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Characterization of Polyester Cloth as an Alternative Separator to Nafion Membrane in Microbial Fuel Cells for Bioelectricity Generation Using Swine Wastewater

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Polyester cloth (PC) was selected as a prospective inexpensive substitute separator material for microbial fuel cells (MFCs). PC was compared with a traditional Nafion proton exchange membrane (PEM) as an MFC separator by analyzing its physical and electrochemical properties. A single layer of PC showed higher mass transfer (*e.g.*, for O_2/H^+/ions) than the Nafion PEM; in the case of oxygen mass transfer coefficient (k_o), a rate of 50.0×10^{-5} cm·s^{-1} was observed compared with a rate of 20.8×10^{-5} cm/s in the Nafion PEM. Increased numbers of PC layers were found to reduce the oxygen mass transfer coefficient. In addition, the diffusion coefficient of oxygen (D_O) for PC ($2.0–3.3 \times 10^{-6}$ cm^2/s) was lower than that of the Nafion PEM (3.8×10^{-6} cm^2/s). The PC was found to have a low ohmic resistance (0.29–0.38 Ω) in the MFC, which was similar to that of Nafion PEM (0.31 Ω); this resulted in comparable maximum power density and maximum current density in MFCs with PC and those with Nafion PEMs. Moreover, a higher average current generation was observed in MFCs with PC (104.3 ± 15.3 A/m^3) compared with MFCs with Nafion PEM (100.4 ± 17.7 A/m^3), as well as showing insignificant degradation of the PC surface, during 177 days of use in swine wastewater. These results suggest that PC separators could serve as a low-cost alternative to Nafion PEMs for construction of cost-effective MFCs.

Keywords: Microbial fuel cell, polyester cloth, proton exchange membrane, separator, swine wastewater

Introduction

Microbial fuel cells (MFCs) are attractive owing to their ability to repurpose wastewater for electricity generation through electrochemically active bacteria (EAB). The MFCs generally comprise anode and cathode compartments separated by a proton exchange membrane (PEM) [3]. Protons and electrons produced by the oxidation of organic compounds (through catabolism by EAB) in the anode compartment are transferred to the cathode compartment through the PEM and external circuit, respectively. Subsequent H_2O formation at the cathode occurs by the reduction of oxygen through a reaction with the transferred proton and electron. The PEM used as a separator plays a crucial role in MFCs; it governs both substrate and oxygen diffusion and transfer of protons to the cathode compartment, which are factors that significantly influence their performance [2, 7, 19]. MFCs without a separator (*e.g.*, PEM) show a decreased internal resistance, resulting in improved power density [20]; however, the diffusion of oxygen and substrate leads to lower activity of EAB and reduced coulombic efficiency (CE) [19, 21].

Nafion is commonly used as the PEM in MFCs. However, the Nafion PEM in the MFCs has several problems such as high cost, high oxygen and substrate crossover, biofouling, cations transfer rather than protons, and pH imbalance [1, 7, 24]. Importantly, the high cost of the Nafion PEM is prohibitive for practical applications of MFCs when the

financial impact of scaling up MFCs to industrial scale is considered. [6, 9]. Furthermore, through a PEM, the concentration of cationic species (including NH_4^+, K^+, Na^+, Ca^{2+}, and Mg^{2+}) is five orders of magnitude higher than the concentration of protons in the anolyte; this results in an unbalanced pH between the anode and cathode, which inhibits proton transport through the PEM. This considerably decreases the performance of MFCs when synthetic wastewater is used as feedstock [24]. Swine wastewater, which contains a higher concentration of cationic species than synthetic wastewater, would therefore suffer much more severe limitations to proton transport through the PEM [14, 15]. As such, when choosing a separator to replace the Nafion PEM in MFCs, both cost and performance must be major considerations. Various separators such as salt bridges, ion-exchange membranes, composite membranes, and porous materials have been explored for application in MFCs as less expensive materials than Nafion PEM. Salt-bridge MFCs showed low oxygen diffusion and higher CE than a cation exchange membrane (CEM) MFC, but low power density of the MFC was observed in the salt-bridge variant due to high internal resistance [22].

