HIGHLY SULFONATED POLYPHENYLSULFONE NANOCOMPOSITE MEMBRANES FOR IMPROVING PROTON EXCHANGE MEMBRANE FUEL CELL PERFORMANCE

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ABSTRACT

Fuel cell has become a rising technology that has widely been explored due to its promising efficient energy conversions. Currently, proton exchange membrane fuel cell (PEMFC) is the most studied fuel cell systems because of the simple structure and wide application range. Recent research has been devoted to develop a proton exchange membrane (PEM) using sulfonated aromatic polymers with high proton conductivity and good durability. The state-of-the-art of PEM-based polyphenylsufone (PPSU), which has excellent thermal stability and appropriate mechanical strength with high proton conductivity and increases along with the degree of sulfonation have been widely explored. Unfortunately, increasing the degree of sulfonation always results in swelling and physical expansion of the materials leading to mechanical failures. To keep the benefit of having high proton conductivity of high sulfonation degree, hybrid/blend membranes and applying crosslinking step become alternatives to solve the critical issue of the mechanical failure. Therefore, this research aimed to develop PEM using highly sulfonated polyphenylsulfone (SPPSU) membrane, incorporating different types and structures of inorganic fillers for PEMFC applications. PPSU was directly sulfonated using sulphuric acid and had achieved the desired degree of sulfonation, which was DS~2. This SPPSU was highly soluble in water upon heating at 80 ºC. The thermal crosslinking process was applied up to 180 ºC to improve the mechanical properties of the SPPSU membrane, and it is suggested that heat promoted the crosslinking between the SPPSU polymer matrix. The proton conductivity achieved about 1.12×10^{-2} S/cm, which is still lower than the requirements for the desired proton conductivity values for PEMFC applications (10×10^{-2} S/cm). Consequently, in this study, the different types and structures of fillers which were carbon nanodots (CND), sulfonated polyhedral silsesquioxane (SPOSS), and imogolite (Im) were incorporated into the SPPSU polymer matrix. All three fillers exhibited proton conductivity of about 3 to 4 fold higher compared to the SPPSU membrane. SPPSU-2% CND, SPPSU-2% SPOSS, and SPPSU-1% Im nanocomposite membrane were chosen for further performance testing and membrane durability. The SPPSU and composite SPPSU membrane exhibited excellent dimensional stability after prolonged exposure to water for 720 h. At 80 °C under fully hydrated conditions, the maximum power density of the SPPSU membrane was 81.05 mW/cm^2. SPPSU with fillers showed a significantly improved performance compared to the SPPSU membrane. The highest maximum power density belongs to the SPPSU-2% SPOSS nanocomposite membrane that reached up to 131.53 mW/cm^2. This is followed by SPPSU-2% CND, Nafion 117 and SPPSU-1% Im each with 118.75 mW/cm^2, 111.76 mW/cm^2, and 89.76 mW/cm^2, respectively. Besides, SPPSU and composite SPPSU showed stable potential voltage during the 8 h operation. It is interesting to state that the membrane electrode assembly using the SPPSU-2% SPOSS nanocomposite membrane showed excellent electrochemical properties under operating conditions of 80 °C and 100% relative humidity, which is comparable to commercial Nafion 117. It can be deduced that the incorporation of CND, SPOSS, and Im into SPPSU has improved not only the membrane properties but also the PEMFC performance and membrane durability.

