SYNTHESIS AND CHARACTERIZATION OF SULFONATED POLY (ETHER ETHER KETONE) / GRAPHENE OXIDE NANOCOMPOSITE FOR POLYMER ELECTROLYTE MEMBRANE

NUOR SARIYAN BINTI SUHAIMIN

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DEDICATION

This thesis is dedicated to my father, ***Suhaimin Hamat*** *who taught me that the best kind of knowledge to have is that which is learned for its own sake and can be shared with others. It is also dedicated to my mother,* ***Makelsom Sa'amah*** *who taught me that even the largest task can be accomplished if it is done one step at a time. To all my sisters,* ***Nasuyanim, Nadzera, Nasrenim, Nurshahma*** *and* ***Nurfilzah*** *thanks for always being my pillars*

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ABSTRACT

The challenge of methanol crossover, which seriously occurred using commercial Nafion® membrane, urging the number of studies on the development of novel proton exchange membrane (PEM). The main objective of this research was to prepare and characterize sulfonated poly (ether ether) ketone (SPEEK)/graphene oxide (GO) nanocomposite as PEM. The influences of oxidation conditions particularly oxidant ratio, reaction temperature, and oxidation time on the GO properties were investigated. GO prepared with loading ratio 1:3, at reaction temperature 35°C and oxidation time 5 days was found to be the optimum GO which demonstrated excellent desired properties which abundantly available hydroxyl and epoxide groups. A fully exfoliated GO with high graphitic integrity was confirmed by x-ray diffraction (XRD) and was supported by x-ray photoelectron spectroscopy (XPS) and field emission scanning electron microscopy (FESEM). Subsequently, this optimized GO with various loading amounts was chosen to be incorporated into the SPEEK solution and further studied. The effect of GO filler incorporation was investigated based on their physicochemical properties such as ion exchange capacity (IEC), swelling behavior, activation energy, proton conductivity, and methanol permeability. A fully exfoliated GO in SPEEK matrix was confirmed by XRD and was supported by FESEM and transmission electron microscopy (TEM). Besides, the resultant SPEEK/GO nanocomposite membranes characteristics were compared with the blank SPEEK and commercially Nation®115 membranes. SPEEK/GO nanocomposite with 2.5 wt.% GO loading possessed a strong interfacial interaction between SPEEK matrix and GO fillers, which offered a high proton conductivity (0.13 $S.cm^{-1}$) with lower methanol permeability (3.69 x $10^{-7}.cm^{2}s^{-1}$) compared to commercial Nation®115 membranes. Hence, owing to its promising improved characteristics and performance, SPEEK/GO0.25 had been identified as the best composite formulation for PEM.