Li *et al*. [17] reported in 2011 that CEMs commonly suffer from poor proton transfer ability. Anion-exchange membranes (AEMs) usually have higher substrate diffusion rates [13] and have been found to be more prone to deformation than CEMs, leading to high internal resistance [26]. Sulfonated polyether ether ketone (SPEEK), an example of a composite membrane, was compared with Nafion 117 in MFCs [9]. Although SPEEK showed higher chemical oxygen demand (COD) removal (88%) than Nafion 117 (76%), lower overall power production (77.3 mW/m^2) than Nafion 117 (106.7 mW/m^2) was observed. Porous separator materials that enable nonselective charge transfer can be used as a replacement for Nafion membranes in MFC separators. Nonwoven fabric filters (NWFs) were found to have the advantages of lower cost (between 350^{th} and 700^{th} of the Nafion price), less ohmic loss, and higher power production than the Nafion PEM as well as long-term stability over 300 days [5]. Additionally, glass fiber mats with a thickness of 1 mm used as separators in MFCs showed a high CE value (81%), due to a low oxygen mass transfer coefficient ($k_O = 5.0 \times 10^{-5}$ cm/s).

Polyester, which is an inexpensive porous material, had not been evaluated as a separator for MFC application. In this study, we investigated the use of polyester cloth (PC) as an alternative separator to Nafion PEMs in MFCs. For this task, the physical and electrochemical properties of PC relevant to MFC performance were compared with those of a Nafion PEM.

Materials and Methods

Characterization and Preparation of Separators

Polyester cloth (PC, 100% polyester; linear density: 21.6 × 22.4 D; warp & weft fabric count: 234 × 175 yarns/inch) was used in this study and the test of its mechanical properties, including morphology observation, bursting strength, water resistance, weight, and thickness, were conducted by the Korea Apparel Testing & Research Institute. The proton exchange membrane (Nafion NAF NR424) was purchased from the DuPont Company (USA). The PC was not pretreated and the PEM was pretreated by boiling with 3% hydrogen peroxide, 0.5 M sulfuric acid, and deionized water for 1 h at 80°C in each case (as described by [11]) before use.

Mass Transport through Separators

Uninoculated acrylic reactors (working volume: 1.8 L) were constructed for measurement of mass transport (*i.e.*, proton and oxygen transport) through the separators. The single-, double-, and triple-layer PCs and Nafion 424 (working area: 15 cm × 15 cm) were inserted between the anode and cathode compartments. The double- and triple-layer PCs were prepared by just overlapping layers of PC without any physicochemical treatment. For the proton transport test, 0.5 M HCl was initially injected into the anode compartment, and the pH in the cathode compartment (only filled with deionized water) was measured over time for 420 min. Dissolved oxygen (DO) was analyzed using the same uninoculated reactors. Before the test, oxygen-free conditions in the anode compartment were created by purging with nitrogen gas (99.99%) and the air-saturated conditions in the cathode compartment were maintained using an air pump. For conductivity testing, 0.1 M NaCl solution was used as a conductive chemical compound. All experiments were conducted with stirring in the anode and cathode compartment using magnetic bars at a constant temperature of 24°C.

Oxygen mass transfer coefficients (k_O, cm/s) were calculated using the following equation [27]:

$$k_O = -\frac{V}{At}\ln\left[\frac{C_{1,0}-C_{2,0}}{C_{1,0}}\right]$$

where V is the liquid volume in the anode compartment (180 ml); A is the separator cross-sectional area (225 cm^2); $C_{1,0}$ is the saturated DO concentration in the cathode compartment; and $C_{2,0}$ is the DO concentration in the anode compartment at time t (sec). The diffusion coefficient (D_O, cm^2/s) for the tested separators was calculated as follows:

$$D_O = k_O \times L_t$$

where k_O is oxygen mass transfer coefficient (cm/s) and L_t is the separator thickness (cm).

