MAGNETIC FIELD ALIGNED FERROCENE FUNCTIONALIZED POLYBENZIMIDAZOLE MEMBRANE FOR HIGH TEMPERATURE PROTON EXCHANGE MEMBRANE FUEL CELL

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DEDICATION

'*Glory is to Allah, and praise is to Him to the extent of the number of His creation and to the extent of His pleasure and to the extent of the weight of His Throne and to the extent of the ink of His words,'* [Sahih Muslim: 6913].
This thesis is specially dedicated to my beloved
MOM, Miskiah
DAD, Sean
SISTERS, Kartini and Tumirah
BROTHER, Mohd Khairul
Thank you very much for
the support,
prayer,
and
love.

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ABSTRACT

Proton conductors that proficiently operate at high temperatures (over 100^{o}C) have been gaining increasing attention for proton exchange membrane fuel cells (PEMFCs). Many approaches have been taken to improve the proton conductivity of the membrane. The main challenge is in developing PEM with improved unidirectional proton-conducting channels through the membranes. Thus, this research has focussed on enhanced proton transport properties of ferrocene functionalized polybenzimidazole (Fc-PBI) membrane in oriented microstructures via magnetic field-assisted solvent casting method. Commercially available PBI solution (Celazole® PBI S26) and ferrocene carboxylic acid (FCA) were used as the polymer matrix and the alignment agent, respectively. Before the fabrication of the membrane, the magnetic properties of PBI and FCA were investigated using a vibrating sample magnetometer (VSM), and both displayed enough magnetic susceptibility for alignment under a magnetic field. Therefore, it was hypothesized that magnetic field-aligned Fc-PBI membrane will improve the proton conductivity through the alignment of the proton-conducting channel in PBI microstructures. Fc-PBI membrane was prepared from the mixture of FCA and PBI solution, followed by casting the mixture onto a glass plate using a scrapper. The casted solution underwent 0.3 Tesla magnetic field treatment in in- and through-plane directions. The physical properties of Fc-PBI membranes were characterized using Fourier transform infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), diffuse reflectance Ultraviolet-visible (DR UV-vis) spectroscopy, X-ray diffraction (XRD) spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and thermogravimetry analysis (TGA). The FTIR analysis of the Fc-PBI membrane identified the appearance of N-H amide peaks, indicating that the ferrocene moiety successfully bonded to PBI main chain. The FTIR results were in good agreement with XPS data, detecting the N-(C=O) signal. SEM and AFM images showed that the polymer microstructure was aligned towards the magnetic field direction. The TGA results confirmed that the thermal stability of the membranes was satisfactorily high to operate at high temperatures. Fenton's test was performed, and the results showed a decrease in oxidative stability with a high amount of Fc content. In this case, the bivalent state of iron (Fe^{2+}) in Fc transforms to ferric ion (Fe^{3+}), initiating the Fenton reaction to decompose the membranes. Even with low oxidative stability, the proton conductivity of aligned Fc-PBI in through-plane direction with 5 wt% FCA at 180^{o}C is 0.024 Scm^{-1}, which is better than that of pristine PBI. The protonic conductivity was found to increase with the formation of through-plane aligned proton channels, reflecting the ease of proton transportation through the short and continuous pathway through the membrane and the effect is more prominent at the high amount of Fc. Therefore, it is suggested that the magnetic field-aligned Fc-PBI would be a strong candidate for high-temperature PEMFC applications.

