wastewater with minimal chemical oxygen demand (COD) and refine the resulting effluent [17]. Activated carbon-carbon black and activated carbon-heat maintained a consistent power generation of 960 - 970 mW/m², while the activated carbon alone produced only 860 mW/m². Combining metal-organic framework with activated carbon has emerged as the most effective method for enhancing power generation, yielding the highest peak power density reported for air-cathode microbial fuel cell (MFC) at around 4,200 - 4,700 mW/m² [18,19].

Currently, fuel cell utilization faces several significant challenges. The cost of fuel cell technology presents difficulties in terms of growth and market expansion. The oxygen reduction reaction can be slow, but using an air cathode made from cotton fabric with activated carbon, polytetrafluoroethylene and with or without Nafion can help address this issue. This research focused on the power generation of the air cathode constructed using PTFE and Nafion. It showed that this power output closely resembled the performance achieved with an air cathode supplied by a Taiwanese company.

Materials and methods

Preparation of carbon cotton fabric for air cathode electrodes

Preparation of carbon cotton fabric air cathode electrodes without Nafion

To prepare air cathode electrodes from cotton fabric, 10, 15, 20 and 25 wt% polytetrafluoroethylene (PTFE) (Merck company) without Nafion (Merck

company) was added. In a mixing container, 0.075 g of Vulcan XC-72R carbon was combined with the desired amount of PTFE (10, 15, 20 or 25 wt%). Five drops of deionized water are added to the carbon and PTFE mixture along with 5 drops of ethanol. The ingredients

are mixed thoroughly until the mixture becomes viscous and spreads evenly on the cotton cloth. The coated cotton cloth was maintained at room temperature for the drying process as shown in **Figure 1**.



Figure 1 Preparation of carbon cotton fabric air cathode electrodes without Nafion.

Preparation of carbon cotton fabric air cathode electrodes with Nafion

Carbon fabric from cotton fabric was mixed with different percentages of polytetrafluoroethylene (PTFE), specifically 10, 15, 20 and 25 wt%. The process followed the same steps as those described in

preparation of carbon cotton fabric air cathode electrodes without Nafion section. Nafion solution was applied to the synthetic cotton fabric after the PTFE mixture was evenly spread on the cotton cloth. The prepared electrode was then left at room temperature for further processing as shown in **Figure 2**.



Figure 2 Preparation of carbon cotton fabric air cathode electrodes with Nafion.

Oxygen inlet and outlet measurements

In this test, the oxygen concentration at both the inlet and outlet was assessed. The measurement can accurately determine the oxygen content at room

temperature, typically 20.1 %, as shown in **Figure 3**. Additionally, it can effectively measure oxygen levels ranging from 0 to 100 % of the total oxygen in the air at both the inlet and outlet.

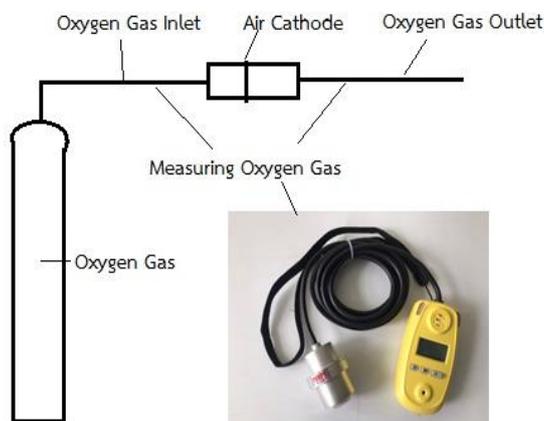


Figure 3 Oxygen inlet and outlet measurement device.

Catalyst synthesis

Synthesis of 20 wt% Pd/C as the anode

The process began by dissolving 0.33 g of palladium (II) chloride (PdCl_2) (99.9 %, Merck company) in 50 mL of deionized water. The solution was continuously stirred with magnetic stirrers for 2 h. The pH of the mixture was adjusted to within the range of 9 - 11 using a 2 M NaOH solution. In this solution, 1.50 g of sodium borohydride (NaBH_4) (98.0 %, Merck company) was introduced. After constant stirring for 30 min, the resulting sediment was collected. This sediment was then subjected to a series of rinses with deionized water and dried at 80 °C for 24 h. This meticulous process yielded the (20 wt%) Pd/C catalyst.