ABSTRAK

Cabaran aliran silang metanol yang berlaku dengan serius dalam penggunaan membran Nafion® komersial telah mendesak diadakan beberapa kajian pembangunan membran elektrolit polimer baharu (PEM). Objektif utama penyelidikan ini ialah untuk mensintesis dan mencirikan poli(eter eter keton) sulfonat (SPEEK)/grafin oksida (GO) sebagai PEM. Kesan-kesan keadaan pengoksidaan terutamanya nisbah bahan pengoksidaan, suhu, dan masa pengoksidaan terhadap sifat-sifat GO telah dikaji. GO yang disediakan dengan nisbah muatan 1:3, pada suhu tindak balas 35°C dan masa pengoksidaan 5 hari telah dikenalpasti sebagai optimum yang menunjukkan sifat-sifat yang dikehendaki dengan mempunyai banyak kumpulan berfungsi hidroksil dan epoksida. GO yang telah terkelupas dengan sempurna dan mempunyai struktur grafit yang yang berintegriti tinggi telah disahkan menerusi pembelauan sinar-x (XRD), dan disokong oleh spektroskopi fotoelektron sinar-x (XPS) dan mikroskopi elektron pengimbas pemancaran medan (FESEM). Seterusnya, GO yang optimum dengan pelbagai jumlah muatan telah dipilih untuk digabungkan ke dalam larutan SPEEK dan dikaji secara terperinci. Kesan penggabungan pengisi GO telah dikaji berdasarkan sifat-sifat kimia-fizik misalnya kapasiti penukaran ion (IEC), sifat kebengkakan, tenaga pengaktifan, kekonduksian proton dan ketelapan metanol. GO yang terkelupas sepenuhnya di dalam matriks SPEEK telah disahkan melalui XRD dan disokong oleh FESEM dan mikroskopi elektron penghantaran (TEM). Selain itu, ciri-ciri hasil membran nanokomposit SPEEK/GO yang terhasil telah dibandingkan dengan membran SPEEK kosong dan membran komersial Nafion® 115. Nanokomposit SPEEK/GO dengan muatan GO 2.5 wt.% mempunyai interaksi antara muka yang kuat antara matriks SPEEK dan pengisi GO, yang menawarkan kekonduksian proton yang tinggi (0.13 S cm^{-1}) dan ketelapan metanol yang rendah (3.69×10^{-7} cm^2 s^{-1}) berbanding membran komersil Nafion®115. Oleh itu, dengan menjanjikan peningkatan ciri-ciri dan prestasi, SPEEK/GO0.25 telah dikenalpasti sebagai formulasi komposit terbaik bagi PEM.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AFC	-	Alkaline fuel cell
PAFC	-	Phosphoric acid fuel cell
MCFC	-	Molten carbonate fuel cell
EFC	-	Enzymatic fuel cell
PEMFC	-	Polymer electrolyte membrane fuel cell
DMFC	-	Direct methanol fuel cell
DEFC	-	Direct ethanol fuel cell
SOFC	-	Solid oxide fuel cell
PFSA	-	Perfluorinated sulfonic acid
PVDF	-	Polyvinylidene
SPEEK	-	Sulfonated poly(ether ether ketone)
DS	-	Degree of Sulfonation
IEC	-	Ion exchange capacity
GO	-	Graphene oxide
FET	-	Field-effect transistors
AFM	-	Atomic Force Microscopy 7
XRD	-	X-Ray Diffraction
XPS	-	X-Ray Photoelectron Spectroscopy
TEM	-	Transmission electron Microscopy
TGA	-	Thermogravitmetric analysis
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infra-Red Spectrometer
^{1}HNMR	-	Hydrogen Nuclear Magnetic Resonance
^{13}CNMR	-	Carbon Nuclear Magnetic Resonance
H^+	-	Proton

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Energy plays a significant role in sustainable development, as it provides underpin almost all aspects of human activity including social, economic as well as political. Since the beginning of modernization and industrialization among developing countries, world energy consumption has increased significantly and more rapidly rather than the world population. If the current trend perpetuates, people may suffer from an energy shortage crisis soon. Coal, oil, and natural gas are three types of fossil fuels continue to dominate as the primary sources for current energy production. However, the problem is fossil fuels are non-renewable and they are limited in supply and eventually will run out. In addition to the limited resources, exhaust gases such as carbon dioxide (CO_2), sulfur dioxide (SO_2) and nitrogen oxide (NO) resulting from the combustion of fossil fuels contributes an adverse effect on the environment in terms of global climate disruption, human health problem, air, water, and soil pollution. Therefore, extensive development and research have been conducted on renewable energy resources as it offers clean alternatives to fossil fuels based energy. They produce little or no greenhouse gases and importantly will never run out (Hockenberry, 2008; Takashima *et al.*, 2014)

Renewable are sources that are continuously replenished by the action of the sun on earth. They include wind, hydro-power, solar, biofuels, and geothermal. Nevertheless, these technologies also have some limitations, such as high cost and difficult to generate large quantities of electricity as produced by traditional fossil fuel generators. Besides, it is also limited by the reliability of supply. When the resources are unavailable so the capacity to make energy from them are limited (Turner, 1999; Varun *et al.*, 2009) This can be unpredictable and inconsistent, hence, another alternative is required to overcome these drawbacks.