ABSTRAK

Konduktor proton yang beroperasi dengan cekap pada suhu yang tinggi (melebihi 100^{o}C) semakin mendapat perhatian bagi sel bahan api membran pertukaran proton (PEMFC). Banyak pendekatan telah diambil untuk meningkatkan kekonduksian proton membran tersebut. Cabaran utama ialah dalam menghasilkan PEM dengan saluran konduksi proton satu arah yang lebih baik. Dengan itu, penyelidikan ini tertumpu kepada peningkatan sifat pengangkutan proton membran polibenzimidazol berkefungsian ferosena (Fc-PBI) di dalam struktur mikro berorientasi melalui kaedah tuangan pelarut berbantukan medan magnet. Larutan PBI yang tersedia secara komersial (Celazole® PBI S26) dan asid karboksilik ferosena (FCA) masing-masing digunakan sebagai matriks polimer, dan agen penjajaran. Sebelum fabrikasi membran, sifat magnetik PBI dan FCA telah dikaji menggunakan magnetometer sampel bergetar (VSM), dan kedua-duanya mempamerkan kerentanan magnetik yang cukup untuk penjajaran di bawah medan magnet. Oleh itu, dihipotesiskan bahawa membran Fc-PBI yang sejajar medan magnet akan meningkatkan kadar kekonduksian proton melalui penjajaran saluran konduksi proton di dalam struktur mikro PBI. Membran Fc-PBI disediakan daripada campuran larutan FCA dan PBI, diikuti dengan menebarkan campuran tersebut ke atas plat kaca menggunakan pengikis. Campuran yang telah ditebarkan itu menjalani rawatan medan magnet 0.3 Tesla dari arah dalam satah dan melalui satah. Sifat fizikal membran Fc-PBI dicirikan menggunakan spektroskopi inframerah transformasi Fourier (FTIR), spektroskopi fotoelektron sinar-X (XPS), spektroskopi pantulan serakan ultralembayung-cahaya nampak (DR UV-vis), spektroskopi pembelauan sinar-X (XRD), imbasan mikroskop elektron (SEM), mikroskopi daya atom (AFM), dan analisis termogravimetri (TGA). Analisis FTIR membran Fc-PBI telah mengenalpasti kemunculan puncak amida N-H, yang menunjukkan bahawa bahagian ferosena berjaya terikat pada rantai utama PBI. Hasil FTIR sesuai dengan data XPS, yang mengesan isyarat N-(C=O). Imej SEM dan AFM menunjukkan bahawa struktur mikro polimer adalah sejajar ke arah medan magnet. Hasil TGA mengesahkan bahawa kestabilan terma membran adalah memuaskan untuk beroperasi pada suhu tinggi. Ujian Fenton dilakukan, dan hasilnya menunjukkan penurunan kestabilan oksidatif dengan jumlah kandungan Fc yang tinggi. Dalam kes ini, keadaan besi dwivalen (Fe^{2+}) dalam Fc yang berubah menjadi ion ferik (Fe^{3+}), memulakan reaksi Fenton untuk menguraikan membran. Malah dengan kestabilan oksidatif yang rendah, kekonduksian proton bagi membran Fc-PBI yang sejajar dalam arah melalui satah dengan 5 wt% FCA pada 180^{o}C adalah 0.024 Scm^{-1}, yang lebih baik daripada PBI asli. Kekonduksian proton didapati meningkat dengan pembentukan saluran proton yang sejajar melalui satah, yang menggambarkan mudahnya pengangkutan proton melalui laluan yang pendek dan berterusan melalui membran dan kesannya lebih ketara pada jumlah Fc yang tinggi. Oleh itu, membran Fc-PBI yang sejajar dengan medan magnet sebagai calon yang berpotensi bagi aplikasi PEMFC pada suhu tinggi.

TABLE OF CONTENTS

CHAPTER 3 RESEARCH METHODOLOGY

LIST OF TABLES

LIST OF FIGURES

LIST OF SCHEMES

LIST OF ABBREVIATIONS

SO_x	-	Sulfuric oxide
NO_x	-	Nitrogen oxide
AFC	-	Alkaline fuel cell
MCFC	-	Molten carbonate fuel cell
PAFC	-	Phosphoric acid fuel cell
SOFC	-	Solid oxide fuel cell
PEMFC	-	Proton exchange membrane fuel cell
HT-PEMFC	-	High temperature-proton exchange membrane fuel cell
PEM	-	Proton exchange membrane
FTIR	-	Fourier transforms infrared
XPS	-	X-ray photoelectron spectroscopy
DR UV-vis	-	Diffuse reflectance Ultraviolet-visible spectroscopy
XRD	-	X-ray Diffraction
SEM	-	Scanning electron microscopy
AFM	-	Atomic force microscopy
TGA	-	Thermogravimetric analysis
EIS	-	Electrochemical impedance spectroscopy
NASA	-	National Aeronautics and Space Administration
MEA	-	Membrane electrode assembly

LIST OF SYMBOLS

kW - Kilo watt

^{o}C - Degree celcius

K - Kelvin

B - Magnetic field

T - Tesla

kOe - Kilo oersted

S/cm - Siemen per centimetre

% - Percentage

wt % - Weight percentage

σ - Proton conductivity

σ_o - Pre-exponential factor

Ea - Activation energy

R - Boltzmann constant

kJ/mol - Kilo Joule per mole

M_s - Magnetization saturation

M_r - Remanent magnetization

H_c - Coercivity

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Since ancient times, fossil fuels such as coal, natural gas (NG), and petroleum (oil) for energy generation have dominated the energy market. These great energy sources make modern life possible since they play a vital role in generating electricity, power systems, and manufacturing goods in the industry. However, the use of fossil fuels leads to the emission of billion tons of toxic gases that leads to global warmings, such as carbon dioxide (CO_2), sulfuric oxide (SO_x), and nitrogen oxide (NO_x) species. This high number is largely attributed to the fossil fuels burning in China and India (1). To overcome this problem, some countries like the United Kingdom have decided to ban fossil fuel, especially in vehicles, by 2040 and looking forward to more green energy (2).