ABSTRAK

Sel bahan api telah menjadi teknologi yang semakin terkenal dan diterokai secara meluas kerana pertukaran tenaganya yang cekap. Pada masa ini, sel bahan api membran pertukaran proton (PEMFC) merupakan sistem sel bahan api yang paling kerap dikaji disebabkan struktur yang mudah dan aplikasi yang meluas. Penyelidikan kini tertumpu kepada membangunkan membran pertukaran proton (PEM) menggunakan polimer aromatik bersulfona dengan proton konduktiviti dan ketahanan yang tinggi. Keadaan seni PEM yang berasaskan polifenilsulfona (PPSU), mempunyai kestabilan terma yang sangat baik dan kekuatan mekanikal dengan kekonduksian proton tinggi yang meningkat mengikut tahap sulfonasi telah dikaji dengan meluas. Malangnya, peningkatan tahap sulfonasi mengakibatkan pembengkakan dan pengembangan bahan secara fizikal yang menyebabkan kegagalan mekanikal. Untuk mengekalkan faedah memiliki kekonduksian proton yang tinggi oleh tahap sulfonasi yang tinggi, membran hibrid/campuran dan proses pemaut silang menjadi alternatif untuk menyelesaikan masalah utama kegagalan mekanikal. Oleh itu, penyelidikan ini bertujuan untuk membangunkan PEM menggunakan membran polifenilsulfona dengan tahap sulfonasi yang tinggi (SPPSU) serta menggabungkan pelbagai jenis dan struktur bahan tambahan bukan organik untuk aplikasi PEMFC. PPSU disulfonasi menggunakan asid sulfurik dan mencapai tahap sulfonasi yang diinginkan iaitu DS~2. SPPSU sangat larut dalam air semasa pemanasan pada suhu 80 ºC. Proses pemaut silang terma dijalankan hingga 180 ºC untuk meningkatkan sifat mekanikal membran SPPSU, dan didapati haba menggalakkan pemaut silang di antara matrik polimer SPPSU. Kekonduksian proton mencapai 1.12×10^{-2} S/m di mana nilainya masih lagi lebih rendah berbanding nilai kekonduksian proton yang diinginkan untuk aplikasi PEMFC (10×10^{-2} S/cm). Oleh itu, dalam kajian ini, pelbagai jenis dan struktur bahan bukan organik seperti nanopartikel karbon (CND), sulfonasi polihedral silsesquioksana (SPOSS), dan imogolit (Im) diadunkan ke dalam matriks polimer SPPSU. Ketiga-tiga bahan ini menunjukkan kekonduksian proton sekitar 3 hingga 4 kali ganda lebih tinggi berbanding dengan membran SPPSU. Membran komposit SPPSU-2% CND, SPPSU-2% SPOSS, dan SPPSU-1% Im dipilih untuk ujian prestasi dan ketahanan membran selanjutnya. SPPSU dan membran komposit SPPSU mempamerkan kestabilan dimensi yang sangat baik di dalam rendaman air selama 720 jam. Pada 80 °C di dalam keadaan terhidrat sepenuhnya, ketumpatan kuasa maksimum membran SPPSU ialah 81.05 mW/cm^2. Membran komposit SPPSU menunjukkan prestasi yang lebih baik berbanding dengan membran SPPSU. Ketumpatan kuasa maksimum tertinggi adalah membran komposit SPPSU-2% SPOSS yang mencapai sehingga 131.53 mW/cm^2. Ini diikuti oleh SPPSU-2% CND, Nafion 117 dan SPPSU-1% Im masing-masing dengan 118.75 mW/cm^2, 111.76 mW/cm^2, dan 89.76 mW/cm^2. Selain itu, membran SPPSU dan membran SPPSU dengan pengisi menunjukkan voltan yang stabil semasa 8 jam operasi. Adalah menarik untuk dinyatakan bahawa pemasangan himpunan elektrod membran menggunakan membran komposit SPPSU-2% SPOSS menunjukkan sifat elektrokimia yang sangat baik pada 80 °C dan 100% kelembapan relatif, setanding dengan membran komersial Nafion 117. Dapat disimpulkan bahawa penggabungan CND, SPOSS, dan Im ke dalam SPPSU telah menambahbaik bukan sahaja sifat-sifat membran malah meningkatkan prestasi PEMFC dan ketahanan membran.

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LIST OF ABBREVIATIONS

A-POSS	-	Acrylo-Polyhedral Oligomeric Silsesquioxane
CND	-	Carbon Nanodots
DMSO	-	Dimethyl Sulfoxide
Fe_2SO_3	-	Ferrous (II) Sulfate
GDL	-	Gas Diffusion Layer
GO-PSBMA	-	Graphe Oxide-Poly(Sulfobetaine Methacrylate)
HPEI	-	Hyperbranched Polyehylenimine
H_2O_2	-	Hydrogen Peroxide
Im	-	Imogolite
KOH	-	Potassium Hydroxide
K_2CO_3	-	Potassium Carbonate
MEA	-	Membrane Electrode Assembly
$MgSO_4$	-	Magnesium Sulfate
MWCNT	-	Multi Walled Carbon Nanotube
Na_2SO_4	-	Sodium Sulfate
NMP	-	N-Methyl Pyrrolidone
OCV	-	Open Circuit Voltage
PAES	-	Poly(Arylene Ether Sulfone)
PBI	-	Polybenzimidazole
PFSA	-	Perfluorosulfonic Acid
PGM	-	Platinum Group Metal
POSS	-	Poly Octahedral Silsesquioxane
PPO	-	Poly (Phenylene Oxide)
PPOs	-	Poly(2,6-Dimethyl-1,4-Phenylene Oxide)s
PTFE	-	Polytetrafluoroethylene
PVdF-co-HFP	-	Poly(Vinylidene Fluoride-co-Hexafluoropropylene)
RH	-	Relative Humidity
SiO_2	-	Silicon Dioxide/Silica
SPEEK	-	Sulfonated Poly(Ether Ether Ketone)
SPOSS	-	Sulfonated Poly Octahedral Silsesquioxane

