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Simultaneous wastewater treatment and electricity generation by microbial fuel cell: Performance comparison and cost investigation of using Nafion 117 and SPEEK as separators

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HIGHLIGHTS

- Applying different methods for wastewater treatment.
- Production of 77.3 $mW \cdot m^{-2}$ electricity and 88% COD removal by MFC working with SPEEK.
- Economical comparison between SPEEK and Nafion 117

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ABSTRACT

Microbial Fuel Cell (MFC) is a new device for simultaneous electricity production and wastewater treatment. The major concern of this technique is the high operational cost that is caused by Nafion 117 used as a Proton Exchange Membrane (PEM) and Pt as a cathode catalyst for oxygen reduction. Sulfonated poly ether ether ketone (SPEEK) was used as a PEM in MFC for this study. The results indicate that even though the power production of MFC with SPEEK (77.3 $mW \cdot m^{-2}$) was lower than Nafion 117 (106.7 $mW \cdot m^{-2}$), it is more cost effective, due to its lower price. Our cost evaluation suggests that the use of SPEEK in power production is about two times more cost effective than that of Nafion 117. Furthermore, its chemical oxygen demand (COD) removal (88%) is higher than Nafion 117 (76%); and thus, SPEEK, given substantial increases in power density may be a promising PEM in MFCs at an industrial scale.

1. Introduction

Because of the high energy requirements of conventional wastewater treatment systems, the demand for alternative cost effective and commercialized treatment technologies is increasing [1,2]. Moreover, due to increasing concerns on energy security and climate change impact of conventional fuels worldwide, there is a high interest in finding new sources of clean and sustainable sources of energy, with zero hydrocarbon content. Microbial fuel cell is a new device that converts the chemical energy of substrates into electrical energy using microorganisms [3,4]. Microbes in the anode part of the device oxidize the substrates to generate electrons and protons. Electrons are transferred to the cathode compartment through the external circuit, while the protons pass through the membrane [5,6]. MFCs are simultaneously used for wastewater treatment and the production of clean energy. Thus, this use of MFCs has a great potential in broad applications, such as home electrical generators and self-feeding robots [7]. However, applications have become limited, due to low power output, short stability, and non-economic reasons [8]. Generally, MFCs consist of two chambers (i.e., anode and cathode), which are separated by a Proton Exchange Membrane (PEM). Several factors affect the performance of MFCs, including types of microorganisms, substrates, electrodes, and membranes. Of these, membranes play a very important role of separating the anode from the cathode. The membrane should be able to allow the protons to pass from the anode to cathode, while preventing the passing of oxygen and substrates [9,10].

The high cost of MFCs is mainly due to the expensive price of cathode catalyst, which is usually Pt, and the Nafion 117 PEM, which is commonly

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used as the separator in MFCs [11]. Together, they cover 90% of the total cost of MFCs. Therefore, the development of an economical catalyst and PEM has attracted wide attention from scientists. In 2011, Rahimnejad et al., [12] used a poly ether sulfone (PES)/Fe_3O_4 nanocomposite membrane as an alternative for Nafion 117 PEM. They concluded that it could produce higher amounts of energy compared to Nafion 117. However, the problem with the nanocomposite membranes, was their porous structure, which made long-term operation impossible [13,14]. In another study by our group, Ghasemi et al., [15] used Nafion/activated carbon nanofiber (ACNF) as a PEM. The power generated by this novel carbon nanocomposite membrane was 30% higher than Nafion 117. However, because the membrane was still a nafionic membrane, the capital cost of the MFC was still infeasible and the membranes used were still porous.

Sulfonated poly ether ether ketone (SPEEK) can be a good alternative to Nafion 117, due to its good hydrophilic properties and proton conductivity. It has some negative fixed charge ions (SO_3^-) that allow cations to pass through, while it rejects the passing of anions [16,17]. Furthermore, from previous use as a PEM in direct methanol fuel cell (DMFC), it produced good results. Due to the expensive price of Nafion 117 [18], which makes MFC non-commercial, and the properties of SPEEK as a good PEM, the objective of this study is to compare the power generation performance of two types of membrane in microbial fuel cell and their cost effectiveness.