The next step involved combining the Pd/C catalyst with 0.8 g of Vulcan XC-72R carbon (Cabot company) and 50 mL of deionized water. This mixture was continuously stirred with magnetic stirrers for 2 h and then subjected to filtration. After filtration, the sample was washed thoroughly with deionized water and dried at 80 °C for an additional 24 h.

Synthesis of 20 wt% AgMnO₂/C as the cathode

The synthesis of silver manganese oxide on the carbon catalyst was achieved by dissolving 0.15 g of silver nitrate (AgNO_3) (99.0 %, Merck company) and 0.1 g of manganese(II) oxide (MnO_2) (99.0 %, Merck company) in 50 mL of deionized water, followed by stirring the mixture using magnetic stirrers for 2 h. After adjusting the pH of solution to within the range of 9 - 11 with a 2 M NaOH solution, 1.5 g of sodium borohydride (NaBH_4) and 0.8 g of Vulcan XC-72R carbon were mixed and stirred using magnetic stirrers for 2 h. The

resulting precipitate was filtered, subjected to multiple rinses with deionized water and finally dried at 80 °C for 24 h, to form silver manganese (II) oxide on the carbon catalyst.

To incorporate 0.8 g of Vulcan XC-72R carbon into the catalyst, it was added to the prepared solution along with 50 mL of deionized water. The mixture was stirred with magnetic stirrers for 2 h. Next, the resulting precipitate was filtered and subjected to multiple rinses with deionized water before being dried at 80 °C for 24 h.

Physical characterization of the catalyst

The morphology of the catalyst was analyzed using an FEI Quanta 450 FEG model with a field emission scanning electron microscope (FE-SEM) and scanning transmission electron microscope (STEM). The elemental composition of the catalyst samples was assessed with an Oxford Instruments X-Max 50 energy dispersive X-ray spectrometer (EDS), while the X-ray diffraction patterns were obtained using a D8 advance machine from Bruker in Germany. The patterns were obtained using CuK α radiation with a wavelength of 1.5406 Å while operating at 40 kV and 30 mA.

Efficiency measurement of the community alcohol membraneless alkaline fuel cell

To create the community alcohol fuel solution, 0.1 M was mixed with a 0.1 M potassium hydroxide solution and dissolved. Then, the fuel is delivered to the anode at a steady rate of 0.5 mL/min while air is simultaneously fed into the cathode side fuel cell at a rate of 4 mL/min.

Results and discussion

Analyzing the surface characteristics of carbon cotton fabric using scanning electron microscopy before assembly into a membraneless alkaline fuel cell prototype

Physical Characteristics: The carbon fabric derived from cotton with 20 wt% PTFE without Nafion has a smooth appearance with particles evenly adhering to the surface. Conversely, the carbon cotton fabric with 20 wt% PTFE and Nafion had a smooth, glossy surface, indicating the strong adhesion of carbon particles to the fabric. The addition of Nafion significantly improved the adhesion of carbon on the fabric surface and

enhanced electrochemical efficiency. Scanning electron microscopy (SEM) was used to study the surface properties of the carbon cotton fabric before measurements in the fuel cell and to observe the enlargement of the carbon cotton fabric surface after the application of Nafion. The Nafion-coated carbon layer exhibited greater expansion than the carbon fabric from cotton with 20 wt% PTFE without Nafion, as shown in **Figure 4**. Nafion occupies the pores and gaps between carbon particles, resulting in an overall volume increase. It forms a thin coating around the carbon particles, effectively enlarging their size.

