# ENHANCEMENT OF SULFONATED POLY ETHER ETHER KETONE BASED ELECTROLYTES FOR BIPOLAR MEMBRANE FUEL CELL

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# DEDICATION

This thesis is dedicated to my beloved *Abah*, *Sayed Daud Bin Sayed Deraman* and *Ma*, *Roslina Binti Mohamed* for their endless love, affection, support, encouragement and prays of day and night make me able to get such success and honor. It is also dedicated to my sister, *Syarifah Noorsyuhada* and brother, *Sayed Ahmad* for being my side and giving me the strength to chase my dreams.

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# ABSTRACT

The limitation of the existing bipolar membrane fuel cell (BPMFC) is the produced power output which is not efficient due to water flooding at the junction layer of the proton exchange membrane (PEM) and anion exchange membrane (AEM). In worse situations, this membrane fuel cell leads to delamination between the PEM and AEM layer of a bipolar membrane (BPM) or disintegration between the membrane layer and electrode part. The electrolyte material and the design of the hot-press parameters for BPM electrode assembly are influential factors in hydrated membrane disintegration that reduce the BPMFC performance. Thus, this research developed BPM-based sulfonated poly(ether ether ketone) (sPEEK) by considering the membrane synthesizing method and designing adhesion parameters to improve the compatibility of the PEM/AEM junction layer. The sPEEK was composited with two different materials; titanium dioxide ($TiO_2$) with 0.5 – 2.0 wt.% and polyethersulfone (PES) with 5 – 20 wt.%, as PEM anode. Meanwhile, AEM cathode was prepared by the crosslinking of a sulfonic acid group from sPEEK with a quaternary ammonium group from trimethyl-amine that is known as crosslinked quaternary ammonium PEEK (cQAPEEK) membrane at three different chloromethylation times of 48, 72, and 96 hours. The phase inversion via heating technique was applied for preparing PEM and AEM electrolytes. The developed PEM and AEM electrolytes were characterized according to their structures, morphologies, thermal and mechanical stabilities, and physiochemical and electrochemical properties. Then, the PEM and AEM were hot-pressed to develop BPM. The design of the hot-pressed parameters was based on pressure and temperature while the time was made constant. To obtain the optimum parameters, the design was determined based on response surface methodology (RSM) analysis. The results for PEM showed that sPEEK/PES possessed the highest proton conductivity of 7.18 mS $cm^{-1}$ for 5 wt.% PES, whereas sPEEK/$TiO_2$ PEM obtained the highest proton conductivity of 9.08 mS $cm^{-1}$ for 0.5 wt.% $TiO_2$ with excellent mechanical and thermal stabilities. Both of these optimum composites membranes were used in BPM with the best cQAPEEK AEM which had the highest anion conductivity of 5.38 mS $cm^{-1}$ when chloromethylized for 72 hours. Based on RSM analysis, the optimal pressure and temperature for hot-pressed PEM and AEM were 3 tons/square inch and 120 °C. It produced the best adhesion of membranes where no gap existed between AEM and PEM as proven through scanning electron microscopy analysis. Furthermore, there was no excessive attachment at the PEM/AEM junction and this provided low ionic resistance and created a better ion pathway at the junction. The ionic conductivity of BPM sPEEK/$PES_5$-$cQAPEEK_{72h}$ was 8.16 mS $cm^{-1}$, while BPM sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ showed 8.39 mS $cm^{-1}$ of ionic conductivity. In terms of power output, the sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ showed higher peak power density, which is 53.12 mW $cm^{-2}$ with increment of about 3.13 % due to better ionic conductivity than sPEEK/$PES_5$-$cQAPEEK_{72h}$ (51.51 mW $cm^{-2}$). However, the sPEEK/$PES_5$-$cQAPEEK_{72h}$ showed lower hydrogen/oxygen fuel permeation during operation than sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ and Nafion 117-$cQAPEEK_{72h}$ according to voltage versus time graph which indicates that it has excellent membrane durability. By considering power output as the main investigated parameter, this study chose sPEEK/$TiO_2$-$cQAPEEK_{72h}$ as the best BPM electrolyte due to its high performance and sufficient durability. Remarkably, all the developed membranes were in good condition without any disintegration of the layers after testing at various temperatures and environments. This research showed that the BPM electrolyte based modified PEEK provided the best BPM material and adhesion design of the 3 tons/square inch pressure and 120 °C temperature demonstrated a better degree of adhesion between the PEM and the AEM.