In addition, nowadays, people who work in the industry are highly concern about fossil fuels depletion. According to the International Energy Agency (IEA), world oil production is reached to maximum and is expected to decline steadily year after year (3). This phenomenon will affect the industrial, transportation and the socio-economic growth of the country (4). Thus, the world is now shifting from fossil fuels to another alternative and sustainable energy called fuel cells.

Fuel cells were inverted back then in the middle of the 19th century with the principle of converting the free energy of a chemical reaction into electrical energy via an electrical current (5). Fuel cells offer zero-emission of toxic gases, high efficiency, design variability, and flexibility of fuel used (6). Figure 1.1 shows the efficiency characteristics of fuel cells compared with other electric power systems (7). Among various types of fuel cells for instance alkaline fuel cell (AFC), molten carbonate fuel cell (MCFC), phosphoric acid fuel cell (PAFC), and solid oxide fuel cell (SOFC), proton exchange membrane fuel cells (PEMFC) are recognized as the best choice due to its low operating temperature (<90^{o}C), easy start-up and fast response to changes in load and operating conditions, high power density, and have a robust system (8,9).

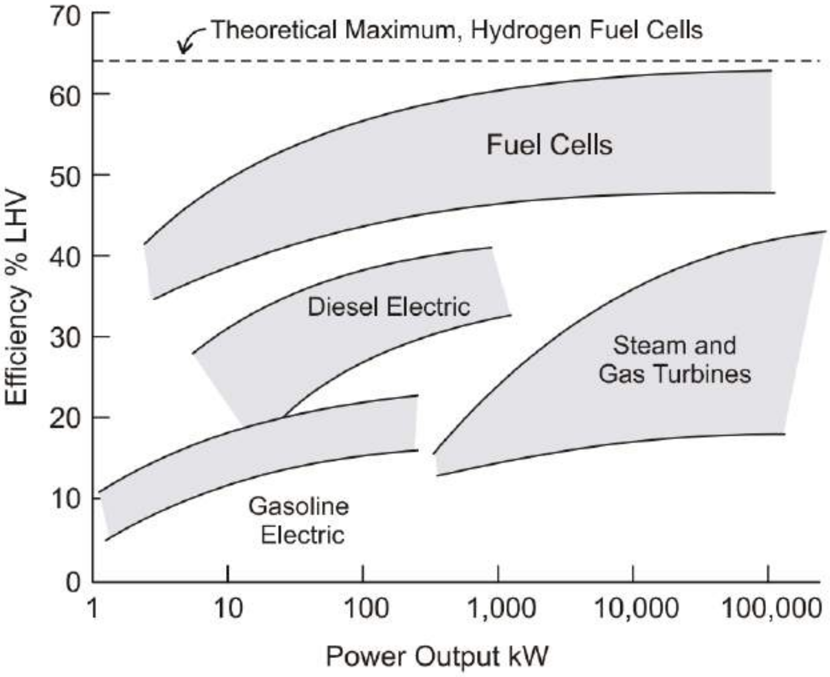

Figure 1.1 Comparison of power generating systems efficiency (7).

PEMFC generates electricity by combining the hydrogen as fuel and oxygen from the atmosphere as the oxidant, passing through the mixture via a membrane, and generating electricity with the release of water (H_2O) as a waste product (10). To date, PEMFC has been widely used as commercial products in the residential home, batteries for portable devices, for instance, cell phones and laptops, and fuel cell engines for automotive applications by displacing the internal combustion engine (11). Despite all the advantages provided, PEMFC deals with the use of high purity of hydrogen gas fuel, which is hard to control, experienced carbon monoxide (CO) poisoning at the platinum (Pt) catalyst electrode, and humidity problem (12). By increasing the operating temperature, the rate of H_2 adsorption increases and the rate of CO adsorption onto the anode catalyst decreases, hence reduce the poisoning of CO.