Nowadays, fuel cell technology could be considered as a smart technology as well as a leading sustainable alternative to conventional energy generation techniques, whereby it able to operate with low emissions and high efficiency, thus, low consumption of primary energy. Besides, the fuel cell generates electrical power without releasing the pollutant agent as it's by-product is heat and water (Sundmacher, 2010). In the fuel cell, chemicals will constantly flow into the cell hence it will never go dead, as long as there is a flow of chemicals into the cell. Most fuel cells in the market today use hydrogen and oxygen as their chemicals. There are several different types of fuel cells, and each of them using different functional layers. Fuel cells are classified by their operating temperature and the type of electrolyte they use. Based on their operating temperature, low temperature (60-80°C) fuel cells are useful for small portable applications or powering the vehicles, whereas the high-temperature fuel cells work well for stationary power generation plants. Main types of fuel cells include alkaline fuel cell (AFC) system, phosphoric acid fuel cell (PAFC) system, molten carbonate fuel cell (MCFC) system, enzymatic fuel cell (EFC) system, polymer electrolyte membrane fuel cell (PEMFC) system, direct methanol fuel cell (DMFC) system, direct ethanol fuel cell (DEFC) system and solid oxide fuel cell (SOFC) system. Most fuel cells are powered by hydrogen, which can be fed to the fuel cell system directly or can be generated within the fuel cell system by reforming hydrogen-rich fuels such as methanol, ethanol, and hydrocarbon fuels.

Among these fuel cells, PEMFC has been projected as promising power sources for many potential applications due to its capability to generate high power density and have low weight and volume compared to other fuel cells (Wu, 2016). Due to their low sensitivity to orientation, favorable power to weight ratio, and fast start-up time, PEMFC is used mainly in stationary applications and transportation applications such as cars and buses. However, the issue of hydrogen storage has become an ultimate barrier for PEMFC extensively applied in vehicles.

To counter this drawback, higher-density liquid fuels, such as methanol, ethanol, natural gas, liquefied petroleum gas, and gasoline can be used to substitute fuel, but the vehicles must have an on-board fuel processor to reform the methanol to hydrogen. This requirement would increase the costs as well as maintenance. The reformer also tends to releases carbon dioxide (a greenhouse gas), though less than that emitted from current gasoline-powered engines. Therefore, DMFC as a variation of the PEMFC become a solution for this issue (Kumar *et al.*, 2014).

Though DMFC technology is relatively new compared with other fuel cells powered by pure hydrogen and DMFC research and development is roughly 4-5 years behind for other fuel cell types, DMFC still be considered as a highly promising power source. It is based on polymer electrolytes membrane (PEM) fuel cell technology. DMFCs exhibit several advantages such as liquid fuel, quick refueling, low cost, and compact cell design, thus making it suitable for various potential applications including stationary and portable applications (Li and Faghri, 2013). DMFCs are also environmentally friendly, despite carbon dioxide is produced, however, there is no production of other pollutants (sulfur or nitrogen oxides). Nevertheless, the development of commercial DMFCs has been hindered by some limitations, hence, extensive research and development have been conducted with the ultimate goal for improving the performance of DMFC system, however, the main focus of the studies should be emphasized on the drawbacks of PEM as it is the heart of DMFC system.

1.2 Problem Statement

Polymer electrolyte membrane (PEM): a thin membrane which is the heart of the DMFC system enables fuel cells to conduct its electron by attracting the protons, and enabling them to diffuse through the layer while maintaining their proton state. To date, Perfluorinated sulfonic acid (PFSA) polymer electrolytes membranes (tradenames; Nation®, Flemion, Aciplex, Dow) is the most frequently used PEM as it exhibits excellent chemical, mechanical and thermal stability besides providing high ionic conductivities in a humid environment (Raj, 2005). However, these PFSA membranes are quite expensive and suffer from several shortcomings such as high methanol crossover, low conductivity at low water contents, and relatively low mechanical strength at a higher temperature. Methanol crossover from the anode to the cathode through the membrane is one of the most critical barriers to the commercialization of the direct methanol fuel cell (DMFC) as it limited its performance and application. Most of the limitations associated with PFSA membranes can be overcome by modifying current PFSA or developing alternative membranes that could be operated at a temperature higher than 100 °C.