# ABSTRAK

Kekangan sel bahan bakar membran bipolar (BPMFC) yang sedia ada adalah penghasilan kuasa keluaran yang tidak cekap yang disebabkan oleh pengumpulan air pada lapisan persimpangan membran pertukaran proton (PEM) dan membran pertukaran anion (AEM). Dalam keadaan yang lebih teruk, sel bahan bakar membran ini membawa kepada pemisahan antara lapisan PEM dan AEM membran bipolar (BPM) atau pemecahan antara lapisan membran dan bahagian elektrod. Bahan elektrolit dan parameter tekanan panas untuk pemasangan elektrod BPM adalah faktor yang mempengaruhi pemecahan membran terhidrat yang mana mengurangkan prestasi BPMFC. Oleh itu, penyelidikan ini bertujuan untuk menghasilkan BPM berasaskan sulfonat poli(eter eter kiton) (sPEEK) dengan mengambil kira kaedah mensintesis membran dan parameter rekabentuk lekatan untuk meningkatkan keserasian lapisan simpang PEM/AEM. SPEEK dikomposit dengan dua bahan berbeza iaitu titanium dioksida ($TiO_2$) dengan komposisi 0.5 – 2.0 wt.% dan polietersulfon (PES) dengan komposisi 5 – 20 wt.%, sebagai anod PEM. Sementara itu, katod AEM dihasilkan melalui ikatan sambung silang dari kumpulan asid sulfonik sPEEK dengan kumpulan amonium kuartener dari trimetil-amina yang dikenali sebagai membran bersambung silang amonium kuartener PEEK (cQAPEEK) pada tiga waktu klorometilasi yang berbeza iaitu 48, 72, dan 96 jam. Pembalikan fasa melalui teknik pemanasan digunakan untuk menghasilkan elektrolit PEM dan AEM. Elektrolit PEM dan AEM dicirikan mengikut struktur, morfologi, kestabilan terma dan mekanikal, dan sifat fisiokimia dan elektrokimia. Kemudian, tekanan panas digunakan terhadap PEM dan AEM untuk menghasilkan BPM. Parameter tekanan panas divariasikan berdasarkan tekanan dan suhu, manakala, tempoh tekanan ditetapkan. Bagi mendapatkan parameter optimum, reka bentuk tekanan panas ditentukan berdasarkan analisis kaedah sambutan permukaan (RSM). Hasil untuk PEM menunjukkan bahawa sPEEK/PES memiliki kekonduksian proton tertinggi iaitu 7.18 mS $cm^{-1}$ untuk komposisi PES 5 wt.%, manakala sPEEK/$TiO_2$ PEM memperoleh kekonduksian proton tertinggi (9.08 mS $cm^{-1}$) untuk 0.5 wt.% komposisi $TiO_2$ dengan kestabilan mekanikal dan terma yang sangat baik. Kedua-dua membran komposit optimum ini digunakan dalam BPM dengan cQAPEEK AEM terbaik yang mempunyai kekonduksian anion tertinggi 5.38 mS $cm^{-1}$ ketika diklorometilisasi selama 72 jam. Berdasarkan analisis RSM, tekanan dan suhu optimum untuk PEM dan AEM bertekanan panas adalah 3 tan/inci persegi dan 120 °C. Ia menghasilkan lekatan membran yang terbaik di mana tidak ada ruang antara AEM dan PEM seperti yang dibuktikan dengan analisis mikroskopi elektron imbasan. Selain itu, lekatan berlebihan tidak berlaku di simpang PEM/AEM dan ini mewujudkan rintangan ion rendah dan jalur ion yang lebih baik di simpang. Kekonduksian ion BPM sPEEK/$PES_5$-$cQAPEEK_{72h}$ ialah 8.16 mS $cm^{-1}$, sementara BPM sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ menunjukkan kekonduksian ionik sebanyak 8.39 mS $cm^{-1}$. Dari segi kuasa keluaran, sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ menunjukkan puncak yang lebih tinggi ketumpatan kuasa, iaitu 53.12 mW $cm^{-2}$ dengan kenaikan sekitar 3.13 % kerana kekonduksian ion yang lebih baik daripada sPEEK/$PES_5$-$cQAPEEK_{72h}$ (51.51 mW $cm^{-2}$). Walau bagaimanapun, sPEEK/$PES_5$-$cQAPEEK_{72h}$ menunjukkan kebolehtelapan bahan api hidrogen/oksigen yang lebih rendah semasa operasi daripada sPEEK/$TiO_{2(0.5)}$-$cQAPEEK_{72h}$ dan Nafion 117-$cQAPEEK_{72h}$ mengikut graf voltan melawan masa yang menunjukkan bahawa ia mempunyai ketahanan membran yang sangat baik. Dengan mengambil kira kuasa keluaran sebagai parameter utama yang disiasat, kajian ini memilih sPEEK/$TiO_2$-$cQAPEEK_{72h}$ sebagai elektrolit BPM terbaik kerana prestasi tinggi dan ketahanan yang mencukupi. Luar biasanya, semua membran yang dihasilkan berada dalam keadaan yang baik tanpa berlaku pemecahan lapisan setelah diuji pada pelbagai suhu dan persekitaran. Penyelidikan ini menunjukkan bahawa elektrolit BPM berasaskan modifikasi PEEK menghasilkan bahan BPM terbaik dan rekabentuk lekatan pada tekanan 3 tan/inci persegi dan suhu 120 °C memberikan tahap lekatan yang lebih baik antara PEM dan AEM.