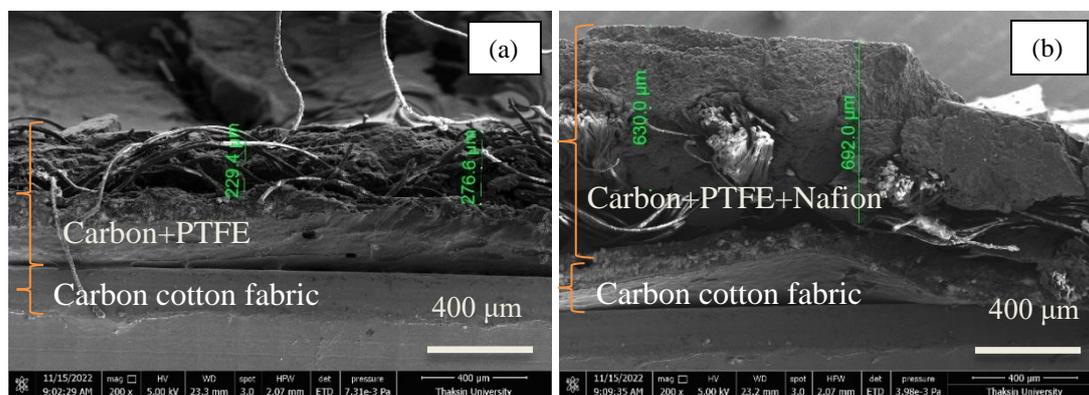


Figure 4 Scanning electron microscopy image of the carbon cotton fabric surface before measurement in a membraneless alkaline fuel cell prototype. The carbon cotton fabric with (a) 20 wt% PTFE without Nafion and (b) the carbon cotton fabric with 20 wt% PTFE with Nafion.

Analysis of the surface characteristics of the catalyst in a membraneless alkaline fuel cell prototype

The surface characteristics of the catalyst were investigated using a scanning transmission electron microscope (STEM). The catalyst was found to have a round shape and was grouped together on the surface. **Figures 5** and **6** display the unique surface characteristics of each elemental catalyst.

Figure 5 shows that the AgMnO_2/C catalyst has a granular morphology with dense clusters on the carbon surface, as shown in **Figures 5(b) - 5(d)**. Energy dispersive X-ray spectroscopy (EDS) analysis of these

small particles revealed that they were composed of 93.54 wt% carbon, 0.67 wt% silver and 0.89 wt% manganese, as shown in **Figure 5(a)**. The AgMnO_2/C catalyst showed an average diameter of 2.74 μm.

In **Figures 6(b)** and **6(c)**, the Pd/C catalyst is characterized by small lumps tightly clustered on the carbon surface, accompanied by small particles. Elemental dispersion spectroscopy (EDS) analysis of the composition of this catalyst revealed that it consisted of 83.46 wt% carbon and 5.15 wt% palladium (**Figure 6(a)**). The Pd/C catalyst showed an average diameter of 3.13 μm.