Thus, as an alternative, a high temperature-proton exchange membrane fuel cell (HT-PEMFC) is being developed.

HT-PEMFC offers high efficiency, easy water and thermal management, faster chemical kinetics at the anode and cathode, and heat utilization (13,14). HT-PEMFC performs under 120 to 180^{o}C operating temperature at ambient pressure and without humidity (12). The key material in HT-PEMFC performance is the proton exchange membrane (PEM). PEM acts as the medium in transporting protons from anode to cathode. In other words, PEM creates a pathway for proton conductivity. Moreover, it also acts as the barrier between anode and cathode, prevents the inlet gases from mixing, and avoids the electron from passing through the membrane due to the presence of negative charge from SO_3^- and electron repelling (10).

The current state of the art for PEM is Nafion. It was first discovered in the late 1960s by Walther grot of Dupont, and further developed and produced by the E.I. Dupont Company (15). Nafion is the commercial name for sulfonated tetrafluoroethylene-based fluoropolymer-copolymer. According to Kumar and his co-workers, Nafion membrane provides good proton conductivity, excellent mechanical and thermal stability, and has a long lifetime (16,17). However, the proton conductivity of Nafion is strongly dependent on humidification. Thus, limit its performance at high temperature up to 100^{o}C, since the capability to operate at high temperature offers more advantages than at low temperature. Thus, as an alternative to Nafion, many kinds of research had been done on non-fluorinated polymer membranes for HT-PEMFC application. Non-fluorinated polymeric membranes have high thermal and mechanical stability, oxidation resistance, and cheaper than Nafion.

Previously, polybenzimidazole (PBI) has been studied widely as it promises good mechanical, chemical, and thermal stability. Nevertheless, the use of pristine PBI permits shallow proton conductivity values compared to Nafion. Hence, an acid-doped PBI membrane has been introduced to improve the performance of PBI in PEMFC. Furthermore, without humidification, this membrane can produce excellent proton conductivity at a temperature as high as 200^{o}C (13,18).

The performance of HT-PEMFC is not only dependent on the operation temperature but also on the structural properties of the polymer membrane. Recently, various ordered structure of proton-conducting channel has been developed to enhance the performance of HT-PEMFC for example, by adding nanoparticles fillers into the polymer matrix. Those fillers create an additional route for proton transfer via the formation of mesopores. Other than that, Gong and his co-workers have constructed nano-scale proton conductive channels via electrospinning and electric field (19). Such design has been proven to increase performance. Though, these synthesis methods are restricted to dielectric breakdown and expose to physical contact that could cause shear alignment.

In the present work, a novel approach was proposed in fabricating well-aligned proton conductive channels of ferrocene functionalized PBI membrane under a low magnetic field (0.3 T) (Figure 1.2). Magnetic field alignments of the polymer can minimize the structural disorder of polymer membrane and enhance the proton conductivity of HT-PEMFC. This is due to the production of the straight proton-conducting channel in the in- and through-plane direction of the membrane. In addition, over electrospinning and electric field, the magnetic field is free from dielectric breakdown, physical contact, and very practical for scalable production. In this perspective, the ferrocene-based compound was chosen as the functionalization group to PBI. Lin reported that ferrocene-type materials have many excellent properties towards the magnetic field; for instance, they exhibit ferromagnetic properties, low magnetic loss, low relative density, thermally stable (1.5 K to 450 K), and good resistance to radiation (20). Thus, with the presence of ferrocene, the membrane may align under the low magnetic field and provide excellent proton conductivity of HT-PEMFC.