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## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| AEM | - | Anion exchange membrane |
| AFC | - | Alkaline fuel cell |
| BBD | - | Box-Behnken design |
| BPM | - | Bipolar membrane |
| BPMFC | - | Bipolar membrane fuel cell |
| CCD | - | Central composite design |
| CCRD | - | Central composite rotatable design |
| CNT | - | Carbon nanotubes |
| cQAPEEK | - | Crosslink quaternary ammonium poly(ether ether ketone) |
| DC | | Degree of chloromethylation |
| DoE | - | Design of experiment |
| DOF | - | Degree of freedom |
| DMAC | - | Dimethylacetamide |
| DS | - | Degree of sulfonation |
| DSC | - | Differential scanning calorimetry |
| EOD | - | Electroosmotic drag |
| EW | - | Equivalent weight |
| FTIR | - | Fourier transform infrared spectroscopy |
| $^{1}$H NMR | - | Nuclear magnetic resonance |
| HOR | - | Hydrogen oxidation reaction |
| IEM | - | Ion exchange membrane |
| I-V | - | Current-voltage |
| KOH | - | Potassium hydroxide |
| MEA | - | Membrane electrode assembly |
| MCFC | - | Molten carbonate fuel cell |
| MOFs | - | Metal-organic frameworks |
| MS | - | Mean of square |
| NP | - | Nanoparticle |
| OCV | - | Open circuit voltage |
| ORR | - | Oxygen reduction reaction |

| | | |
|---|---|---|
| PAEK | - | Poly(aryl ether ketone) |
| PAES | - | Poly(aryl ether sulfone) |
| PAFC | - | Phosphoric acid fuel cell |
| PAN | - | Polyacrylonitrile |
| PBI | - | Polybenzimidazole |
| PEFC | - | Polymer electrolyte fuel cell |
| PEI | - | Polyetherimide |
| PEM | - | Proton exchange membrane |
| PES | - | Polyethersulfone |
| PEEK | - | Poly(ether ether ketone) |
| PPO | - | Polyphenylene oxide |
| PSF | - | Polysulfone |
| PTFE | - | Polytetrafluoroethylene |
| RH | - | Relative humidity |
| RSM | - | Response surface methodology |
| SEM | - | Scanning electron microscope |
| sPEEK | - | Sulfonated poly(ether ether ketone) |
| sPEES | - | Sulfonated poly(ether ether sulfone) |
| SS | - | Sum of square |
| SOFC | - | Solid oxide fuel cell |
| $T_d$ | - | Thermal degradation |
| $T_g$ | - | Glass transition temperature |
| $TiO_2$ | - | Titanium dioxide |
| TGA | - | Thermal gravimetric analysis |
| TMSCl | - | Chlorotrimethylsilane |
| V-T | - | Voltage-Time |
| XRD | - | X-ray diffraction analysis |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| $AH_A$ | - | Integration area under the graph for $H_A$ region |
| $AH_a$ | - | Integration area under the graph for $H_a$ region |
| $AH_b$ | - | Integration area under the graph for $H_b$ region |
| C | - | Carbon |
| $CO_2$ | - | Carbon dioxide |
| $C_2H_5ClO$ | - | Chloromethyl methyl ether |
| $C_9H_{19}ClO$ | - | Chloromethyl octyl ether |
| $(CH_2Cl)_2O$ | - | Bis(chloromethyl) ether |
| $e^-$ | - | Electron |
| $H^+$ | - | Hydrogen ion |
| $H_A$ | - | Region of the proton at 7.5 ppm |
| $H_a$ | - | Region of the anion at 4.6 ppm |
| $H_b$ | - | Region of the proton at 7.5 ppm |
| $H_{B,C,D}$ | - | Region of the doublets at ~7.2 ppm |
| $H_2O$ | - | Water |
| $H_3O^+$ | - | Hydrated hydrogen |
| $H_2SO_4$ | - | Sulfuric acid |
| $L_d$ | - | Length of the dry membrane |
| $L_w$ | - | Length of the wet membrane |
| $m_d$ | - | Weight of the dry membrane |
| $m_W$ | - | Weight of the wet membrane |
| $N(CH_3)_3$ | - | Trimethylamine |
| $OH^-$ | - | Hydroxyl ion |
| Pt | - | Platinum |
| $SiO_2$ | - | Silicon dioxide |
| $SnCl_4$ | - | Stannic Chloride |
| $SO_3^-$ | - | Sulfonate group |
| $T_d$ | - | Thickness of the dry membrane |
| $T_w$ | - | Thickness of the wet membrane |
| $\sigma$ | - | Ionic conductivity |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the Study