SnO_2	-	Tin (IV) Oxide
SS	-	Sulfonated Styrene
$STiO_2$	-	Sulfonated Titanium Dioxide
T_g	-	Glass Transition Temperature
UV	-	Ultraviolet
ZIF-8	-	Zeolitic Imidazolate Framework
ZnO	-	Zinc Oxide
ZrO_2	-	Zirconium Dioxide/Zirconia

LIST OF SYMBOLS

c	-	Molar of NaOH solution for IEC titration (mol)
DS	-	Sulfonation degree
F_w	-	Formula weight
f_t	-	Maximum load (N)
l_w	-	Wet length (cm)
l_d	-	Dry length (cm)
M_n	-	Number average molecular weight
M_w	-	Average molecular weight (g/mol)
W_w	-	Wet weight (g)
W_d	-	Dry weight (g)
R	-	Membrane resistance (Ω)
S	-	Area of electrode (cm2)
t	-	Membrane thickness (cm)
t_w	-	Wet Thickness (µm)
t_d	-	Dry Thickness (µm)
$T_{d5\%}$	-	Temperature at 5% weight loss (ºC)
v	-	Volume of neutralised NaOH (mL)
w	-	Membrane sample width (mm)
λ	-	Water content
σ	-	Proton conductivity (S/cm)
σ_t	-	Tensile strength (MPa)

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Access to energy is a crucial pillar for human wellbeing, economic development, and poverty alleviation. Ensuring everyone has sufficient energy access is an ongoing and pressing challenge for global growth. The world consumes about 154,000 terawatt-hours of primary energy in 2017, which is more than 27 times from 1800 (5650 terawatt-hours). Typically, higher energy consumers come from higher country average income (Ritchie and Roser, 2019). Historical and current energy systems are dominated by fossil fuels such as coal, oil, and gas, which produce carbon dioxide and other greenhouse gases that become the fundamental driver of global climate change. Nowadays, the world has transitioned from an energy system dominated by fossil fuels to a low-carbon emission system. Renewable energy technologies, including fuel cells, bioenergy, hydropower, the solar, wind, and geothermal energy, are options for replacing fossil fuels.

Nowadays, fuel cells have become a rising technology that has been widely explored due to promising clean and efficient energy conversions. Despite the modern world technology, fuel cells have been known to science for more than 150 years (Coralli *et al.*, 2019). Though generally considered a curiosity in the 1800s, fuel cells became the subject of intense research and development during the 1900s (Kirubakaran *et al.*, 2009). Fuel cells offer several advantages over conventional power sources, including reduced dependence on fossil fuels, long useful life, high efficiency, relatively safe, essential zero toxicity, minimal maintenance costs, and free carbon emission (Giorgi and Leccese, 2013). This cell system also prepared a clean, quiet, and highly efficient process in converting the fuel to energy via an electrochemical process.

The essential purpose of fuel cell applications is to produce an electrical current directed outside the cell to do work, such as powering an electric motor or illuminating a light bulb. The chemical reactions that produce the current are the key to how a fuel cell works (Chaurasia *et al.,* 2003). Proton exchange membrane fuel cell (PEMFC) comprises advantages over other types of fuel cells. PEMFC operates at a lower temperature, light, and compact, making it ideal for applications such as cars (Sethuraman *et al.,* 2009). Proton exchange membrane (PEM) is a heart component of PEMFC that strongly influences the cell performance. PEM is a semipermeable membrane generally made from ionomers and designed to conduct protons through it. The PEM's essential function in the fuel cell system is as the reactant separator and protons transportation medium while blocking an electronic pathway through the membrane (Kyu and Nazir, 2013).