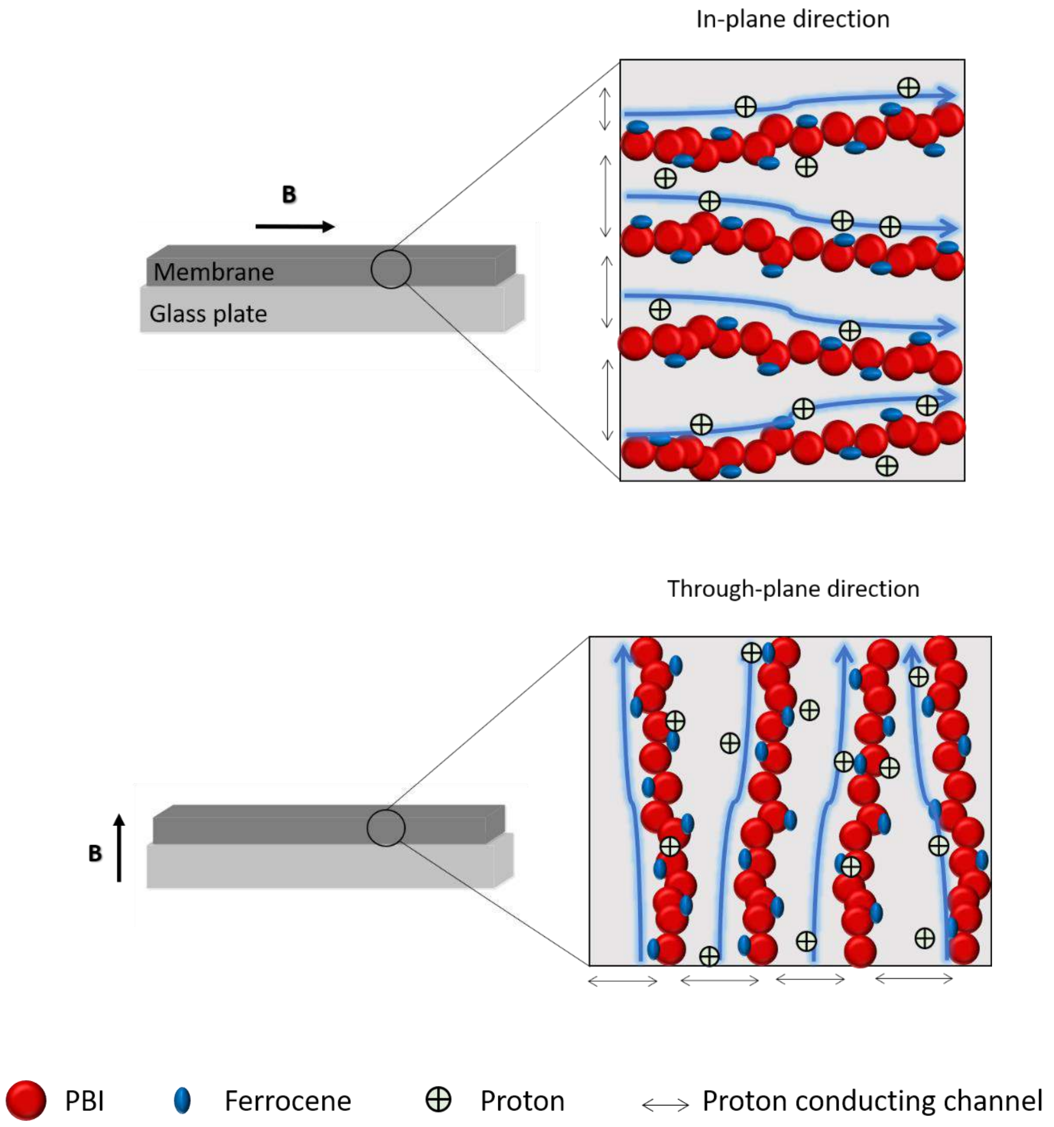

Figure 1.2 The feasible effect of magnetic field on the casted ferrocene functionalized PBI membrane under the low magnetic field of 0.3 T.

1.2 Problem Statement

This research is emphasized on the effects and the influence of magnetic fields towards well-aligned ferrocene functionalized PBI membrane synthesized under magnetic field (0.3 T) and its proton-conducting performances at high temperature (100 to 180°C).

PEM that proficiently operate at high temperatures for PEMFCs have been gaining great attention. Many strategies have been taken to improve the proton conductivity of the membrane. Incorporating nanoparticles (NPs) into the polymer matrices and developing unidirectional proton-conducting channels through the membranes are some of the strategies to increase the proton conductivity of PEM. It was reported that iron oxide (Fe_2O_3) NPs could align the proton conducting channel in the polymer matrix under the magnetic field and increase the performance of PEMFCs. Nonetheless, the problem is, only the Fe_2O_3 NPs aligned directly towards the magnetic field, and it is not clearly confirmed through SEM micrograph that the desired channel is aligned or vice versa. Moreover, according to Hasanabadi, with the incorporation of NPs, the polymer chains of the membrane become separated from each other and results in blocking effect of NPs that reduce the proton conductivity of PEM (22).