Nowadays, the increase in global awareness on ensuring clean, safe and efficient energy utilization and management has contributed to the development of fuel cell as a future promising and eco-friendly technology for energy production. The fuel cell operates through an electrochemical principle that generates electricity out of various fuels like hydrogen, oxygen, methanol or natural gas. Fuel cell can be categorized into various types, depending on its electrolyte, whereby its operating temperatures and pressures are also varied [1]. There are five main classes of fuel cell, which are polymer electrolyte fuel cell (PEFC), alkaline fuel cell (AFC), molten carbonate fuel cell (MCFC), phosphoric acid fuel cell (PAFC) and solid oxide fuel cell (SOFC). Among them, the PEFC fed with hydrogen/oxygen ($H_2/O_2$) fuel has received the greatest attention in research. This is due to its cleaner by-products, higher performance and efficiency, smoother operations, and better design compactness compared to other classes of fuel cells [2–4]. The heart of PEFC is the membrane electrode assembly (MEA) which consist of platinum/carbon (Pt/C) electrodes, catalyst and solid polymer electrolyte or membrane.

The electrolyte or membrane plays a key role in the PEFC that acts as a media for transmitting ion charges from anode to cathode side with the help of water molecules. There are two types of electrolytes that are commonly used in fuel cell, which are proton exchange membrane (PEM) and anion exchange membrane (AEM). The PEM electrolyte mobilizes the proton ($H^+$) while the AEM electrolyte transmits the anion ($OH^-$). However, the PEFC with single layer electrolyte suffers water management issues due to imbalance water distribution that contributes to insufficient membrane humidification and electrode flooding, which affecting the stack performance, system performance, fuel cell life, and system costs [5–7]. The presence of water inside electrolyte membranes facilitates the ions mobilization either via the Grotthus mechanism or vehicular mechanism which is highly dependent on the water content or degree of humidification especially for dense-structured membrane, where high ionic conductivities are achieved at maximum humidification level [8]. Failing to obtain suitable membrane hydrations will cause high ohmic resistance, fuel crossover, performance degradation, and durability problems [7,9–12].

Membrane dehydration is one of the crucial phenomena that resulted from ineffective water management in fuel cells which likely occurs at the anode side of the membrane. There are three main reasons which contribute to membrane dehydration and these are: (1) sufficient humidification cannot be maintained when feeding the cell with low-humidified or dry reactant gas streams, (2) water formation reaction at the cathode alone is not able to compensate the lack of water, especially when operating at higher cell operating temperatures, and (3) electro-osmotic drag can also lead to dehydrated condition at the anode [9]. However, the main effect of dehydration is the drying of the electrolyte membrane which leads to the decrease in conductivity, higher ionic resistance, and larger ohmic losses. In consequence, it always resulted in a substantial drop in cell potential and thus a temporary power loss. Therefore, a strategy for managing the water properly is required to optimize the fuel cell performance.

Generally, the strategies to manage the water in the fuel cell can be divided into: (1) system design and engineering, which is the addition of auxiliary systems to the basic fuel cell system and (2) material design and engineering or MEA design that involves changing of material structures and properties of the cell components. Among these strategies, the MEA design is the most preferred because it provides less complex cell structure, time saving, and low budget of manufacture [13–17]. With the MEA design strategy, modifications on cell structure, arrangement and position of cell component, electrolyte material and usage of advanced cell component material have been made and applied.

Most recently, several researchers had introduced novel MEA design by integrating PEM anode and AEM cathode electrolytes together. This novel MEA design is also known as a bipolar membrane fuel cell (BPMFC) [18–20]. Similar to the PEFC is the BPMFC, which is also fed with $H_2/O_2$ gases to produce electricity, heat, and water molecules ($H_2O$) as by-products. The difference between them is the number of electrolyte layers where the PEFC consists of single layer electrolyte, while BPMFC has bilayer electrolytes as shown in Figure 1.1.