2. Experimental methods

2.1. Synthesis of SPEEK and the determination of the Degree of Sulfonation (DS)

For the preparation of SPEEK, 20 g of PEEK powder (Goodfellow Cambridge Limited, UK) was dissolved slowly in 500 mL of 95–98% concentrated sulphuric acid (R & M Chemicals, Essex, UK). This solution was stirred vigorously until the entire PEEK was dissolved completely. Next, the homogenous solution was continuously and thoroughly stirred at a controlled temperature of 80 °C for 30 h (in this study), in order to obtain 52% DS. The SPEEK solution was then poured into a large excess of ice water, in order to precipitate the SPEEK. The solid was then collected by filtering the solution through a Whatman filter paper. Finally, the SPEEK was dried at 70 °C to removing any remaining water before use.

2.2. Determination of DS

The degree of sulfonation was measured by 1H Nuclear Magnetic Resonance (FT-NMR ADVANCE 111 600 MHz with Cryoprobe) spectroscopic analysis (Bruker, Karlsruhe, Germany). Before measurement, the SPEEK was dissolved in dimethyl sulfoxide (DMSO-d_6). The DS can be calculated by the following equation:

$$\frac{DS}{S-2DS}=\frac{A_1}{A_2}(0\leq DS\leq 1) \quad (1)$$

where "S" is the total number of hydrogen atoms in the repeat unit of the polymer before sulfonation which is 12 for PEEK, $A_1(H_{13})$ is the peak area of the distinct signal and A_2 is the integrated peak area of the signals corresponding to all other aromatic hydrogen [19]. To calculate DS in percent (DS %), the answer for DS has to be multiplied by 100.

2.3. MFC configuration

Two cubic shaped chambers were constructed from Plexiglas, with a height of 10 cm, width of 6 cm, and length of 10 cm (giving a working volume of 420 ml). They were separated by a Nafion 117 proton exchange membrane (PEM). Oxygen was continuously fed into the cathode by an air pump at a rate of 80 ml/min. Both the cathode and the anode projected the surface areas of 12 cm^2. The cathode was carbon paper, coated with 0.5 mg/cm^2 Pt and the anode (as described above) was plain carbon paper [12,20]. The schematic figure of MFC was shown in Fig. 1.

2.4. Enrichment

Palm oil mill effluent (POME, Indah Water Konsortium, pH = 4.7, BOD = 25,000 mg/l, COD = 50,000 mg/l) anaerobic sludge was used for the inoculation of MFCs. The media contained 5 g of glucose, 0.07 g of yeast extract, 0.2 g of KCl, 1 g of NaH_2PO_4.4H2O, 2 g of NH_4Cl, 3.5 g of $NaHCO_3$ (all from the Merck company), 10 ml of Wolfe's mineral solution and 10 ml of Wolfe's vitamin solution (added per liter). All experiments were conducted in an incubator at 30 °C. Furthermore, the cathode chamber contained a phosphate buffer solution, which consisted of 2.76 g/l of NaH_2PO_4, 4.26 g/l of Na_2HPO_4, 0.31 g/l of NH_4Cl, and 0.13 g/l of KCl (all from the Merck company) [21].

2.5. Analysis and calculation

Nicolet 5700 FTIR (Thermo Electron, USA) was performed to identify the functional group of Nafion 117 and SPEEK membranes. Scanning electron microscopy (SEM, Supra 55vp-Zeiss, Germany) was implemented to observe the attachment of microorganisms on to the surface of the anode electrode. Moisture had to be removed from the biological samples (POME mix culture sludge) by critical drying. They were then coated with a conductive material (such as gold or carbon), with a thickness of approximately 20–50 nm, in order to make them conductive for the SEM analysis.