Microbial Fuel Cell Setup

Ten identical two-compartment MFC reactors were used in this

study; each anode and cathode compartment consisted of transparent polyacrylate sheets, which were separated by the PC or Nafion 424. Silicon gaskets were used for prevention of leakage. The working surface area of both PC and PEM facing the anolyte and catholyte was 25 cm^2 (5 cm × 5 cm). The volumes of the anode and cathode compartments were equal (50 ml; 2 cm length × 5 cm width × 5 cm height). Stainless steel wires (MSBL-35; 3M, Korea) were used for both electrodes [10]; neither were pretreated or coated with any catalyst. They were fully packed into the anode and cathode compartments of the MFCs. The actual working volume in both the anode and cathode compartments was 35 ml. A platinum wire (0.3 mm diameter) was connected to both the anode and cathode as a current collector.

Microbial Fuel Cell Operation

In order to enrich the electroactive bacteria on the anode, the anode compartment was continuously filled with the oxygen-free artificial mixtures that contained 0.560 g/l $(NH_4)_2SO_4$, 0.200 g/l $MgSO_4 \cdot 7H_2O$, 0.015 g/l $CaCl_2$, 0.001 g/l $FeCl_3 \cdot 6H_2O$, 0.020 g/l $MnSO_4 \cdot 4H_2O$, 2.00 g/l CH_3COONa, 1 ml/l of trace mineral mixture [12], 50 mmol/l phosphate buffer (pH 7.0), 10% (v/v) of raw swine wastewater (obtained from a resource recycling plant, Jeongeup-si, Korea), and 10% (v/v) of activated sludge (collected from a wastewater treatment plant, Iksan-si, Korea) as inoculum sources under open-circuit conditions for 12 h and then at closed-circuit conditions (external load of 50 Ω) for 24 h. After 36 h, 5 L of swine wastewater (~2 g/l COD) purged with nitrogen in a 10 L reservoir bottle was circularly fed into the anode compartment at a rate of 1.7 ml/min using a peristaltic pump (505S; Watson-Marlow, UK) until the current generation increased and stable current production was obtained. The swine wastewater was replaced every 84 h before the COD concentration and current production decreased. Air-saturated deionized water was used as a catholyte and was continuously circulated at a rate of 2.0 ml/min using the peristaltic pump. The deionized water was also replaced whenever the swine wastewater was exchanged. The MFCs were operated at a constant room temperature of 24°C.

Water Analyses

The pH, DO, and conductivity were measured by using a pH meter (Orion 3 star; Thermo Scientific Instrument Inc., USA), a DO meter (YSI-500A; YSI Inc., USA), and a conductivity meter (HI 8633; Hanna Instruments) after calibrations, respectively.

Electrochemical Analyses

The cell voltages were monitored and recorded every 5 min by a multi-data acquisition system (Model 2700; Keithley Instruments Inc., USA) that was connected to a computer. The polarization curves of the MFCs were obtained by varying the external resistance from 5 Ω to 300 kΩ with a Ag/AgCl reference electrode (MF-2052; Bioanalytical Systems Inc., USA) placed near the anode electrode (<5 mm) when voltages and potentials remained at a steady level for 10 min. The current density (A/m^3) was calculated according to Ohm's law, I = V/Rv, where I is the current (A), V is the measured cell voltage (V), R is the external resistance (Ω), and v is the measured electrode volume of the anode (m^3). The power density (W/m^3) was calculated as P = VI/v. Coulombic efficiency was calculated using $CE = Q_s/Q_{th} \times 100\%$, where Q_s is the total coulombs calculated by integrating the current over time, and Q_{th} is the theoretical amount of coulombs available depending on the COD removal in the MFC.

A potentiometer (Reference 600; Gamry Instruments Inc., USA) was used to measure the electrochemical impedance spectra (EIS) over a frequency range from 10^{-2} Hz to 10^5 Hz by applying a sinusoidal excitation signal of 10 mV.