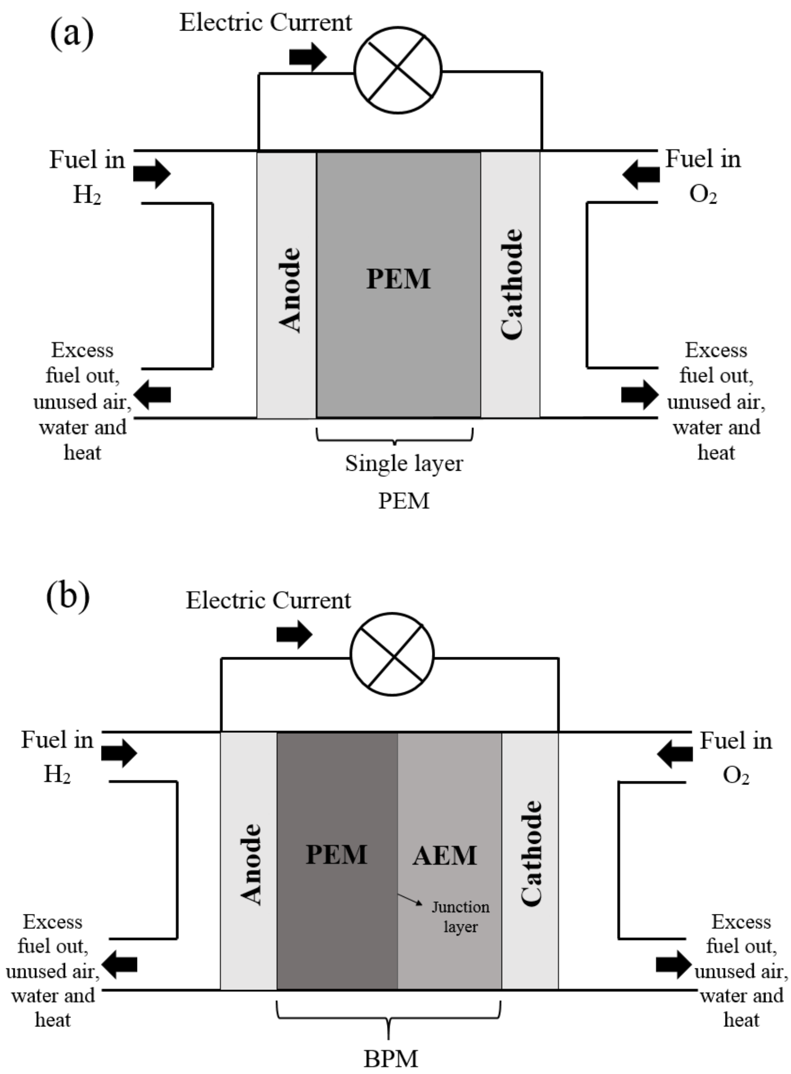


Figure 1.1 Schematic diagram: (a) traditional polymer electrolyte fuel cell and (b) novel bipolar membrane fuel cell [20,21]

The advantage of having bilayer electrolytes is that the cells are able to carry the proton and anion simultaneously where the reaction between ions at PEM/AEM junction will form water molecules that makes BPMFC to have self-humidify behaviour. Therefore, two sources of water that are generated in the BPMFC are via oxygen reduction reaction (ORR) at the cathode and the reaction between $H^+$ and $OH^-$ at the PEM/AEM junction layer. As for the PEFC, the water can only be generated via ORR. Therefore, at certain operating condition such as high temperature, and low relative humidity, the PEFC can easily suffer membrane dehydration due to insufficient water content and limited water formation source. Additional sources of water formation in fuel cell helps the electrolyte to remain hydrated, which minimize the drying phenomenon when relative humidity is low and operating temperature increases.

The ability of BPMFC to self-humidify during operation allows effective water molecules movement. Although BPMFC has the potential to solve membrane dehydration problems, it still requires modification on cell design and electrolyte material selection, since its development is still in the early stage. Also, previous studies claimed that the BPMFC is facing poor electrochemical cell performance and low durability [18,22–24]. These problems resulted from incompatible PEM/AEM materials and poor adhesion contact of PEM/AEM junction layer. Thus, the critical problem of material compatibility and cell design of BPMFC is of great concern in the utilization of BPMFC.

The ability of BPMFC in self-humidifying the electrolytes utilizing water formation at the PEM/AEM junction layer during operations had benefited the cell which makes it able to maintain its hydration even when operating at high temperature and drier conditions. Unfortunately, the only commercially available BPM (Fumasep FBM – FuMATech Germany) in the market is not suitable for BPMFC usage. It is mainly designed for water dissociation purposes where the water molecules are broken down into hydrogen ($H^+$) and hydroxyl ($OH^-$) ions at the PEM/AEM junction. This means that this commercial BPM is not suitable to be employed as an electrolyte for $H_2/O_2$ fuel cell because it leads to membranes dry-out, causing inadequate water content and resulting in poor electrochemical performance [18,25].

The water dissociation and water formation configurations in BPM, are illustrated in Figure 1.2. As an alternative to the development of BPM electrolyte for water formation configuration, some researchers have studied on producing a suitable BPM for BPMFC by utilizing the conventional method of integrating polymeric-based PEM with AEM electrolytes either via hot-press or layer-by-layer casting process [18,26]. This is because, these methods can be more cost-effective and time saving. However this study focuses on the hot press method only.

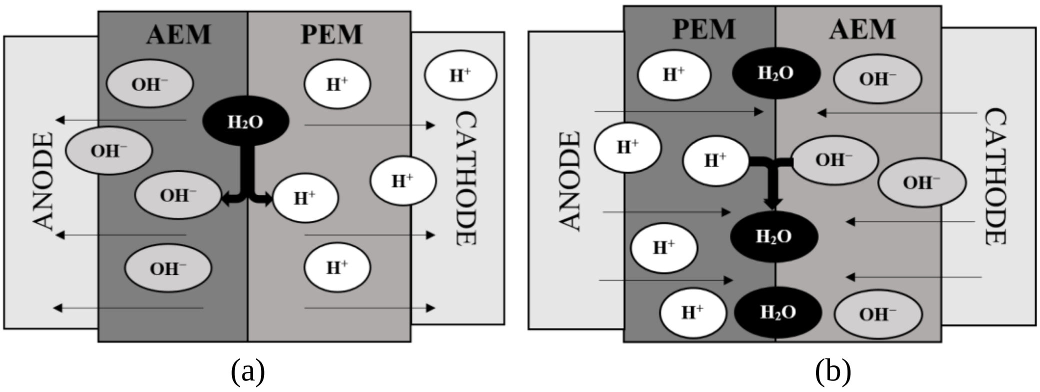


Figure 1.2 Bipolar membrane in (a) water dissociation configuration and (b) water formation configuration [18]

