# FABRICATION OF NANOCOMPOSITE MEMBRANE VIA COMBINED ELECTROSPINNING AND CASTING TECHNIQUE FOR DIRECT METHANOL FUEL CELL

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***My understanding husband***
Mohd Helmi Bin Abdullah
*Thanks for understanding and always support me in no matter what conditions*

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# ABSTRACT

In the past decades, the emerging of nanotechnology has brought to the introduction of electrospinning process in polymer electrolyte nanocomposite membrane due to its specialty in providing a very large specific surface area which contributed by a small size of fillers and an outstanding nanovoids interconnectivity between the fillers. The objective of this study was to fabricate and characterize sulfonated poly (ether ether ketone) (SPEEK) nanocomposite membrane consist of electrospun Cloisite15A® (e-spun CL) for direct methanol fuel cell (DMFC) applications. Poly (ether ether ketone) polymer was sulfonated by sulfuric acid to obtain degree of sulfonation of 63%. SPEEK63/e-spun CL nanofibers were fabricated via electrospinning process in which SPEEK63 was used as carrier polymer while SPEEK63/e-spun CL nanocomposite membrane was obtained by casting method. Characterizations on physical, morphological and thermal properties of SPEEK63/e-spun CL were conducted and compared to SPEEK nanocomposite membrane with 2.5wt.% Cloisite15A® and 5.0wt.% triaminopyrimidine (SPEEK63/2.5CL/5.0TAP). Scanning electron microscopy (SEM) showed that Cloisite15A® was well electrospun with the nanofiber diameter ranging from 62.5 to 375 nm. Moreover, field emission scanning electron microscopy (FESEM) revealed that Cloisite15A® particles at nanometer range were uniformly distributed and 66% smaller than in SPEEK63/2.5CL/5.0TAP. In addition, x-ray diffraction proved that the dispersion state of Cloisite15A® fell into intercalated phase. A very small amount of Cloisite15A® (0.05wt.%) in SPEEK63/e-spun CL had successfully enhanced the proton conductivity up to 50% whereas, methanol permeability value was unfortunately 27 times higher than SPEEK63/2.5CL/5.0TAP. Proton conductivity and methanol permeability of SPEEK63/e-spun CL were 24.49 x $10^{-3}$ $Scm^{-1}$ and 3.74 x $10^{-7}$ $cms^{-1}$, respectively. Even though this study contributed to a selectivity of 95% lower than SPEEK63/2.5CL/5.0TAP, the electrospinning process had shown a promising technique to further reduce the original size of Cloisite15A® particles from mixed size (µm and nm) to nanometer size as well as by fine tuning the dispersion of Cloisite15A® can enhance SPEEK63/e-spun CL performance in DMFC applications.

# ABSTRAK

Pada dekad yang lalu, kemunculan teknologi nano telah membawa kepada pengenalan proses elektro pemintalan dalam membran polimer elekroli nanokomposit disebabkan oleh keistimewaannya dalam menyediakan luas permukaan spesifik dan yang kasar disebabkan oleh pengisi yang bersaiz kecil dan kesalinghubungan rongga nano yang cemerlang antara pengisi. Objektif kajian ini adalah untuk menghasilkan dan mencirikan membran nano komposit poli (eter eter keton) tersulfonasi (SPEEK) yang terdiri daripada Cloisite15A® terelektropintal (e-spun CL) untuk aplikasi bahan api metanol terus (DMFC). Polimer poli (eter eter keton) telah disulfonasi dengan asid sulfurik untuk mendapatkan darjah sulfonasi 63%. Gentian nano SPEEK63/e-spun CL telah dihasilkan melalui proses elektro pemintalan yang mana SPEEK63 digunakan sebagai polimer pembawa manakala membran nanokomposit SPEEK63/e-spun CL diperoleh melalui kaedah penuangan. Pencirian sifat-sifat fizikal, morfologi dan kestabilan terma dijalankan ke atas SPEEK63/e-spun CL dan dibandingkan dengan membran nanokomposit SPEEK63 dengan 2.5% jisim Cloisite15A® dan 5.0% jisim triaminopyrimidina (SPEEK63/2.5CL/5.0TAP). Mikroskop imbasan elektron (SEM) menunjukkan Cloisite15A® telah dielektropintal dengan baik dan diameter gentian dalam lingkungan 62.5 hingga 375 nm. Mikroskop medan pancaran imbasan elektron (FESEM) menunjukkan taburan zarah Cloisite15A® adalah seragam pada julat nanometer dan 66% lebih kecil daripada yang dalam SPEEK63/2.5CL/5.0TAP. Pembelauan x-ray membuktikan bahawa penyebaran Cloisite15A® jatuh ke fasa interkalasi. Cloisite15A® yang sangat sedikit (0.05% jisim) dalam SPEEK63/e-spun CL telah berjaya meningkatkan kekonduksian proton sehingga 50%, manakala, kebolehtelapan metanol adalah 27 kali lebih tinggi berbanding SPEEK63/2.5CL/5.0TAP. Kekonduksian proton dan kebolehtelapan metanol SPEEK63/e-spun CL menunjukkan 24.49 x $10^{-3}$ $Scm^{-1}$ dan 3.74 x $10^{-7}$ $cms^{-1}$ masing-masing. Walaupun kajian ini menyebabkan kememilihan 95% lebih rendah berbanding SPEEK63/2.5CL/5.0TAP, telah menunjukkan bahawa proses pemintalan elektro adalah teknik yang berpotensi dalam mengurangkan saiz asal zarah Cloisite15A® daripada campuran saiz (μm dan nm) kepada saiz nanometer dan juga dengan keadaan penyebaran Cloisite15A® boleh meningkatkan prestasi SPEEK63/e-spun CL dalam aplikasi DMFC.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| CLTE | - | Coefficient of Linear Thermal Expansion |
| DMAc | - | Dimethyl Acetamide |
| DMF | - | Dimethyl Formamide |
| DS | - | Degree of Sulfonation |
| EDX | - | Energy Dispersive X-ray |
| e-spun CL | - | Electrospun Cloisite15A® |
| FESEM | - | Field Emission Scanning Electron Microscopy |
| $H_2SO_4$ | - | Sulphuric Acid |
| $H^1NMR$ | - | Hydrogen Nuclear Magnetic Resonance |
| HDT | - | Heat Distortion Temperature |
| PEEK | - | Poly(ether ether ketone) |
| PEM | - | Polymer Electrolyte Membrane/Proton Electrolyte Membrane |
| PVDF | - | Polyvinylidene Fluoride |
| R&D | - | Research and Development |
| SEM | - | Scanning Electron Microscopy |
| SPEEK,SP | - | Sulfonated Poly(ether ether ketone) |
| Si | - | Silica |
| TGA | - | Thermogravimetric Analyzer |
| XRD | - | X-ray Diffraction |