Results

Characterization of PC

In Fig. 1A, the field emission scanning electron microscopy (FE-SEM) images show the morphology of the PC surface with a woven mesh support layer. Fig. 1B presents the cross-sectional view of the PC, showing the single or double layer of crossed thin polyester fibers. The weight and thickness of the PC were 39.2 g/m^2 and 0.004 cm, respectively. The bursting strength and water resistance of the PC was 640 kPa and 44.2 cmH_2O, respectively. The water flux of a single-layer PC in the uninoculated reactor was 0.02 $l \cdot m^{-2} \cdot h^{-1}$, but the water that permeated through double- and triple-layer PCs in the cathode compartment was not observed during 48 h of testing.

Mass Transfer of PC and Nafion

Fig. 2A shows the pH variation over time in the cathode compartment of uninoculated reactors into which the PC or the Nafion 424 separators had been inserted between the

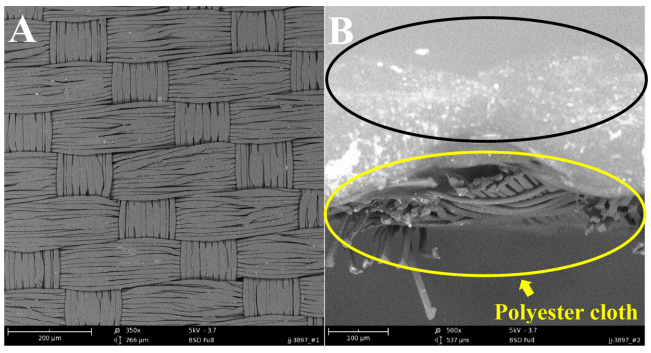

Fig. 1. SEM images of the polyester cloth: (**A**) front view and (**B**) side view.

* Black circle: A glass tape for supporting the polyester cloth to take a photo of the side layer from the FE-SEM. * Yellow circle: The side view of polyester cloth.

anode and cathode compartments. The pH in the anode compartment was maintained at ~0.31 by injection of 0.5 M HCl (theoretical value: pH 0.301) and the pH values in the cathode compartment of the uninoculated reactors at initial sampling time were approximately 5.3. The pH values in the cathode compartment of uninoculated reactors using PC and Nafion 424 separators dropped dramatically during 3 h and then stabilized, except in the case of triple-layer PC. The uninoculated reactors with single-layer PC showed the largest pH change in the cathode compartment, from 5.30 to 1.80, followed by the double-layer PC (from 5.30 to 2.97), Nafion 424 (from 5.28 to 2.86), and finally the triple-layer PC (from 5.26 to 4.50). Fig. 2B presents the changes in conductivity in the cathode compartment of uninoculated reactors using single-, double-, and triple-layer PC and Nafion 424 separators. The largest increase in conductivity was observed for single-layer PC, followed by Nafion 424, double-layer PC, and triple-layer PC. Fig. 2C indicates the diffused DO concentrations in the anode compartment. The DO concentration of PC decreased with the increasing number of PC layers. The change in diffused DO concentration (from 0.42 to 1.77 mg/l) through the triple-layer PC was similar to Nafion 424 (from 0.70 to 1.57 mg/l) over 420 min. Furthermore, even though the oxygen mass transfer coefficient (k_O) of single-layer PC (50.0×10^{-5} cm/s) was higher than that of Nafion PEM (20.8×10^{-5} cm/s), the k_O values of PC were reduced by increasing the number of PC layers (Table 1). Thus, the diffusion coefficient of oxygen (D_O) of PC (2.0 to 3.3×10^{-6} cm^2/s) was lower than that of Nafion PEM (3.8×10^{-6} cm^2/s).