## 1.2 Problem Statement

The current existing issue in BPMFC study is that it suffers poor contact at PEM/AEM adhesion area, water flooding phenomenon, layer disintegration and incompatible electrolyte materials. These factors have led to the degradation of electrochemical performance and the low durability of the BPMFC. In tackling this issue, many materials have been paired on finding the most compatible PEM and AEM materials combination such as Nafion-quaternary ammonium poly(aryl ether sulfone), Nafion-FUMAPEM FAA-3, Nafion-quaternary benzyl trimethylammonium polyphenylene oxide, Nafion-quaternary ammonium polysulphone, and sulfonated poly(ether ether sulfone)-quaternary ammonium polysulphone [18,22,23,26,27]. Unfortunately, the combined PEM and AEM material, thus far has the problems of poor power density and limited operating working time.

These flaws are mainly due to the low water absorption by electrolyte, delamination between PEM and AEM layer, disintegration between BPM layer and electrode, and the accumulation of water at PEM/AEM junction. The main reason that contributes to these flaws is excess water formation at the BPM junction layer. The excess water forming led to a water flooding phenomenon at the junction and cathode side which cause severe membrane swelling. The swell membrane is unable to absorb excess water effectively due to membrane pore blocking (with water) which reduces its transportation rate. The changes in membrane dimension and microstructure contribute to the decrease of membrane elasticity and mechanical strength. Therefore, the adhesion contact between PEM/AEM layer and membrane/electrode layer will be loosened and thus disintegrate with the increase in operation time. Therefore, it is necessary to control the water formation in BPMFC by manipulating its cell component materials, design, and operating condition to avoid the water flooding phenomenon.

Most of the past BPMFC studies paired the commercial perfluorinated Nafion-PEM with the hydrocarbon-based polymer material AEM except the recent study by Manohar *et al.* 2019 [26] that used fully hydrocarbon polymer-based BPM. Although the Nafion PEM showed excellent performance and mechanical properties, it still has several limitations, such as high cost that limit its large-scale application, high fuel permeation that degrade electrolyte durability, and poor performance at high operating temperature and dry conditions. Given these limitations, a sulfonated hydrocarbon-based polymer, such as sulfonated poly(ether ether ketone) (sPEEK) is considered in this research as an alternative for Nafion-PEM in BPMFC to improve the aforementioned limitations. SPEEK is a familiar membrane material in PEFC that has comparable performance with the commercial membrane. This type of membrane has rich sulfonic acid attached to the hydrophobic polymer backbone that allows ionic charge to pass through. The amount of sulfonic acid exists depend on the degree of sulfonation (DS).

High DS allows better ion transportation which led to excellent power output but degradation on mechanical properties due to a worse swelling degree. The high DS of pristine sulfonated membrane is not suitable to be used as PEM of BPM because BPMFC generates a high amount of water than traditional PEFC. This phenomenon makes the electrolyte to be high water-absorbing and consequently, worsenly swell and cracks the electrolytes which reduce its mechanical properties. Therefore, it is necessary to modify the highly sulfonated PEEK while preserving its mechanical properties to take full advantage of its excellent proton conductivity. In this research, two types of modifiers, which are hydrophilic titania or titanium dioxide ($TiO_2$) nanoparticle (NP) and hydrophobic polyethersulfone (PES) polymer were used to modify the highly sulfonated PEEK to investigate its effect towards electrolyte properties. These modifiers were selected because in the past studies compositing of PES and $TiO_2$ in sulfonated-based membrane had improved some of membrane properties especially the mechanical properties [28–35].

The modified sPEEK could also be preferred for synthesizing AEM electrolyte. Similar polymer backbone like that of the PEEK is also used in this research to achieve better compatibility between PEM and AEM electrolyte. To obtain AEM electrolyte, the quaternary ammonium group were crosslinked with sulfonic acid group of PEEK polymer to produce ionic crosslink quaternary ammonium poly(ether ether ketone) (cQAPEEK) membrane. It is expected that the crosslink reaction between acidic and alkaline ionic groups provide excellent anion conductivity and better mechanical properties which may benefit the BPM in terms of performance and durability. To the best of our knowledge, this is the first reported research that employs PEEK-based polymer as an electrolyte for BPM in BPMFC applications. This research postulated that the pairing between composite sPEEK with cQAPEEK will solve the water management issues in BPMFC and address the compatibility problem of electrolyte materials.