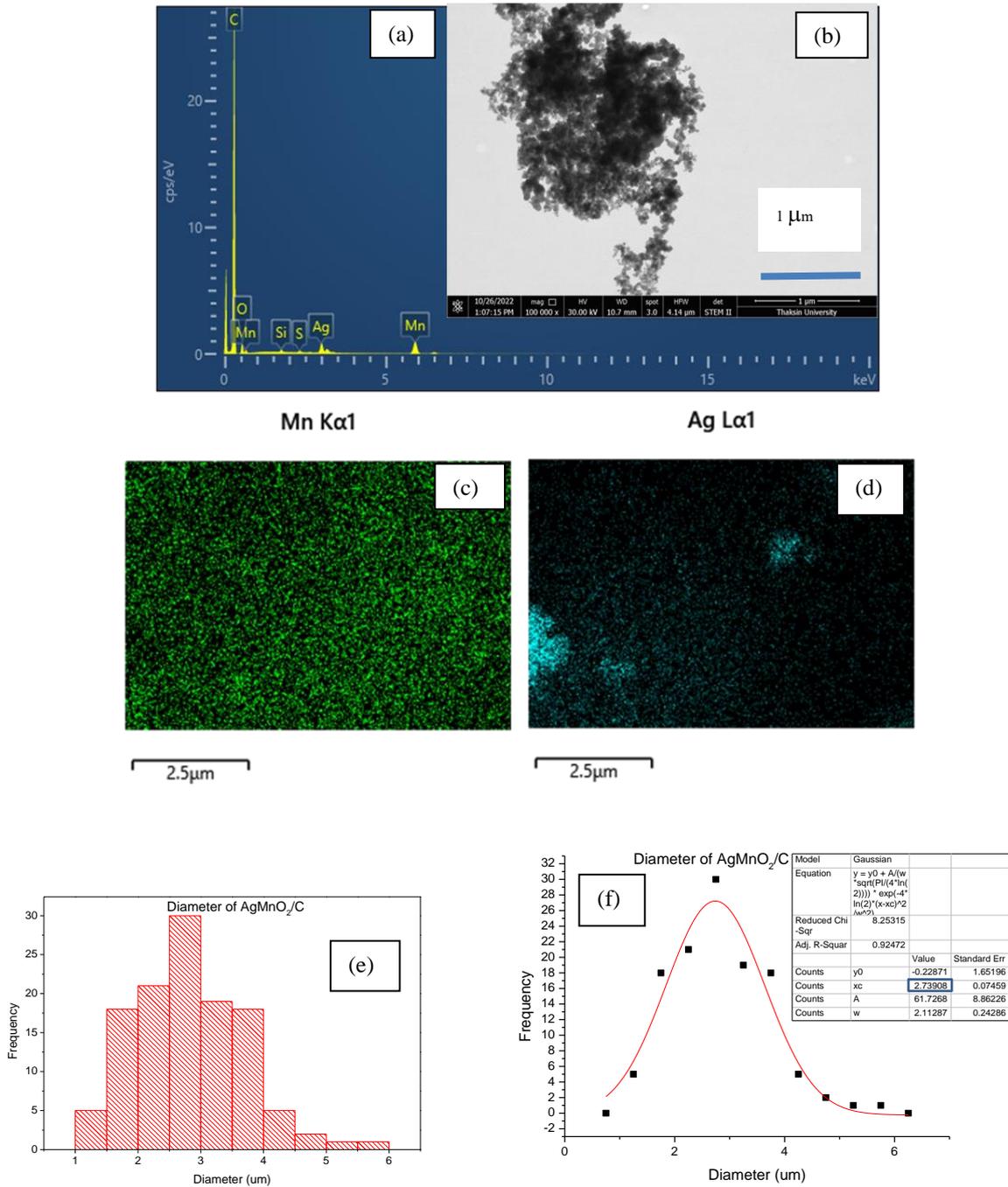


Figure 5 Physical characteristics and elemental composition of the AgMnO₂/C catalyst (a), a scanning transmission electron microscopy micrograph (STEM) image illustrating the physical characteristics of the AgMnO₂/C catalyst (b), elemental mapping images of Mn and Ag from the scanning electron microscopy (SEM) images (c,d), size distribution of AgMnO₂/C catalyst (d,e).