The developments of polymer membranes can be classified into four groups: (1) modified PFSA membranes, (2) alternative sulfonated polymers and their inorganic composite membranes, (3) acid-base complex membranes, and (4) ionic liquid-based gel-type proton conducting membranes. Among them, sulfonated polymer membranes seem to have a promising future as replacements for the PFSA. The polymer electrolyte membrane is synthesized by incorporating of various inorganic filler into polymer matrix such as silicate, silica-aluminum oxide, zeolite, and titanium oxide (Nunes *et al.*, 2002;.Ismail, Othman and Mustafa, 2009; Wu *et al.*, 2007; Mishra *et al.*, 2012). Numerous of polymer materials such as sulfonated polystyrene, poly(phenylene sulfide), polyvinylidene fluoride (PVDF), polybenzimidazole, and sulfonated poly(ether ether ketone) (SPEEK) have been prepared and functionalized as potential membrane electrolytes for DMFCs application and the developments mainly aim to lower material cost for low-temperature operation (Jiang *et al.*, 2012).

Many types of research have been done on graphene oxide (GO) by investigating its performance as functional materials in the various applications, such as polymer composites, energy-related materials, sensors, 'paper'-like materials, field-effect transistors (FET), and biomedical applications, due to its excellent electrical, mechanical, and thermal properties (Gao, 2015; Zhu, Murali, Cai, Li, Suk, Jeffrey R. Potts, *et al.*, 2010). Besides, GO has shown its high dispersion property in the polymer matrix. It is easily dissolved in a variety of solvents, hence further helps in improving interfacial interactions with the polymer matrix. GO composite has been demonstrated good physical and chemical properties to be used as PEMs.

Chemically and thermally stable aromatic polymer, SPEEK has received considerable interest as the most promising alternative PEM. SPEEK is basically prepared by sulfonating PEEK with concentrated sulphuric acid (H_2SO_4) resulting sulfonic acid group substituted directly to the aromatic backbone (Li *et al.*, 2005). These copolymers possess many good attributes in the DMFC system, such as good thermal stability, mechanical strength, low price, and improvable proton conductivity (Tsai, Kuo, and Chen, 2009; A.F.Ismail, Othman and Mustafa, 2009). Although it is improvable, the conductivity of the SPEEK membrane is still lower than PFSA. This proton conductivity greatly depends on the degree of sulfonation (DS).

Highly sulfonated polymers will swell significantly at high temperature and humidity (Xing *et al.*, 2005). While both proton conductivity and methanol crossover increase with the increase of ion exchange capacity (IEC) of the membrane. Therefore, for practical use, the properties of the plain SPEEK membrane have to upgrade, which could be achieved by incorporating them with carbonaceous fillers. Many studies have shown that the incorporation of carbonaceous can greatly enhance the usability of SPEEK as PEM in PEMFCs (Kayser *et al.*, 2010; Marani *et al.*, 2010; Gupta *et al.*, 2013). In this work, we aim at preparing and characterizing nanocomposite, GO based on the dispersion of nano-scaled GO into the SPEEK matrix. The optimized GO with enriched oxygen functional groups acts as a carbonaceous filler is expected could improve the diffusion of protons through continuous channels as well as provide the tortuous path for methanol, therefore producing better attributes of PEMs in terms of physicochemical properties, as well as thermal stability.

1.3 Objectives

Based on the research background and problem statement identified the objectives of the research are:

(a) To synthesize and characterize graphene oxide using modified Hummer’s method

(b) To develop and characterize the sulfonated poly (ether ether ketone)/graphene oxide membranes in terms of physicochemical properties, thermal stability, and membrane structural morphologies.

(c) To study membrane behaviors in terms of proton conductivity, ion exchange capacity, liquid uptakes, swelling behaviors, and methanol permeability of sulfonated poly (ether ether ketone)/graphene oxide.

1.4 Scope of the Study

To achieve the aforementioned objectives, the following scopes have been drawn:

1) Identifying the suitable fillers for polymer solution formulation based on data available in the open literatures.