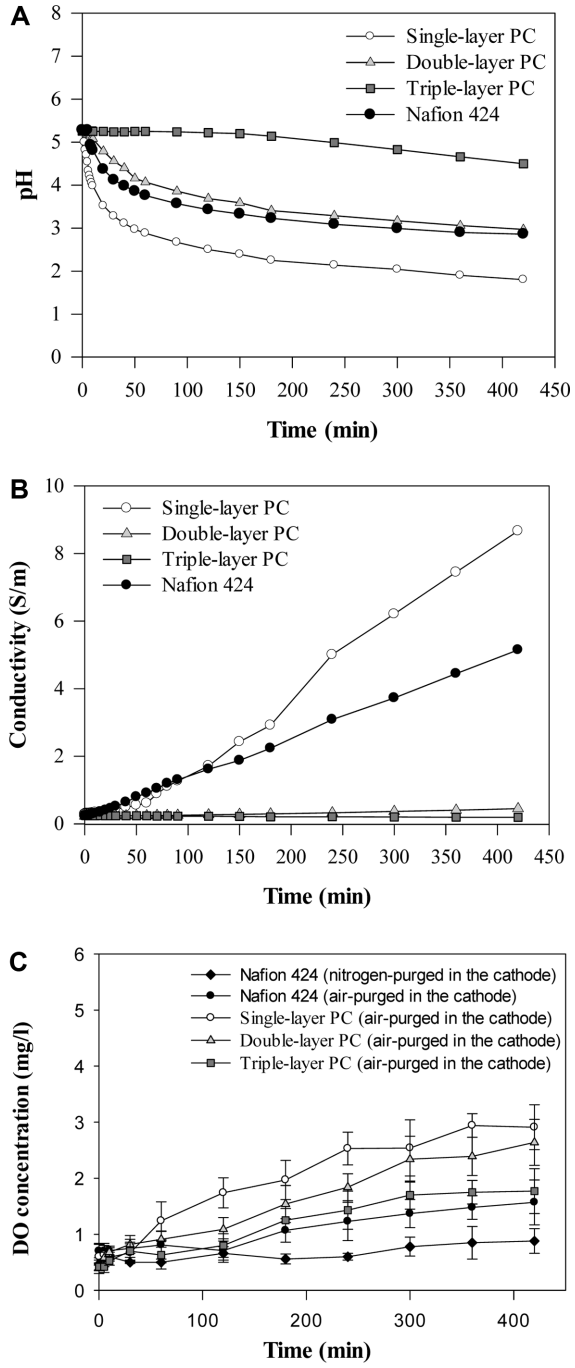

Fig. 2. Change of pH (**A**), conductivity (**B**), and DO concentration (**C**) in the anode compartment through the single-, double-, and triple-layer PCs, and Nafion 424 membrane over time.

MFC Performance

Two-compartment water-cathode MFCs, equipped with the triple-layer PC or the Nafion 424 as a separator, continuously generated electricity under 50 Ω of external resistance from the swine wastewater for 177 days, as seen in Fig. 3. After the first 3 days, the current production

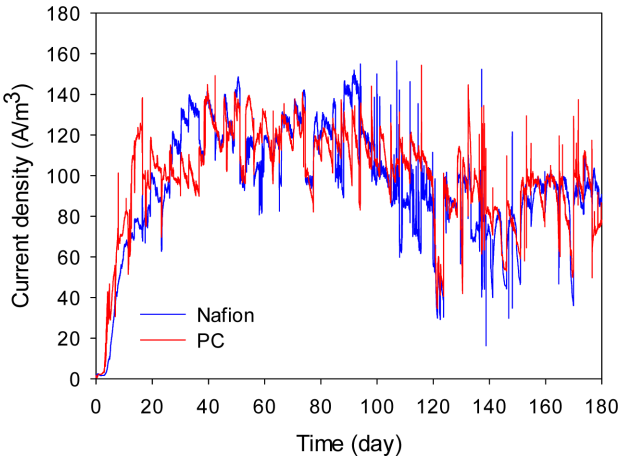

Fig. 3. Evolution of current density in the triple-layer PC-separated MFC and N424-MFC over time.

Table 1. Thickness, oxygen mass transfer coefficient (k_o), diffusion coefficient of oxygen (D_O), ohmic resistance, and cost of different separators in MFCs.