# LIST OF SYMBOL

| | | |
|---|---|---|
| $°C$ | - | Degree Celsius |
| $\sigma$ | - | Proton Conductivity |
| $P$ | - | Methanol Permeability |
| $R$ | - | Resistance |
| $\Theta$ | - | Angle of Maximum Point of The First Peak (Lowest ϴ) In The Spectra |
| $\lambda$ | - | Wavelength |
| $g$ | - | Gram |
| $cm^3$ | - | Centimeter Cubic |
| $mg$ | - | Miligram |
| $wt.\%$ | - | Percentage Weight |
| $d$- | - | Spacing Between The Layers of The Clay |
| $L$ | - | Thickness of Hydrate Membrane |
| $D$ | - | Methanol Diffusivity |
| $t_o$ | - | Time Lag |
| $V_B$ | - | Volume of Water Compartment |
| $A$ | - | Volume Cross Section Area of Membrane |
| $\Phi$ | - | Selectivity of The Membrane |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

The current scenario of uncertainty in oil price increase is beyond any governments' control. An uprising problem regarding on these issues which eventually contributes to increment of household cost as well as electricity and transportation cost. The reason for uncertainty in fossil fuels price is mainly due to the rapid depletion which will increase the energy price (Wong, 2006). In order to avoid this to happen, the alternative choice should be made available. Until this moment, the research and development of renewable energy are increasing yearly. Among several well-known types of renewable energy which are solar energy, wind energy, geothermal energy, bioenergy, hydropower and ocean energy. Meanwhile, fuel cell is also gaining attention as promising alternative in providing energy sources.

The research and development (R&D) on proton electrolyte membrane (PEM) is foreseen to generate more significant contribution as compared to other parts in fuel cell system. PEM is always expressed as the "nerve" or "heart" of a fuel cell system as it plays the most crucial task in allowing and repelling the protons and electrons, respectively. These characteristics will determine the efficiency of a fuel cell as a whole, directly. As in directly, an efficient fuel cell system could provide a beneficial impact on environmental as well as economic views.

Layered silicates-polymer nanocomposite is a new polymer electrolyte membrane (PEM) that lately concerned a great deal of interest due to the improvement in the mechanical, thermal and barrier properties of the pure polymer (Tien and Wei, 2001). Compared to the corresponding pure polymer membranes as well as commercial Nafion® membranes, many polymer-inorganic nanocomposite membranes are shown much lower fuel permeability along with similar or improved proton conductivities due to the nano-dispersion of layered silicates throughout polymer matrix (Wang and Dong, 2007).

The combination of the advantages from the base materials: i.e., the flexibility and process ability of polymer, as well as the selectivity and thermal stability of the inorganic fillers are contributed to the aforementioned properties. By adding the inorganic nanofillers, it may affect the membrane cell in two ways: 1) the uniform nanosized distribution of inorganic filler particles produces a winding diffusion pathway which can hinder the fuel to transfer through the nanocomposite membrane, and 2) the complete morphological structure allows more cations to mobile and available for conduction (Wang and Dong, 2007). Furthermore, the smaller the particles size, the larger surface area of dispersed nanosized particles within polymer matrix which can decrease the degree of crystallinity of polymer segments and yet will contribute to the larger ionic mobility that eventually increased the proton conduction (Croce *et al*., 1998; Golodnitsky *et al*., 2002).