2) Preparation of graphene oxide from graphite using modified Hummer's method

3) Studying three significant parameters (oxidant ratio, reaction temperature and reaction time) and evaluating its effect on oxygen functional groups formation

4) Analyzing on the GO using X-Ray Diffraction (XRD), field emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR) and Atomic Force Microscopy (AFM).

(Items 1–4: 1st)

5)Synthesizing of SPEEK polymer by sulfonation process

6) Determining the degree of sulfonation (DS) of SPEEK and SPEEK/GO nanocomposite by using hydrogen nuclear magnetic resonance (^{1}HNMR).

(Items 5–6: 2nd)

7) Preparing SPEEK/GO membrane solution for flat sheet membrane.

8) Determining GO dispersion state in SPEEK polymer matrix using XRD, FESEM, FTIR, AFM

9) Characterizing the membrane behavior of developed membranes in terms of liquid uptakes, proton conductivity and methanol permeability.

(Items 7–9: 3rd)

1.5 Significance of Studies

This study is expected to prepare a high ratio with outstanding properties of carbonaceous nanofiller, graphene oxide (GO), which can be applied to develop potential SPEEK/GO membranes that possess better attributes in terms of higher proton conductivity as well as thermal stability and concurrently, lowering the methanol permeability, as a result, improving the efficiency and the durability of the PEM. With these superior characteristics, it is expected to perform excellently in the DMFC system and at once would commercialize the application of DMFCs in the market, thus promoting a green and better environment.

1.6 Thesis Organization

This thesis explains in detail the synthesizing of GO for modifying the structure of the SPEEK polymer electrolyte membrane for DMFC. Though a lot of studies have been done on this area, however, is still challenging and required a lot of effort to produce the PEM with high proton conductivity and simultaneously low methanol permeability. This thesis consists of 7 chapters with specific objectives and outline of each chapter are given below:

Chapter 1 describes general information about fuel technology and its' potential, particularly for energy solutions. Development and research are essentially required for the heart of fuel cell (PEM) to accomplish its commercialization. Also, this chapter highlight the background of the study, problem statement, objectives as well as the scope of studies

Chapter 2 presents a comprehensive literature review of DMFC and PEM in particular. Besides, it also discusses GO and its nanocomposites in depth. Areas covered for GO particularly, include their history of fabrication, properties, characterization techniques, and applications. Next, it explains the criteria have to consider in modifying PEM structure such as blending inorganics and polymers. Overview of polymer nanocomposites interface and interphase are also included.

Finally, the key parameters in the evaluation of PEM performance, methanol crossover, and proton conductivity are presented.

Chapter 3 presents the methodology and description of the research process. It provides information concerning the method that was used in undertaking this research to synthesize GO, SPEEK, and SPEEK/GO. Besides, it also covers all the techniques used to characterize both the filler and nanocomposite membrane. The chapter proceeds to present with the key measurements used in evaluating and determining the interaction between PEM behaviors and its' performance.

Chapter 4 depicts the effect of studied parameters on the synthesized GO. Furthermore, it also discusses in detail the effects of studied parameters on the formation of oxygen functional groups based on various characterization methods used for describing their physicochemical characteristics.

Chapter 5 reveals and confirms the success of the sulfonation process on Poly (ether ether) ketone (PEEK) backbone via FTIR and ^{1}HNMR. Herein it determines the degree of sulfonation for the SPEEK and studies the effect of sulfonation on thermal stability in particular. In addition, it also studies the effect of GO loading on the property enhancement of the SPEEK matrix and covers some characterization methods used to indicate their physicochemical properties changes. The study is performed by using Fourier transform infrared emission spectroscopy and is compared with the DSC study.

Chapter 6 present the development and synthesis of GO that are compatible with the polymer SPEEK matrix. This chapter further presents the effect of GO with SPEEK/GO membrane in terms of liquid uptakes measurement, swelling behavior, hydrophilicity properties, methanol crossover as well as proton conductivity. The main conclusions resulting in this project are summarized in Chapter 7. Possible future work of research is also included.

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