To measure the COD, samples were first diluted 10 times and mixed with 2 ml of diluted samples with a digestion solution of a high-range COD reagent, then heated at 150 °C for 2 h in a thermoreactor (DRB200), which was read with a spectrophotometer (DR 2800). The voltage was measured using a multimeter (Fluke 8846A), and the power density curve was obtained by applying different loads to the system and calculating the power at different loads.

The current was measured using the:

$$I=\frac{V}{R} \quad (2)$$

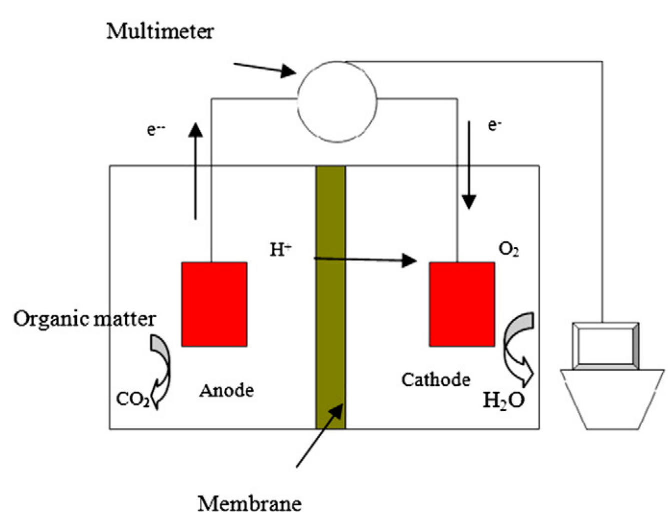

Fig. 1. Schematic of MFC.

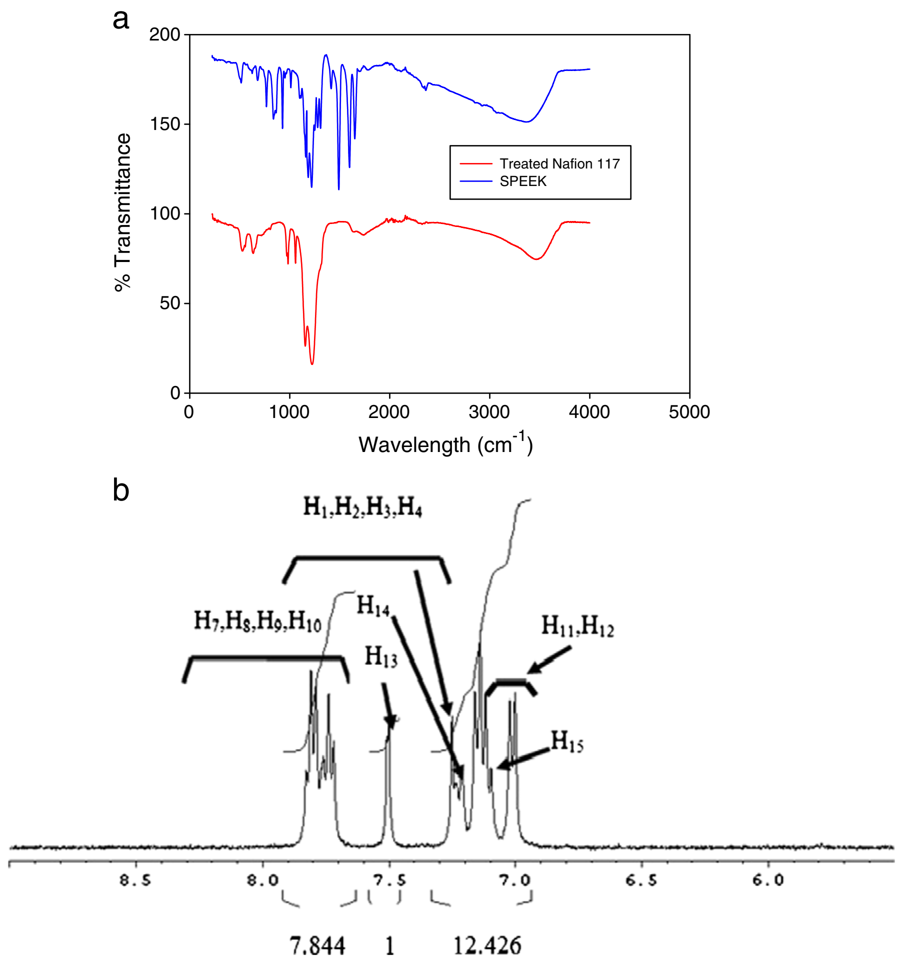

Fig. 2. a. FTIR analysis of SPEEK and pre-treated Nafion 117. b. H NMR spectra of sulfonation.