Generally, there are several required properties of the PEM to be compatible with the fuel cell operation. PEM should be chemically stable as it is acting in strongly acidic medium and high durability along with fuel cell operation (Espiritu *et al.*, 2016). Ideally, the proton conductivity of the developed PEM that meets the PEMFC application requirement is about 0.1 S/cm (Sun *et al.,* 2019). Finally, all these properties should remain unchanged at the working temperature to avoid any structural change during the chemical reactions, and be thermally stable (Sahu *et al.,* 2014). The-state-of-the-art of the PEM fuel cells-based perfluorosulfonic acid ionomers such as Nafion, which shows good durability and high proton conductivity, is recognized as the most used PEM in the market. On the contrary, high cost, lower proton conductivity, rising temperature, and water management issues have directed research towards exploring new materials as an alternative to Nafion (Rosli *et al.,* 2017).

There are a growing number of studies emphasize on developing the proton exchange membrane implementing engineering thermoplastic polymers such as poly (ether ether ketone) (Xing *et al.,* 2004; He *et al.,* 2016; Lyu *et al.,* 2018), poly(phenylene) (Jo *et al.,* 2016), poly (phenylene oxide) (Petreanu *et al.,* 2012), polybenzimidazole (Singha *et al.,* 2016; Devrim *et al.,* 2016), and polyethersulfone (Assumma *et al.,* 2014; Muthumeenal *et al.,* 2016). Among them, polysulfone (Kim *et al.,* 2018a) and polyphenylsulfone (Schuster *et al.,* 2009; Park *et al.,* 2016a) with excellent properties have been widely studied. The main aim of developing the PEM with regards to new membrane materials is to synthesize PEM showing high water uptake, high proton conductivity, high chemical/thermal stability, high chemical resistance, and low cost (Lulianelli and Basile, 2012). Nevertheless, there are still unresolved practical application issues of these membranes due to low proton conductivity under low humidity conditions compared to Nafion.

Sulfonic acid groups are the most common functional groups used for the proton exchange membrane produced by the process of sulfonation using a sulfonating agent (Huang *et al.*, 2016). Many studies have been reported using sulfonic acid as an anionic functional group of the polymer backbone to serve as proton carriers in the proton exchange membrane (Chen *et al.*, 2015). Adding the sulfonic acid group as a charge carrier in the proton exchange membrane enhances the sulfonic acid group density that contributed to the high impact of the membrane's proton conductivity. As the PEM proton conductivity is strongly dependent on the sulfonic acid group's charge carrier density, a higher degree of sulfonation will give better proton conductivity values (Yang *et al.*, 2017).

Various studies in the open literature confirmed that sulfonated polymer membranes show practical application as PEM with good membrane stability and proton conductivity for fuel cell applications (Higashihara *et al.*, 2009; Lee *et al.*, 2019). Nevertheless, depending on the variation of the sulfonation degree, high sulfonation degree results in excellent proton conductivity but mechanical properties could deteriorate progressively. To keep the benefit of having high proton conductivity with a high sulfonation degree, the hybrid/blend membranes and applying the crosslinking step become an alternative to solve the vital issue of mechanical failure of the membrane (Krishnan *et al.*, 2014). Variant types and structure of inorganic fillers have been explored to develop the hybrid/blend PEM by means to improve the membrane stability as well as keep maintaining or even enhancing the proton conductivity of the PEM (Beydaghi *et al.*, 2014; Wang *et al.*, 2018). It is beneficial to study the effect of different types and structures of inorganic fillers incorporated into highly sulfonated polymer on the PEM properties.

1.2 Problem Statement

The Nafion-based polymer electrolyte membrane by DuPont is the most common commercially available PEM in the market with high hydrolytic and oxidative stability and excellent proton conductivity (Yin *et al.,* 2018). However, Nafion has a significant drawback in terms of high operation temperature (>100 °C), leading to a relative decrease in the conductivity values. Operating temperature below 80 °C is too low for cogeneration, and that the PEM must be water-saturated (Dodds *et al.,* 2015). As a result, new anhydrous proton conductor membranes that can withstand under this condition are actively studied to develop suitable PEM with excellent cell performance. Recent research has been devoted to developing PEM using engineering thermoplastic materials as an alternative to Nafion, which would be less expensive and counter the drawbacks of perfluorinated membranes (Meemuk and Chirachanchai, 2018).