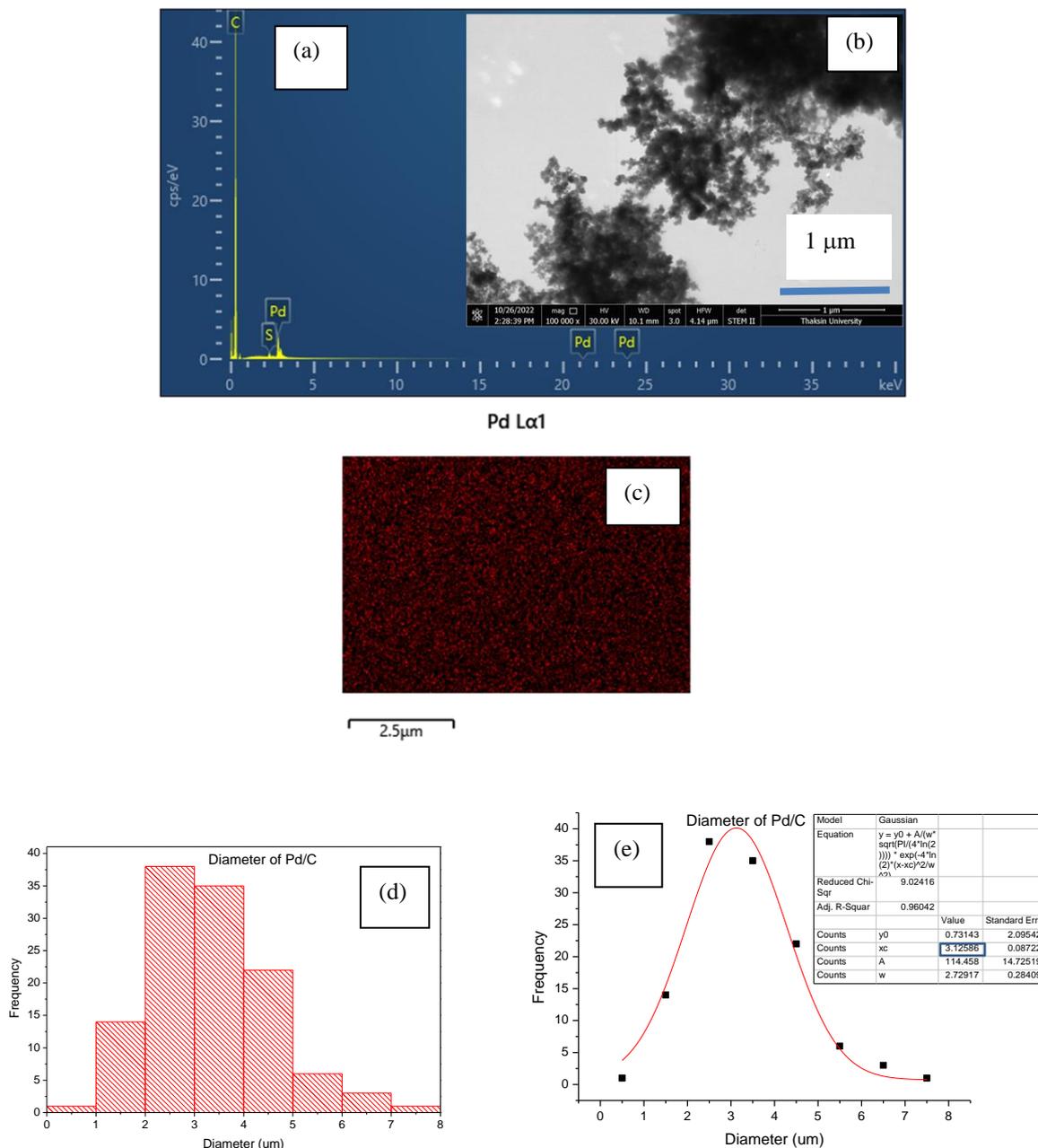


Figure 6 The physical characteristics and elemental composition of the Pd/C catalyst (a), a scanning transmission electron microscopy micrograph (STEM) micrograph illustrating the physical characteristics of the Pd/C catalyst (b), and elemental mapping images of Pd for the scanning electron microscopy (SEM) image (c), size distribution of AgMnO₂/C catalyst (d,e).

XRD structural analysis

Based on the results presented in **Figure 7**, wide-angle XRD confirmed the formation of crystalline AgMnO₂. The XRD pattern of Ag displayed peaks at (111), (200), (220) and (311), confirming its crystalline

nature with a face-centered cubic structure (JCPDS No.: 01-073-6976). Similarly, the XRD pattern of MnO₂ showed peaks at (110), (101) and (211), confirming its crystalline nature with a tetragonal structure (pyrolusite) (JCPDS No.: 01-071-4824).

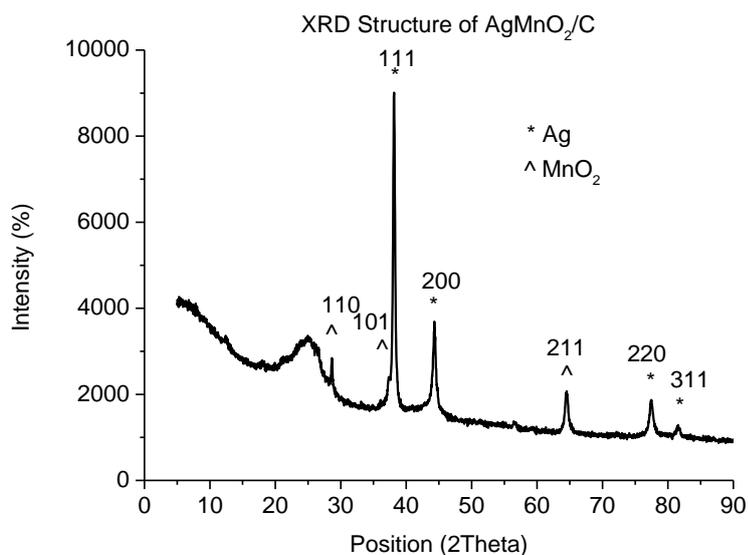


Figure 7 Diffractograms of the AgMnO₂/C catalyst.

Figure 8 confirms the production of crystalline Pd through wide-angle XRD analysis. The XRD pattern of Pd displayed consistent peaks at (111), (200), (220) and

(311), indicating a cubic structure (JCPDS. No.: 01-088-2335). These peaks were specifically associated with the cubic structural phase of Pd.