where, I is the current (amps), V is the voltage (volt), and R is the applied external resistance (ohm).

The power density was calculated using the following equation:

$$P = R \times I^2 \tag{3}$$

where, R is the applied external resistance (ohm) and I is the current (amps) (calculated using Eq. (1)).

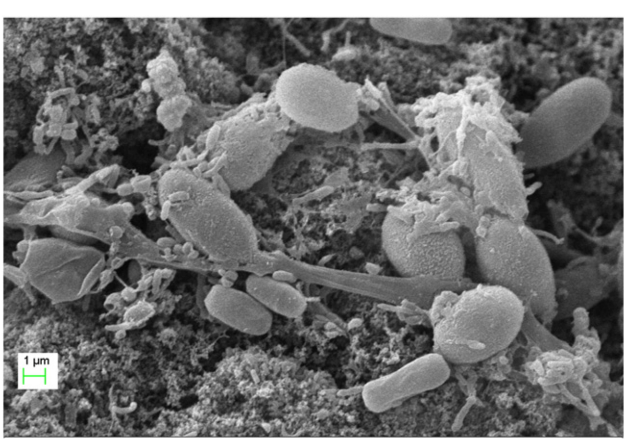

Fig. 3. Picture of attached biofilm to the electrode in the anode chamber.

The coulombic efficiency (CE) was calculated as the current over time; until the maximum theoretical current was achieved. The evaluated CE over time was calculated using the following equation:

$$CE = \frac{M\int_0^t Idt}{FbV_{an}\Delta COD} \tag{4}$$

where, M is the molecular weight of oxygen (32), F is the Faraday's constant, $b = 4$ indicates the number of electrons exchanged per mole of oxygen, V_{an} is the volume of liquid in the anode compartment, and ΔCOD is the change in chemical oxygen demand (COD) over time, '*t*'.

2.6. Pre-treatment of PEMs

Nafion 117 should be pre-treated before use in MFC system. The pre-treatment procedure is boiling in distilled water, 3% hydrogen peroxide, or 0.5 M sulphuric acid for 1 h each one and then stored in water until applying in the system. Also SPEEK should be kept in the water after fabrication till use [22,23].

3. Results and discussion

3.1. FTIR analysis of SPEEK and the degree of sulfonation

Fig. 2a shows the FTIR analysis of SPEEK and pre-treated Nafion 117. As shown, there are two clear bands in both of PEMs; one in the range

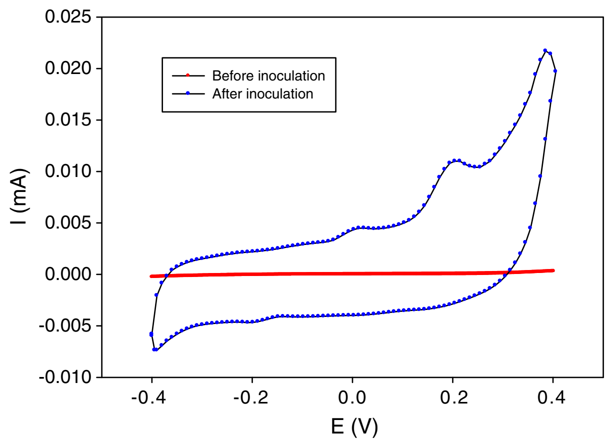

Fig. 4. Catalytic activity of the biocatalysts.