Electrospinning seem to be a good solution in providing a nanosized particles as well as altering the structure of polymer-inorganic electrolyte membrane due to the versatility possessed by electrospinning. The electrospinning process are favorable to be used in developing a highly porous, patterned, nano-fibrous polymeric materials of nanofibers (Zucchelli *et al*., 2009). Other than producing a nanofiber, the advantages possessed by electrospinning due to its low cost, capability and high speed makes it has a high potential in producing nanocomposite fiber (Zhang *et al*., 2009). The unique properties such as extremely long, large surface area, complex pore size and complex alignment on either woven or nonwoven fiber possessed by electrospun nanofibers makes it practicable in various applications (Fang *et al*., 2011; Cavaliere *et al*., 2011; Sautter, 2005; Thavasi *et al*., 2008) especially in polymer electrolyte membrane. Thus, the combination of nanosized particles and specialty of the polymer electrolyte brings the focus to the study on nanocomposite polymer electrolyte membrane within laboratory as well as in industrial aspect.

## 1.2 Problem Statement

Several methods have been studied and developed to fabricate nanofibers, such as template, self-assembly, melt-blowing and phase separation as well as electrospinning (Doshi and Reneker, 1995; Fang *et al*., 2010). However, except electrospinning process, the other methods cannot produce continuous nanofibers on a large scale as well as simply altering the diameter from nanometers to micrometers and vice versa of the nanofibers. Electrospinning is competent in producing conductive fibrous membranes with high specific area, high porosity and tunable fiber diameters, which further broadened conductive polymers applicability in energy applications. Experimental parameters such as molecular weight, solubility, viscosity, surface tension, electrical conductivity, solvent vapor pressure, relative humidity, electric field and feed rate of the solution must be precisely controlled in getting desirable properties of the fibers and by tuning these conditions, a wide range of polymers can be processed.

Nafion, a sulfonated tetrafluoroethylene developed by Walther Grot (DuPont), is an interesting and most commonly materials used as proton exchange membrane in PEM fuel cells (Cason, 2010). Unfortunately, in electrospinning process, Nafion are difficult to electrospin due to its insolubility properties within solvents (Cason, 2010). The inability to electrospin happened due to the formation of micelles that leads to the decreasing in chain entanglement and thus, a high molecular weight carrier is needed to cater the problem facing by Nafion (Thompsett, 2010).

Previously, Jaafar *et al*. (2011) had successfully fabricated Cloisite15A® within SPEEK matrix which is comparable to Nafion. However, their method is still limited due to the size distribution of Cloisite15A® particles. Therefore, in this study by introducing the electrospinning process of SPEEK as the base polymer matrix, while the Cloisite15A® nanoclay as an inorganic filler, it is strongly believed that a novel polymer-nanocomposite electrolyte membrane with reducing filler size up to nanostructure can be successful developed.

## 1.3 Objective of Study

The aim of this study is to fabricate a conductive SPEEK/e-spun Cloisite15A® nanocomposite membrane with an increasing value of proton conductivity and reducing methanol permeability at acceptable value for direct methanol fuel cell system. The specific objectives of the study are:

1. To establish the best electrospinning condition for spinnable solution.
2. To fabricate SPEEK/Cloisite15A® nanocomposite membrane from the dope formulation of SPEEK and electro-spun SPEEK/Cloisite15A® nanofibers.
3. To characterize the performance of membrane based on Cloisite15A® dispersion state in term of physical and thermal stability.

## 1.4 Scope of Study

In order to achieve the aforementioned objectives of the research, the following scopes are outlined:

1. Fabricating electrospun Cloisite15A® at the least amount that is spinnable (0.05 wt. %) via electrospinning process by introduction of SPEEK (20 wt. % at DS 63%) as a carrier polymer and controlling the electrospinning parameters such as voltage (0 ~ 16kV), flow rate (0.6 ml/hr) and needle to collector distance (20 cm).

2. Preparing SPEEK/e-spun Cloisite15A® nanocomposite membrane by stirring-mixing 16wt. % of SPEEK at DS 63% and the electrospun Cloisite15A® nanofiber.

3. Observing the dispersion of the Cloisite15A® particles in electrospun nanofiber mat via SEM.

4. Determining the dispersion state of the Cloisite15A® particles in nanocomposite membrane via FESEM and XRD.

5. Characterizing the physical and thermal properties of the prepared membrane in term of water uptake, proton conductivity, methanol permeability and SPEEK/e-spun Cloisite15A® membrane materials degradation at certain temperature.

## 1.5 Significance of Study

The application of membrane consists of polymer and inorganic filler is interesting within the past two decades in fuel cell applications. In this study, a continuing work in fabricating a series of SPEEK and Cloisite15A® based proton electrolyte membrane for direct methanol fuel cell application was performed by employing electrospinning technique. This route is basically in producing nanosized filler in nanocomposite structure within a short period of time. The contribution of electrospinning on the size reduction and dispersion state of Cloisite15A® has led to the improvement of proton conductivity as well as methanol permeability of the membrane. This research is also in the significance of developing a sophisticated fuel cells device in order to reduce environmental problem as well as reducing the relying cost on transportation and stationary usage of more compact design.

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