Separators	Thickness (cm)	k_O ($\times 10^{-5}$ cm/s)	D_O ($\times 10^{-6}$ cm^2/s)	Ohmic resistance (Ω)	Cost (USD/m^2)	Reference
Polyester (single-layer)	0.004	50.0	2.00	0.29	75	This study
Polyester (double-layer)	0.008	41.6	3.33	0.34	-	This study
Polyester (triple-layer)	0.012	23.8	2.86	0.38	-	This study
PEM (Nafion 424)	0.018	20.8	3.75	0.31	2,600	This study
Glass fiber 1.0	0.1	5.0	5.0	2.26	-	[27]
J-cloth	0.03	290	86.9	0.21	400	[27]
SPEEK	-	-	-	811[a]	45 (12 cm^2)	[9]
PEM (Nafion 117)	0.019	67	11.5	93[a]	1,400	[5]
NWF4	0.013	70	9.1	51[a]	2	[5]
CEM (CMI-7000)	0.046	9.4	4.3	84[a]	200	[13]
AEM (AMI-7000)	0.046	9.4	4.3	88[a]	80	[13]

[a]Internal resistance value.

started to gradually increase, indicating that the biofilm in the anode of MFCs had acclimated, and then the current generation stabilized from day 10 to day 177 at 70–140 A/m^3. For the first 30 days, the current production of the triple-layer PC-separated MFC (TLPC-MFC) was higher and faster to increase than that of the Nafion 424-separated MFC (N424-MFC). During the period of stable current generation (from 10 days to 177 days), the average current production of TLPC-MFCs was higher (104.3 ± 15.3 A/m^3) than that of N424-MFCs (100.4 ± 17.7 A/m^3).

The performance of the MFCs was examined under steady-state conditions, and polarization curves (voltage/potential curves and a power density curve as a function of current density) were graphed, as seen in Figs. 4A and 4B. Open circuit voltages (OCVs) of 0.55 V and 0.61 V were obtained from TLPC-MFCs and N424-MFCs, respectively. The anode potentials (-0.577 V) and cathode potentials (−0.03 V) of TLPC-MFCs (with Ag/AgCl ref. electrode) were observed in OCVs, which were lower than those of N424-MFCs (−0.52 V and 0.08 V, respectively). Even though a lower OCV was observed in TLPC-MFCs, the performance of TLPC-MFCs were comparable to that of N424-MFCs, indicating that TLPC-MFCs had a similar maximum power density of 15.4 ± 2.9 W/m^3 (0.5% lower than in N424-MFCs) and maximum current density of 127.6 ± 19.2 A/m^3 (3.9% higher than in N424-MFCs).