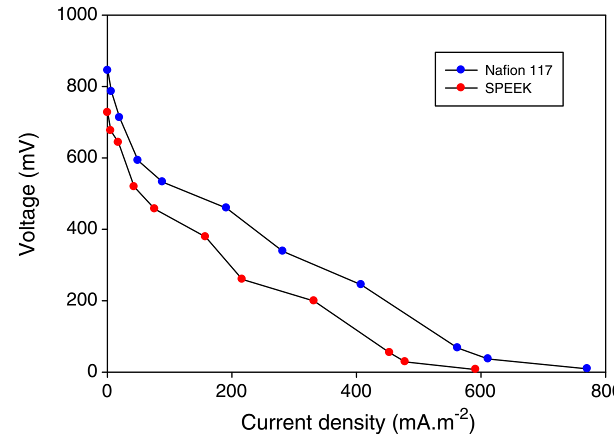

Fig. 6. Polarization curve for SPEEK and Nafion 117 membranes.

1020–1250 cm^{-1} and the other (big peak) at 3200–3500 cm^{-1}. The first band revealed the SO_3^- group of the membrane that was introduced by the pre-treatment Nafion 117 and the sulfonation to SPEEK, which by its negative charge, makes the membrane a promising choice for the passing of protons. Meanwhile, the second band is the O–H group that increases the hydrophilicity of the membrane, which directly increases the conductivity of the membranes. This is an important factor of PEMs [24].

Also, NMR analysis of the SPEEK is shown in Fig. 2b. The reason for producing SPEEK in the range of 50% DS (52%) was because, as the DS increases to 60–70%, proton conductivity enhances. However, this reduces the mechanical and thermal stabilities of the polymer [25]. So, SPEEK with 52% DS was used in this study, based on the better results achieved from the MFC, as reported in pervious study; and from the better mechanical and thermal strengths of PEEK at about a 52% degree of sulfonation [19,26].

3.2. Bacteria attachment

Fig. 3 shows the biofilm attached to the electrode in the anode chamber, when the MFC was in a stable condition. As shown, different types of bacteria (which play the role of a biocatalyst in transferring electrons to the electrode) were attached to the electrode [27].

In order to see the catalytic activity of the biocatalysts, the developed mix culture biofilm (which formed on the electrode's surface) was analyzed by CV under the same conditions of the MFC experiment. As shown in Fig. 4 there was no oxidation and reduction peaks, before inoculation of the anode chamber, with the mix culture microorganisms. However, after inoculation, a clear oxidation peak can be seen. The oxidation peak shows that POME is an active biocatalyst for oxidation in the anode compartment. The maximum oxidation peak is at around 0.01 mA and 200 mV (0.2 V), which is provided by POME sludge microorganism [28].

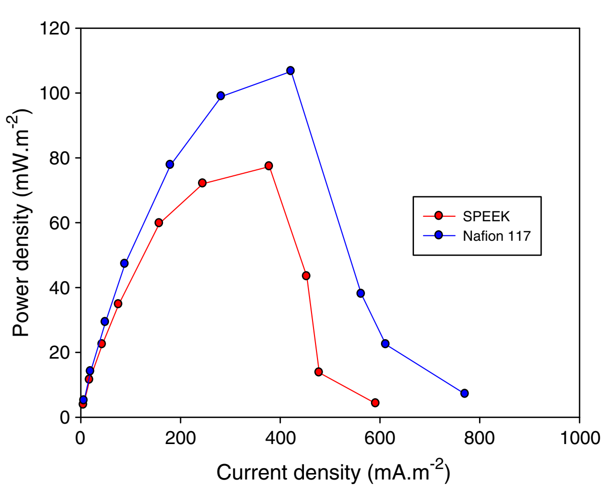

Fig. 5. Power density of the Nafion 117 and SPEEK membranes in microbial fuel cell.