In this regard, selecting the best parameters of hot-press has not received any interest in open literature regarding the BPMFC applications and it was, therefore, considered in this research. This is because the hot press condition influences the degree of attachment between PEM and AEM electrolytes. A suitable hot-press condition is required to produce a good and fit contact between the PEM and AEM electrolytes to lower the electrical resistance at the adhesion area which can improve the ionic conductivity and preserve the durability of BPM. For these reasons, a Response Surface Methodology (RSM) approach was used to design the parameters of hot-press method. Thus, this research showed how the effect of employed modified PEEK electrolyte can enhance the BPM. It also tends to show how the influence of varied hot-press parameters on PEM/AEM can enhance the compatibility, degree of adhesion, cell electrochemical performance, self-humidification behaviour, and durability of electrolyte materials. Therefore, the questions to be answered in this research are:

(a) In what way will the properties of the developed BPM-based sPEEK improve the compatibility state of the PEM/AEM electrolytes junction layer?

(b) How will the method of RSM help to select the optimum hot-pressed parameters of the developed BPM at different operating pressures and temperatures?

(c) How will sPEEK/PES-cQAPEEK, sPEEK/$TiO_2$-cQAPEEK, and Nafion 117-cQAPEEK under different operating parameters of $H_2/O_2$ fuel cell operation impact the self-humidification behaviour, electrochemical performance, and durability?

## 1.3 Objectives of the Research

Based on the highlighted BPMFC issues in the problem statement, this study embarks on the following objectives:

(a) To develop, select, and compare the composition of organic PES and inorganic $TiO_2$ in highly sulfonated PEEK of PEM towards properties, electrochemical performance and compatibility state with crosslinked QAPEEK AEM synthesized at different chloromethylation time.

(b) To determine the optimum hot-pressed parameters at different operating pressures and temperatures for composing PEM with AEM for developing BPM and its effect toward BPM properties based on the RSM approach.

(c) To evaluate and compare the self-humidification behaviour, electrochemical performance, and durability of sPEEK/PES-QAPEEK, sPEEK/$TiO_2$-QAPEEK and Nafion 117-QAPEEK under the various operating parameters of $H_2/O_2$ fuel cell operation.

## 1.4 Scope of the Study

To achieve the research objectives, the following tasks were carried out:

(a) Preparing of PEM electrolyte:

i. Sulfonating the PEEK polymer with concentrated sulfuric acid at 53 °C for 3 h to produce sPEEK.

ii. Compositing the sPEEK with different PES content (5, 10, 15 and 20 wt.%) to produce composite sPEEK/PES membranes.

iii. Compositing the sPEEK with different $TiO_2$ content (0.5, 1.0, 1.5 and 2.0 wt.%) to produce composite sPEEK/$TiO_2$ membranes.

iv. Preparing the membranes based on the solution casting method.

(b) Preparing of AEM electrolyte:

i. Sulfonating the PEEK polymer with concentrated sulfuric acid at 50 °C for 3 h to produce sPEEK.

ii. The sPEEK particles were chloromethylized at different reaction times (48 h, 72 h and 96 h) followed by quaternization and alkalinisation to produce the cQAPEEK membrane.

iii. Preparing the membranes based on the solution casting method.

(c) Characterizing of PEM and AEM electrolytes:

i. The membranes were characterized according to structural, morphology, thermal, mechanical, physicochemical and electrochemical properties.

ii. The most promising sPEEK/PES, sPEEK/$TiO_2$ and cQAPEEK membranes were selected based on characterization results.

(d) Optimizing of hot-press parameter and development of BPM electrolyte:

i. The BPM was developed by hot-pressing the selected sPEEK/PES and sPEEK/$TiO_2$ with cQAPEEK membrane at the desired hot-press parameter such as time, temperature and pressure.

ii. The RSM approach was used to design the optimum hot-press parameter by varying the temperatures between 100 – 120 °C and gauge pressures between 1 – 3 tons/square inch, with constant heating time of 2 minutes.

(e) Evaluating the performance of developed PEM and BPM in $H_2/O_2$ fuel cell system:

i. The PEM and BPM electrode assemblies were prepared by composing membrane with Pt/C electrodes at 0.5 tons/square inch, 100 °C and 1 minute. The PEM and BPM electrode assemblies were tested under $H_2/O_2$ fuel cell operation.

ii. Operating temperature and relative humidity were varied from 40 – 80 °C and 10 – 100 % to determine the ability of PEM and BPM to withstand different temperatures and humidity levels.

iii. The electrochemical performance and durability test were compared between the developed electrolytes based on the current-voltage (I-V) polarization graph and voltage-time (V-T) graph.