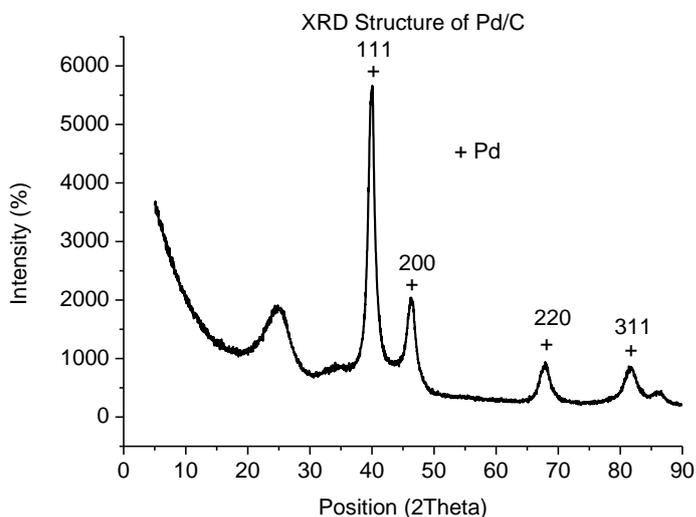


Figure 8 Diffractograms of the Pd/C catalyst.

Table 1 shows that the levels of PTFE (wt%) and Nafion impact oxygen intake and outflow. The oxygen output varies at 10, 15, 20 and 25 wt%. The 20 wt%

PTFE on the carbon cotton fabric showed the most efficient oxygen intake and outflow, possibly because the electrode coating levels affected oxygen passage differently.

Table 1 Study of the oxygen transport volume in and out of the carbon cotton fabric with 10, 15, 20 and 25 wt% PTFE with or without Nafion.

PTFE (wt %)	Nafion	Amount of oxygen entering and exiting of the carbon cotton fabric (%)	
		Oxygen in	Oxygen out
10 wt %	X	98.8	96.8
	√	98.8	97.1
15 wt %	X	98.8	97.1
	√	98.8	97.5
20 wt %	X	98.8	98.0
	√	98.8	98.0
25 wt %	X	98.8	97.7
	√	98.8	97.9

X: Without Nafion

√: With Nafion

Study of the leakage characteristics of carbon cotton fabric in a membraneless alkaline fuel cell prototype

The study of leakage-seepage characteristics in a membraneless alkaline fuel cell prototype revealed that 20 wt% PTFE with and without Nafion did not penetrate

or leak into the KOH solution, as depicted in **Figure 9** and **Table 2**. The 20 wt% PTFE on the carbon cotton fabric showed no surface seepage and no KOH solution leakage, possibly because the electrode coating of 20 wt% PTFE has hydrophobic properties.

Table 2 Leakage-seepage characteristics of carbon cotton fabric in membraneless fuel cell prototypes with 10, 15, 20 and 25 wt% PTFE with or without Nafion.

PTFE (wt %)	Nafion	Characteristics of leakage - seepage of carbon cloth
10 wt %	X	It appears to seep to the surface and a small amount of KOH solution leaks out.
	√	It appears to seep to the surface and a small amount of KOH solution leaks out.
15 wt %	X	It looks like it absorbs the facial skin. However, no KOH solution leaked.
	√	It looks like it absorbs the facial skin. However, no KOH solution leaked.
20 wt %	X	No seepage of the surface and no leakage of KOH solution.
	√	No seepage of the surface and no leakage of KOH solution.
25 wt %	X	It looks like it absorbs the facial skin. However, no KOH solution leaked.
	√	It looks like it absorbs the facial skin. However, no KOH solution leaked.