3.3. Power density and polarization curve

Fig. 5 shows the power density graph of the microbial fuel cells working with Nafion 117 and SPEEK PEM, while the other factors were constant. As shown, both power density graphs go up and reach a maximum value before starting to fall back down [29]. The maximum power generated by the MFC working with SPEEK as the PEM was 77.3 $mW \cdot m^{-2}$, while 106.7 $mW \cdot m^{-2}$ was obtained from the MFC working with Nafion 117 as the PEM. This means that the system working with Nafion 117 as the PEM can produce 27.5% more power than the system working with SPEEK.

The MFC's polarization curve is shown in Fig. 6. The internal resistance of the MFC is the slope of the V–I curve, or in other words, the slope of the current versus the voltage is the internal resistance [30]. Based on that, the internal resistance of the MFC working with Nafion 117 is about 727 Ω. However, for the MFC working with SPEEK, it was higher at about 811 Ω. This could have been due to a higher conductivity and lower activation loss in the MFC working with Nafion 117.

Table. 1 shows a summary of the data taken from the MFC systems working with Nafion 117 and SPEEK as PEMs.

3.4. COD removal and coulombic efficiency

Fig. 7 shows the CE and COD removal of the MFC systems working with Nafion 117 and SPEEK as PEMs. As shown, both systems have a high COD removal of more than 70%. The MFC working with Nafion 117 had 76% COD removal while the system working with SPEEK had 88% removal. However, the CE percentage of the system working with Nafion 117 (29%) was 61% more than the system working with SPEEK (18%). This shows that the COD removal of the SPEEK membrane was higher, but the energy generation of Nafion 117 was better.

Table 1
Summary of the collected SPEEK and Nafion 117 membranes MFC system data.

Types of membrane	P_{max} ($mW \cdot m^{-2}$)	I_{max} ($mA \cdot m^{-2}$)	OCV in steady state condition (mV)	Internal resistance (Ω)
Nafion 117	106.7	421.7	645	727
SPEEK	77.3	378.3	813	811

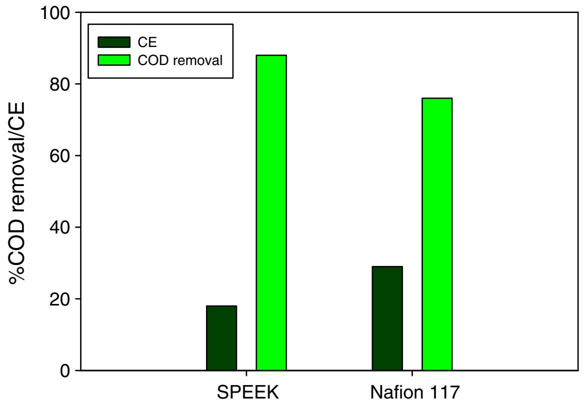

Fig. 7. Columbic efficiency and COPD removal of the Nafion 117 and SPEEK membranes in MFC system.

This could have been due to the higher internal resistance of the SPEEK membrane when the other parameters were the same [29,31].

3.5. Economic analysis

This section compares the cost performance of the two types of membranes in the MFC; namely Nafion 117 and SPEEK, for power generation. While Nafion 117 membranes demonstrated a higher level of electricity generation compared to SPEEK, their cost is significantly higher. As shown in Table 2, the electricity generation from 12 cm^2 of Nafion 117 and SPEEK is 106.7 and 77.3 $mW \cdot m^{-2}$, respectively. However, the cost of 12 cm^2 of Nafion is almost three times higher than that of SPEEK [32]. Therefore, it is imperative to investigate the costs associated with the power generated by each type of membrane. Such an analysis provides decision-makers with useful information to understand which type of membrane is more cost efficient or more economically viable to use.

Table 2 shows the costs associated with the use of 12 cm^2 of Nafion 117 and SPEEK. The feedstock for both types of membranes is raw palm oil mill Effluent (POME). Malaysia as one of the world largest producers of palm oil generates huge amounts of POME each year. In this study, we assumed that the cost of acquiring POME as feed stocks is negligible and that the same amount of POME is used per unit of fuel cell system for the two membranes. We also did not consider the economic benefits of the resulting treated water and reduction in methane emissions for the different types of membrane. Hence, the main cost considered was the purchase price of membranes and the fuel cell units. The latter reflects the balance of the system cost while excluding the membrane component. It should also be noted that this study did not consider the lifespan of the two alternative membranes; rather it evaluates the use of the membranes in their maximum power generation.