## 1.5 Significance of the Study

To ensure clean, safe, and cost-effective energy sources as well as securing future energy production, new processes of fuel cells are being developed continuously through research to overcome the limitations of the commercial BPM fuel cell. One of such processes is the development of BPM-based sPEEK to enhance the compatibility of the PEM/AEM junction layer. To select the promising materials for BPM to enhance the ion migration, water formation at the junction layer, power output, and durability, this research alternatively fabricated PEEK polymer as the main backbone for PEM and AEM. The advantage of using the developed BPMFC in this research does not only produced the internal humidifying effect that substituted the external humidification system but it also cut the cost of applying BPMFC and reduced the cell-complexity. The replacement of the perfluorinated membrane, Nafion with hydrocarbon membrane improved the compatibility of membrane properties and benefited the working operation of the BPMFC and cut its operating cost. Also, the different fabrication techniques applied in this research helped to improve the BPMFC self-humidification behaviour and performance. The significance of these techniques in the improvement of BPMFC applications are promising. The application of a high sulfonation process of PEEK for PEM of BPM increased the $H^+$ migration. Also, the incorporation of the hydrophobic polymer, PES, and inorganic filler, $TiO_2$ in highly sulfonated PEEK improved the mechanical and thermal properties. Further, it reduced the swelling degree, hydrogen crossover, and promising proton conductivity of the modified PEEK. The application of the synthesized crosslinked AEM-PEEK also helped in the enhancement of its mechanical and thermal properties. The swelling degree and promising anion conductivity were also enhanced. Furthermore, by studying the optimization of hot-pressed parameters to obtain the optimum temperature and pressure, the adhesion contact of the PEM/AEM junction layer was improved remarkably.

## 1.6 Organization of the Thesis

**Chapter 1** contained a brief overview and background of BPMFC. It explained the aim and objectives of the research, the research problems, the scopes, gaps in the existing knowledge of the area of the research, and the significance of the research. In **Chapter 2,** a comprehensive review of previous studies connected to the theme of the current research was conducted. The discussion focused on the BPMFC working principle, cell components, past studies, and how it solves the water issues in the single layer membrane fuel cells. **Chapter 3** described extensively the design of the research and the procedures taken to achieve the objectives of the research. This chapter also contained the discussion of membrane synthesizing, preparation, and characterization processes. The design of experiments using the RSM approach of the Design-Expert Software to determine the optimum hot-press parameters were also presented. Meanwhile, the **Chapter 4** discussed the results obtained from the different analytical studies conducted and the outcomes of the performance evaluation of membranes. **Chapter 5** concluded the research with the summaries of the key findings and recommendations for future works.

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# LIST OF PUBLICATIONS

**Journal with Impact Factor**

1. **Daud, S. S.**, Norddin, M. A., Jaafar, J., & Sudirman, R. (2019). High degree sulfonated poly(ether ether ketone) blend with polyvinylidene fluoride as a potential proton-conducting membrane fuel cell. *High Performance Polymer*. 32(*1*), 103-115. (**Q3, IF: 1.568**)

2. **Daud, S. S.**, Norddin, M. A., Jaafar, J., Sudirman, R., Othman, M. H. D., & Ismail, A. F. (2020). Highly sulfonated poly (ether ether ketone) blend with hydrophobic poly ether sulfone as an alternative electrolyte for proton exchange membrane fuel cell. *Arabian Journal for Science and Engineering*. (**Q3, IF: 1.711 In Press**)

3. **Daud, S. S.**, Norddin, M. A., Jaafar, J., & Sudirman, R. (2021). Development of sPEEK/PES-cQAPEEK bipolar membrane electrolyte via hot-press approach for hydrogen/oxygen fuel cell. *International Journal of Energy Research*. (**Q1, IF: 3.741 Accepted**)

**Indexed Journal**

1. **Daud, S. S.**, Jaafar, J., Norddin, M. A., & Sudirman, R. (2019). Poly(ether ether ketone) based anion exchange membrane for solid alkaline fuel cell: A review. *Journal of Membrane Science and Research*. 5(*3*), 205-215. (**Indexed by Scopus**)

**Indexed and Non-Indexed Conference Proceedings**

1. **Daud, S. S.,** Norddin, M. A., Jaafar, J., & Sudirman, R. (2018). A mini review of bipolar membrane as a self-humidifier for proton exchange membrane fuel cell. In 7th International graduate conference on engineering, science and humanities *(IGCESH*), Universiti Teknologi Malaysia, Johor. 13-15 August. (Abstract, **Indexed by Scopus**)

2. **Daud., S. S.,** Norddin, M. A., Jaafar, J., & Sudirman, R. (2018). A blend membrane based on high degree sulfonation poly(ether ether ketone) and poly(vinylidene fluoride) for fuel cells. In National Congress on Membrane Technology, Pulai Spring Resort, Johor. 30-31 October. (Abstract, **Non-indexed**)

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4. **Daud, S. S.,** Norddin, M. A., Jaafar, J., & Sudirman, R. (2019). The effect of material on bipolar membrane fuel cell performance: A review. In Energy security and chemical engineering congress (*ESCHE*), Parkroyal Resort, Pulau Pinang. 17-19 July. (**Indexed by Scopus**)