X: Without Nafion

√: With Nafion

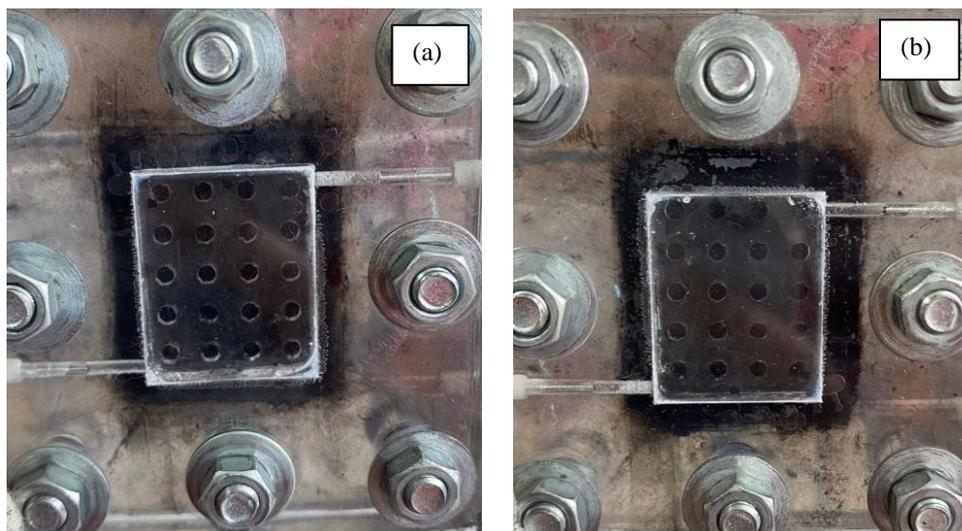


Figure 9 Leakage-seepage images of the membraneless alkaline fuel cell prototype with 20 wt% PTFE, and carbon cotton fabric without Nafion (a) and with Nafion (b).

Characterization of carbon cotton fabric surfaces using postassembly scanning electron microscopy in a membraneless alkaline fuel cell prototype

The physical characteristics of the carbon cotton fabric surface with 20 wt% PTFE, without Nafion showed no peeling or soaking with KOH solution, while the surface with 20 wt% PTFE with Nafion remained intact and did not absorb KOH solution due to its hydrophobic properties. The investigation of the physical properties of the carbon cotton fabric surface in

a membraneless alkaline fuel cell prototype involved the use of scanning electron microscopy (SEM) to examine surface expansion. The carbon cloth was treated with a combination of 20 wt% PTFE and Nafion. The observed expansion is believed to result from the adsorption of Nafion and a 0.1 M KOH solution on the carbon surface. **Figure 10** illustrates the expanded carbon layer surface, contrasting it with the cotton-based carbon cloth with 20 wt% PTFE but without Nafion. Nafion can absorb water and swell, and when hydrated, it can further expand the volume of the carbon-Nafion composite.

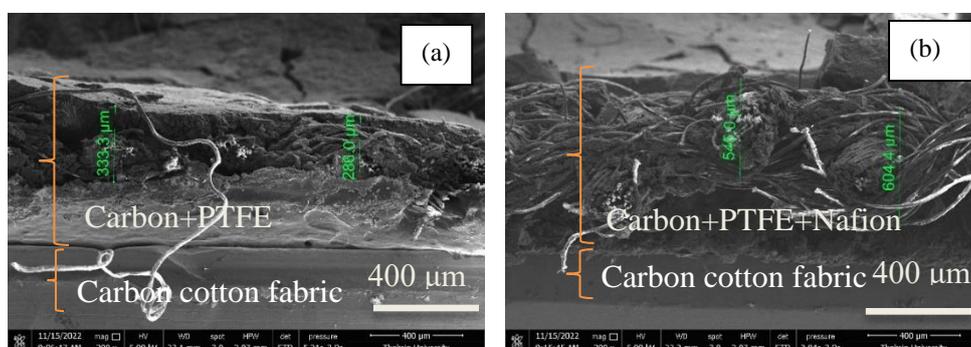


Figure 10 Scanning electron microscopy images of the carbon cotton fabric surfaces after measurements in a membraneless alkaline fuel cell prototype using (a) 20 wt% PTFE-untreated Nafion and (b) 20 wt% PTFE-added Nafion.

Efficiency measurement of an alkaline fuel cell using community alcohol as fuel

The efficiency of an alkaline fuel cell using community alcohol as fuel involved the fabrication of an air cathode electrode using a porous conductive material and coating it with a Pd/C anode catalyst and

AgMnO₂/C cathode catalyst for application in alkaline fuel cells. The experiment used a potassium hydroxide solution with a 0.1 M concentration, while the fuel source was a 0.1 M community alcohol. Additionally, air was supplied to the air cathode side housing at a flow rate of 4 mL/min.