Based on the known cost for each membrane, we calculated and compared the cost of power (per Watt) generated by the respective membrane. The results are shown in Table 2. The results clearly show that even though the amount of electricity generated form Nafion 117 was higher (138%) than that of SPEEK, the lower costs of power generated associated with the use of SPEEK suggests that the use of this membrane is relatively more economical. The balance of system cost besides the membrane was estimated by assigning a numeraire price equal to 1 for every 12 cm^2 fuel cell system. This approach implies that the focus of analysis is on the relative cost differential between the alternative membranes for every unit (W) of power generated at its maximum capacity. Since Nafion 117 generates a lot higher power density, the balance of the system cost on a per Watt basis as shown in Table 2 is substantially lower by 28% (USD9.73) relative to SPEEK (USD12.93). However, the overall cost of power generated per watt by SPEEK is still substantially lower by 47% relative to that of Nafion 117.

While the use of SPEEK is relatively more feasible in terms of cost efficiency, the lower power density of SPEEK is a cause of concern as it is less space efficient to generate a comparable amount of power. Reducing the price of Nafion 117 may be a prospective strategy. Given the observed power density capacity, Nafion 117and SPEEK would be equally cost effective should the market price of the former declines by 48%.

The Feed in Tariff (FiT) mechanism has been introduced in Malaysia since late 2011. This framework provides incentives for the production of power from renewable energy sources including biomass, biogas, waste, solar photo voltaic, and mini hydro. The energy produced will be sold to the national grid through a power purchase agreement under the FiT mechanism which guarantees a favorable purchase rate throughout the duration of the project. The use of POME as the feedstock for power generation through MFCs may qualify for the FiT incentives. However, at this stage, the cost of power generated from MFC, as deliberated in the preceding section is far from being feasible relative to other conventional and renewable energy sources in Malaysia. Latest investigation reveals that capital expenditures for the production of various renewable energies in Malaysia are highly minute relative to MFC, ranging from USD2.5 to USD4 per Watt [33]. Hence, more comprehensive studies are imperative to improve the power density and cost efficiency of MFCs.

4. Conclusion

This study compares the cost efficiency use of SPEEK relative to Nafion 117 in an MFC system. Our results have shown that SPEEK is the better choice, as the cost of power produced with SPEEK is 50% less than that of Nafion 117. While the unit cost of power generated from the MFC is still far higher relative to other types of renewable energy, this study offers a new horizon in the industrialization of MFC; especially in Malaysia, which produces large amounts of POME. It paves the way for the identification of new types of membranes and catalysts, which may be comparable to other renewable energy in terms of power density and cost efficiency.

Table 2
Cost comparison of power generation in MFC by Nafion 117 and SPEEK as PEM.

		Nafion 117	SPEEK	Percent (Nafion 117/SPEEK) × 100	Source
A	Cost per 12 cm^2 (USD)	120	45	266	Neburchilov et al. [32]
B	Electricity Generated per 12 cm^2 (mW)	106.7	77.3	138	Estimated
C	Electricity tariff (per mW) (USD)	0.10×10^{-6}	0.10×10^{-6}	–	Ministry of Energy, Green Technology and Water (2011)[a]
$D = A/B$	Membrane cost of electricity generation per mW (USD)	1.125	0.5821	193	Estimated
$E = D \times 1000$	Cost of electricity generation per W	1125	582	193	Estimated
$F = (1000 / B) \times 1$	Cost (USD) of fuel cell units (balance of system) per W (assuming a numeraire price = USD1 per 12 cm^2)	9.37	12.93	72	Estimated
$G = (E + F)$	Total cost per W (USD)	1134	595	190	Estimated

[a] Ministry of Energy, Green Technology and Water (2011), available at http://talkenergy.files.wordpress.com/2011/02/asean-electricity-tariff-2011.pdf.

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