Polyphenylsulfone (PPSU) represents a sulfone polymer group that has been widely explored as an alternative to proton-conducting membrane instead of expensive perfluorinated membranes. PPSU has excellent thermal stability and appropriate mechanical strength with high proton conductivity, increasing the degree of sulfonation (Kim and Ghil, 2016). Unfortunately, there is a restriction of PPSU membrane to be applied in fuel cell application as the proton conductivity of PPSU polymers is generally lower than perfluoro ionomers based PEM membranes. The interconnected hydrophilic channels of PPSU polymers are not well developed as perfluoro-ionomers PEMs (Park *et al.,* 2016). To have a newly generated PPSU membrane that is comparable or even better than the Nafion membrane, PPSU has been modified for having a high sulfonation degree concerning the second degree of substitution. Unfortunately, increasing the degree of sulfonation always resulting in swelling and the physical expansion of the materials leading to mechanical failures (Yee *et al.,* 2013). It is regrettable to sacrifice the potential to have an excellent proton conductivity due to highly sulfonated polymers that will tremendously reduce the membrane mechanical stability.

The mechanical weaknesses of the highly sulfonated PPSU, abbreviated as SPPSU, have initiated several attempts to prepare a more stable proton-conducting membrane. Various ways have been developed to improve the poor dimensional stability and mechanical properties of the high sulfonated membrane (Liu *et al.,* 2016a; Wu *et al.*, 2019). Recently, the thermal crosslinking process has been studied to improve the poor dimensional stability and mechanical properties of the SPPSU through bridging links to the reactive sulfonic acid functions without deterioration of proton conductivity. Wu *et al.* (2010) claimed that polymer with SO_3H groups could crosslink with itself during heating without other cross-linkers. After heat treatment, the crosslinking occurs through the sulfonic acid groups, and sulfone is formed to connect two rings. Unfortunately, in the proton exchange membrane, the crosslinking of SPPSU results in reducing the proton conductivity values despite having larger active sulfonic acid groups for proton transportation. Thus efforts have been devoted to developing excellent SPPSU membranes for PEMFC.

The doping or hybridizing of the polymer matrix with inorganic nanomaterials is an efficient approach for improving the proton conductivity of the SPPSU membrane. The incorporation of inorganic nanomaterials influences the organic phase's properties towards proton conductivity, membrane stability, improved water retention capacity, and improved mechanical strength by increasing the transport pathway tortuousness (Chen *et al.*, 2012). The intrinsic properties of the inorganic nanomaterials as a filler, such as size, type, structure, and interactions with the polymer matrix, can significantly affect the resultant matrix (Balasubramaniam and Ramesh, 2018; Liu *et al.,* 2019a). It has been demonstrated that the hydrophilic inorganic fillers within the membrane play an important role because they can promote water retention ability of the composite membrane and facilitate more channels for proton transfer in the SPPSU polymer matrix (Oh *et al.*, 2019). In this study, three different types and structures of inorganic fillers are incorporated into the SPPSU matrix. They are carbon nanoparticle (carbon nanodot, CND), silica nanocage (polyhedral silsesquioxane, POSS), and inorganic clay nanotubular (imogolite, Im). These fillers were chosen based on its different molecular structure to study the effect of incorporating different fillers structure towards facilitating proton transportation within the SPPSU molecular structure and improving the membrane durability.

Therefore, the attempts were made to introduce the SPPSU polymers as a polymer backbone for newly developed PEM as a replacement to the Nafion membrane. The crosslinking effects on the mechanical and thermal properties of the SPPSU membrane were studied. Moreover, the effect of incorporating different types and structure of inorganic fillers in newly developed PEM based crosslink SPPSU membrane was reported. The properties of the SPPSU nanocomposite membrane as PEM towards fuel cell application was also presented. It is believed that the different types and structures of inorganic fillers carried their advantages towards the improvements of the crosslink SPPSU membrane properties for PEMFC application.

1.3 Objective of The Study

This project investigates the efficiency of the PEM using the prepared nanocomposite membrane consisting of SPPSU comprised of different type and structure of inorganic nanofillers for proton exchange membrane fuel cell application (PEMFC). The specific objectives of these studies are:

1) To perform SPPSU membrane by applying the thermal crosslinking step and to investigate the effect of thermal croslinking temperature on the kinetic properties and proton conductivity values of the SPPSU membranes.

2) To investigate the effect of different type and structure of inorganic fillers loading on the kinetic properties, mechanical properties, and proton conductivity of the SPPSU nanocomposite membrane.
3) To determine the power density and potential voltage performances of the SPPSU nanocomposite membranes using single-cell test PEMFC under different temperatures and relative humidity conditions.

4) To evaluate the long term stability behavior of the SPPSU nanocomposite membranes in terms of voltage, oxidative, and dimensional stability as a function of time.