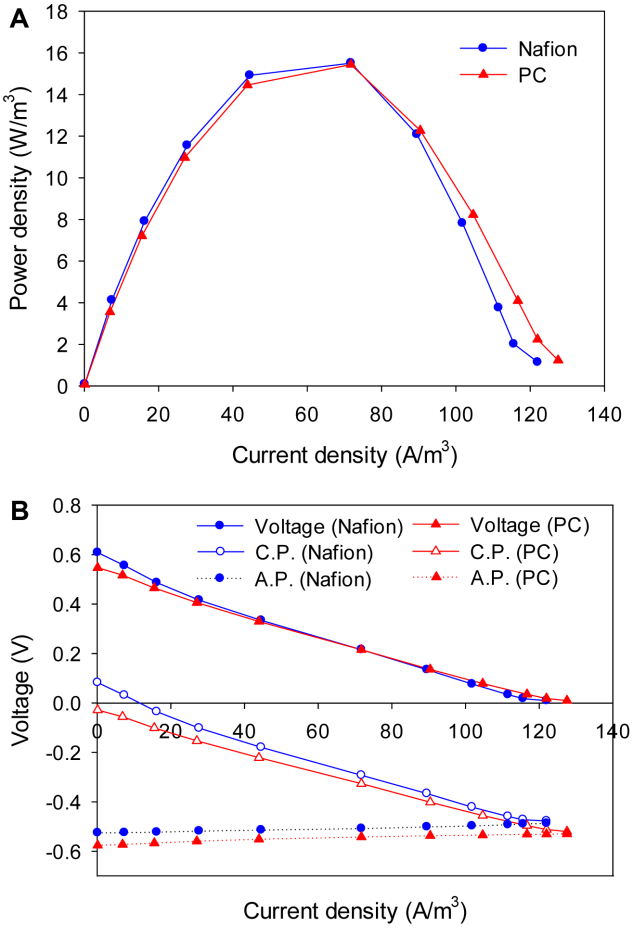

Fig. 4. (**A**) P-I curves and (**B**) V-I curves and anode/cathode potentials of the triple-layer PC-separated MFC and N424-MFC.

Electrochemical Impedance Spectroscopy Analysis

The impedance spectra of the PC-MFCs and N424-MFCs are shown by the Nyquist diagram (Fig. 5), in which the

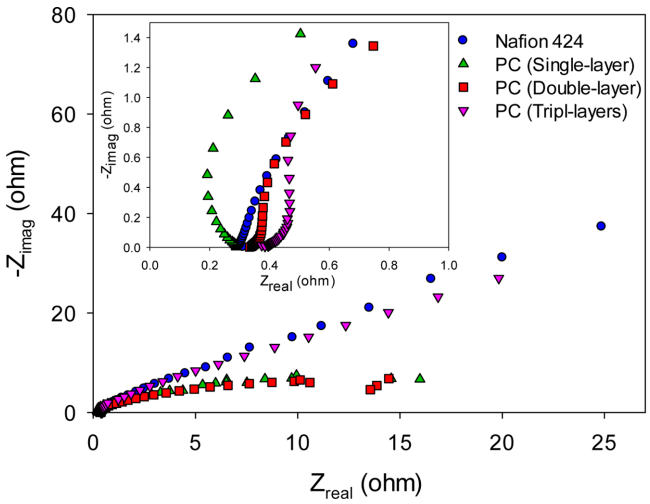

Fig. 5. EIS data representing the ohmic resistance in TLPC-MFCs and N424-MFC.

real impedance (Z_{real}) is plotted against the imaginary impedance ($-Z_{imag}$) at different frequencies (from 1.0×10^{-2} to 1.0×10^{5} Hz) under the static condition (anolyte: swine wastewater containing 2.0 g/l of COD) in the two-compartment MFCs. The Nyquist plots showed that ohmic resistances of PC-MFCs rise according to the number of layers (single-, double-, and triple-layer), resulting in ohmic resistances of 0.29, 0.34, and 0.38 Ω, respectively, and the ohmic resistance in the N424-MFCs was 0.31 Ω. These results indicated that the ohmic resistances from the membrane and electrolyte for both separators were almost equal to the Nyquist plot (a difference of only 0.2–0.7 Ω). However, the overall internal resistances of single- and double-layer PC-MFCs (between 12 and 15 Ω) were much lower than the internal resistance of N424-MFCs (not obtained, but the semi-circle larger than the PC-MFCs).