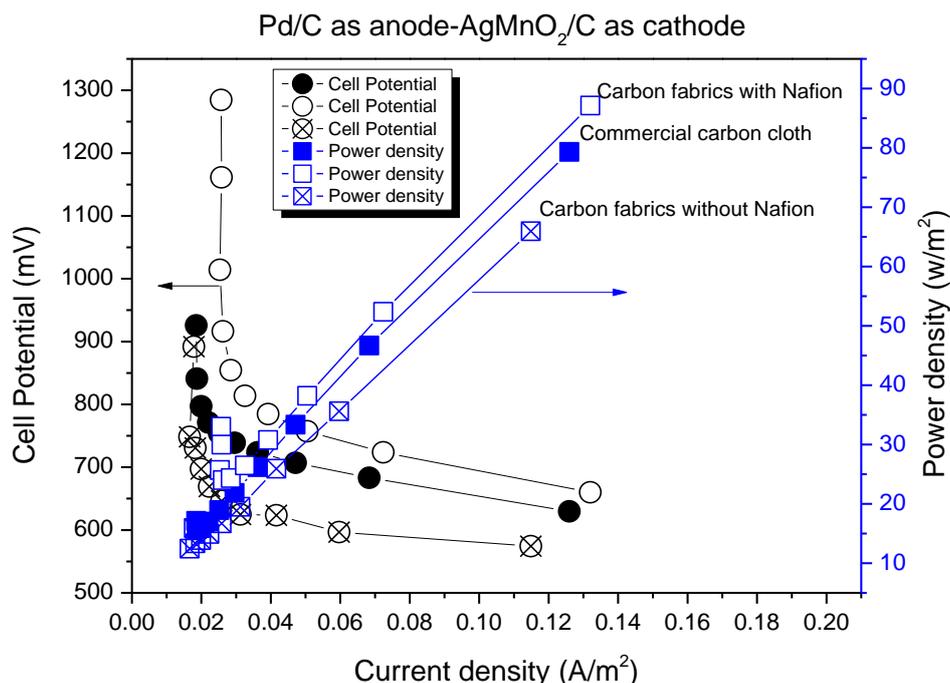
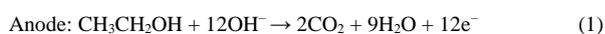


Figure 11 Comparison of the efficiencies of the Pd/C anode and AgMnO₂/C air cathode from carbon cotton fabric and PTFE (20 wt%) with/without Nafion in air cathode alkaline fuel cells at 25 °C.

During the in-situ fuel cell test, the combination of the Pd/C catalyst anode and the AgMnO₂/C catalyst cathode, along with an air cathode made from PTFE (20 wt%) - carbon cotton fabric without Nafion (**Figure 11**), yielded electricity generation of 65.95 W/m². Eq. (3) [20] illustrates that the complete theoretical reaction leads to the formation of carbon dioxide and water.



In a separate in-situ fuel cell test involving the Pd/C catalyst anode and the AgMnO₂/C catalyst cathode, the air cathode was fabricated using PTFE (20 wt%) - carbon cotton fabric and included Nafion (as illustrated in **Figure 11**). It yielded electricity generation of 87.12 W/m², and the power output of the air cathode sourced from a Taiwanese company was 79.31 W/m². The power density output was comparable to that achieved with an air cathode sourced from a Taiwanese company. Nafion boosts proton transport within the electrode structure, aiding in the more effective dispersion and utilization of catalysts on the carbon surface. This polymer binds carbon particles together, enhancing the structural integrity of the electrode.

Additionally, its hydrophilic properties assist in managing the water content within the electrode.

Conclusions

Physical Characterization: The carbon cotton fabric surfaces from cotton fabrics with added PTFE (wt% of 10, 15, 20 and 25) mainly exhibited smooth surfaces with PTFE adhering to the surface. However, upon the addition of Nafion, the adhesion of carbon powder and PTFE was affected, impacting the surface smoothness.

Leakage Characteristics: Investigation of carbon fabric leakage in the membraneless alkaline fuel cell prototype revealed that the carbon cotton fabric from cotton with 20 wt% PTFE, both with and without Nafion, did not experience leakage of the KOH solution or oxygen gas. Oxygen gas meter readings showed that the carbon cloth with 20 wt% PTFE had an oxygen output of 98.0 %, close to the oxygen input of 98.8 %. These results indicate that oxygen gas can effectively pass through the carbon cloth from the cotton fabric with 20 wt% PTFE, regardless of the presence of Nafion within the prototype, which is a membraneless alkaline fuel cell.

During the assessment of fuel cell efficiency involving the Pd/C catalyst anode and the AgMnO₂/C catalyst, it became evident that the power generation of the air cathode constructed using PTFE (20 wt%) and Nafion was comparable to that of the Pd/C catalyst. This power output closely resembled the performance achieved with an air cathode supplied by CeTech Co., Ltd., Taiwan.

Acknowledgements

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