1.4 Scope of The Study

To achieve the above mentioned objective of the research, the following scopes are outlined:

1) Preparing the SPPSU polymers by directly sulfonation with H_2SO_4. The degree of sulfonation, ion exchange capacity, and molecular weight of SPPSU polymer were evaluated.

2) Studying the effect of heat treatment (80 °C to 180 °C) on the development of SPPSU as a proton exchange membrane. This range was selected based on the preliminary results obtained during the early stages of the study and followed by the previous study reported before (Kim and Ghil, 2016). Morphological structure, mechanical strength, thermal stabilities, and kinetic parameters of the SPPSU membrane were studied using SEM, FTIR, tensile test, TGA, IEC, conductivity, water uptake, and swelling ratio.

3) Comparing the proton conductivity of the SPPSU membrane with commercial Nafion 212 membranes under various temperature (40 °C, 60 °C, 80 °C, 100 °C and 120 °C) and relative humidity conditions (40%, 60%, 80%, and 90%).

4) Preparing the SPPSU nanocomposite dope solutions by incorporating various loadings from different types and structures of inorganic fillers. 0.5 wt.% to 10 wt.% of carbon nanodots (CND), sulfonated polyhedral silsesquioxane (SPOSS), and imogolite (Im) was blending with SPPSU/DMSO dope solutions.

5) Developing the SPPSU nanocomposite membrane by slow evaporation technique. The membrane was subjected to thermal treatment and underwent the post-activation process by immersing in different solutions. The effect of loading was evaluated by characterizing the physicochemical properties of the nanocomposite membrane using SEM, FTIR, TGA, water uptake, swelling ratio, IEC, tensile strength, and proton conductivity.

6) Selecting the optimum loading for each filler in the SPPSU nanocomposite membranes. The oxidative stability of the optimum membrane from each filler was compared with the SPPSU membrane by immersing in Fenton Reagent (5 wt.% H_2O_2 with 2 ppm Fe_2SO_3) at 80 °C for 16 h. The dimensional stability of the optimized membrane was subjected under prolonged time in the water at R.T for 720 h.

7) Evaluating the power density and potential voltage by single-cell test performance of SPPSU and SPPSU nanocomposite membrane. The performance of the SPPSU nanocomposite membrane was evaluated using an optimum loading for each filler. The performance was tested under PEMFC conditions by preparing the membrane electrode assembly (MEA). The performance was compared with the commercial Nafion 117.

8) Studying the voltage stability of the SPPSU and optimize SPPSU nanocomposite membrane under 0.1 A applied current from 0 h to 8 h.

1.5 Significance of The Study

Fuel cell applications are currently seen as important energy conversion devices with considerable potential in the Malaysian future energy structure. The fuel cell market in Malaysia is still quite small, and substantial growth to the domestic market must be developed to ensure a significant contribution to Malaysia's energy by 2050. Currently, the research towards the development of all parts of the fuel cell, especially the PEM that can provide high proton conductivity and good durability, is actively being studied. This study was expected to better understand the PEM development for fuel cell applications using highly sulfonated PPSU incorporating different types and structures of an inorganic filler membrane as a replacement to the commercially available Nafion membrane. The primary outcome of this study will benefit the scientific community in the sense of filling in the knowledge gap in multiple fields that encompass the SPPSU nanocomposite membrane as PEM. Considering that the proton conductivity and durability of the SPPSU nanocomposite membrane is comparable to the commercial Nafion membrane, could diversify their potential in fuel cell applications. The resourceful approach, which combined the unique properties of the highly sulfonated polymers with different structures of the inorganic filler is a great potential to replace the commercial Nafion membrane in PEMFC applications.

1.6 Thesis Organization

This thesis consists of 5 chapters. Chapter 1 outlines the research background and problem statements that lead to this study. The research background, problem statement, objective, scope, and significance of this study were highlighted to emphasize this study. Next, chapter 2 described the general information and the previous research that is related to this study. Discussion on the fuel cell working principle, the desired properties of the PEM, the structure of inorganic fillers, and materials used to develop PEM, followed by crosslinking types and techniques, were provided in this chapter. Next, the detailed experimental method and membrane characterizations were discussed in chapter 3. The performance and durability test of the developed membranes were also included in this chapter. Meanwhile, chapter 4 presents all the results and discussion on the membrane properties, single-cell test performance, and membrane stability. Lastly, chapter 5 presents the conclusion and recommendations for future work.

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