Discussion

As a control material, Nafion 424 was employed instead of Nafion 117 (which is normally used in MFC study) to prevent the phenomenon of membrane tearing by the stainless-steel wire electrodes. The ideal attributes of a separator for efficient operation of MFCs on a larger scale are low cost, low oxygen diffusion from cathode to anode due to anaerobically metabolic electrogenesis (or reaction of exoelectrogens) in the anode compartment, selective proton transport (minimal transport of cations) from anode to cathode, low internal resistance between the anode and cathode compartments, and resistance to biofouling and clogging. Nafion membrane is not applicable in large-scale MFCs owing to high cost. The PC used in this study is 35 times cheaper than Nafion 424, as seen in Table 1. Even when compared with Nafion 117, the PC is 19 times lower in price. This economic price of PC can reduce the cost of the MFC construction. By evaluating the physicochemical properties of PC compared with Nafion 424, it was established that the single-layer PC had the highest proton transport, cation transport, oxygen diffusion, and water penetration. It was anticipated that the water penetration would lead to increased mass transfer of all ions between the anode and cathode compartment in the inoculated reactors, as shown in Fig. 2. It has been reported that oxygen diffusion inhibited current generation in the MFCs [20]. The number of PC layers, when increased, beneficially reduced the following: oxygen diffusion from air-saturated catholyte to anaerobic anolyte; water penetration; and mass transfer of ions (ascertained by evaluating the conductivity from injection of 0.5 M NaCl). However, the amount of transported protons from the anode to the cathode was reduced by increasing the number of PC layers. Although low proton transport was recorded in the uninoculated reactors, the total current production of TLPS-MFCs was higher than that of N424-MFCs for 177 days (Fig. 3) and the maximum power and current density were comparable with those of N424-MFCs, as discussed in the Results section. These results showed that proton transport might be a less important parameter for influencing the performance of MFCs, as the amount of transferred protons through the triple-layer PS appeared to be sufficient for current generation during the operation of the MFCs. As mentioned for the ideal attributes of a separator, the internal resistance is also one of the crucial parameters responsible for determining the MFC performance. The whole MFC's resistance is classified as the sum of charge transfer (activation) resistance, ohmic resistance (representing the solution resistance, electrode resistance, membrane resistance), and diffusion (mass transfer) resistance. Lower ohmic resistances (between 0.29 and 0.38 Ω) for both MFCs were obtained, compared with those reported in previous literature [16, 27]. However, although the kinetics-dependent internal resistance (*i.e.*, charge transfer resistance and mass transfer resistance) in MFCs were not measured using EIS owing to the unclear response of EIS, it was observed that the internal resistances of Nafion 424 and triple-layer PC were high owing to incomplete semicircle-plots of charge transfer resistance (generally reflected as the diameter of the half-circle). We assumed that the reason for the charge

transport being limited was cathode limitation [8]. In Fig. 4B, the cathode potentials of TLPC-MFC (from −0.03 to −0.52 V) and N424-MFC (from 0.08 to −0.48 V) sharply decreased, whereas anode potentials of TLPC-MFC (from −0.58 to −0.53 V) and N424-MFC (from −0.52 to −0.49 V) gradually increased. The cathode limitation might be caused by (i) lack of catalyst coated on the cathode, (ii) deionized water being used as the catholyte in MFCs, and (iii) no control of pH in the cathode compartment. The performance of MFCs can be improved by overcoming the cathode limitation; possible solutions to these problems are injection of phosphate buffer (neutral pH) or NaCl into the cathode compartment and using a metal-based catalyst loaded onto the cathode. When ferricyanide was used as an electron acceptor instead of dissolved oxygen in the cathode, the maximum power of MFCs increased by 50–80% [23]. For cost-effective MFC operation, the use of the catalysts and electron acceptors to improve the MFC performance should be investigated with reference to economic viability. Membrane fouling has been a problem in the environmental industry as well as in research. Several reports indicated that fouling of the membrane decreased the performance of MFCs [4, 25]. It was observed that the current generation gradually decreased in both TLPC-MFCs and N424-MFCs over 177 days of operation; however, the change in the measured ohmic resistance as time passed was negligible (from 0.38 Ω to 0.45 Ω for PC and from 0.31 Ω to 0.43 Ω for Nafion 424). This phenomenon might be explained by the small distance between each separator and the anode [18]. Additionally, the PC would be highly resistant to actual wastewater. The non-degraded surface of PC operated for 6 months was observed with both raw and pretreated swine wastewater as feedstock in MFCs.

This study indicated, through the physicochemical and electrochemical analyses of PC, that PC could be utilized as a cost-effective alternative to Nafion separators in MFCs. Therefore, it improves the viability of large-scale MFC use. Low cost of materials is a crucial factor for the successful application of MFCs at a large scale. We demonstrated that the polyester cloth can replace the Nafion membrane, as the former has a lower acquisition cost and shows better performance in MFCs using swine wastewater.

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