Therefore, the main challenge in this study is in developing unidirectional proton-conducting channel of the polymer microstructures with high proton conductivity. By aligning the polymer microstructures, the blocking effect of NPs could be overcome and the tortuous proton-conducting channel is reduced. Previously, it was reported that the orientation of polymer as an ion-conducting membrane can be controlled under a low magnetic field (<0.5 T) by using 4′-(hexyloxy)-4-biphenylcarbonitrile liquid crystals (60 cyanobiphenyl) as alignment agent, and 4-(3-acryloyloxypropyloxy)-benzoesure 2-methyl-1, 4-phenylester (Rotary Mixer 257) as stabilizer (21). However, the limitation to this technique is that the liquid crystals starts to evaporate at 70°C. and become unstable at high temperature.

In dealing with all the difficulties described above, ferrocene functionalized PBI membrane was synthesized using ferrocene carboxylic acid (FCA) as the functionalization group to PBI and the alignment agent under 0.3 T magnetic field. The crucial parts here will be the comprehensive attempt to investigate the correlation between the effect of the magnetic field towards the formation of a polymer membrane with the presence of the ferrocene-based compound, its structural-properties relationship with the proton conduction mechanism, and its proton conductivity. It is hypothesized that the well-aligned membrane can be synthesized under a low magnetic field with FCA as the alignment agent and the functionalization group. This material is more efficient in terms of proton conductivity performance compared to the non-oriented PEM. Based on the above considerations, the statement of the problem can be defined as follows: **The novel cast low magnetic field-aligned ferrocene functionalized PBI or Fc-PBI membrane can be used as an improved performance PEM with high proton conductivity at high operating temperature.**

This study focussed on a novel cast-controlled orientation of Fc-PBI composite membrane. The impact of the bonding formation of ferrocenyl group with polymer matrices can influence the alignment of Fc-PBI molecule in the composite membrane under a magnetic field. A detailed exploration throughout this study can yield a fundamental understanding as well as the structural properties relationship of PEM towards proton transport mechanism and its proton conductivity in high operating temperature.

1.3 Objectives of Study

The novelty of this work lies in the preparation of novel PEM by using the ferrocene-based polymeric compound as the alignment agent under an external magnetic field. The ultimate goal of the present work is as follows:

1. To fabricate Fc-PBI membrane by solvent casting method under 0.3 T magnetic fields in the in- and through-plane direction.

2. To identify the effect of an external magnetic field on the structural, morphological, thermal properties, and oxidative stability of the fabricated

membranes by using FTIR, XPS, DR UV-Vis, XRD, SEM, AFM, TGA, and Fenton’s test.

3. To evaluate the proton conductivity performance of Fc-PBI with respect to its structural properties by using EIS.

4. To relate the structural properties relationship of PBI-based membranes with their proton conduction mechanism.

1.4 Scope of Study

In this research, in producing the well-aligned structure of PEM, 0.3 T magnetic fields were applied with ferrocene as the alignment agent. The controlled orientation of the Fc-PBI membrane was successfully cast by the solvent casting method. The solution was placed under 0.3 T magnetic fields and self-dried for 1 hour with further heating at 80^{o}C for 24 hours to increase the mechanical strength. The Fc-PBI cast without the presence of a magnetic field was prepared as a comparison.

Several techniques were used to characterize the sample, for instance, scanning electron microscopy (SEM), atomic force microscopy (AFM), Fourier transforms infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), diffuse reflectance Ultraviolet-visible spectroscopy (DR UV-vis), X-ray diffraction (XRD) spectroscopy, thermal gravimetric analysis (TGA), and Fenton’s test. These physicochemical properties were related to the proton conductivity performance at high operating temperature, which was tested by using electrochemical impedance spectroscopy (EIS).

1.5 Significant of Study

This research comprehensively investigates the effect of magnetic field on the Fc-PBI membrane, which can be applied in HT-PEMFC application. It would significantly contribute to the fundamental knowledge of the structural-properties relationship towards proton conductivity and its mechanism and would be useful to the energy industry in the future. Besides that, the new structure of PEM cast based on the magnetic field oriented here can be a potential PEM in the administration of the ferrocene-based polymeric compound in the future, with some highlighted value of high proton conductivity, high thermal stability, and better fuel cell performance at relatively low humidity and high operating temperature.

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LIST OF PUBLICATIONS, CONFERENCES AND WORKSHOPS

Publications

Journal with Impact Factor

1. Sean, N. A., Leaw, W. L., Abouzari-lotf, E., & Nur, H. (2021). Magnetic field-induced alignment of polybenzimidazole microstructures to enhance proton conduction. *Journal of the Chinese Chemical Society*, 68, 86-94. (Q3, IF: 1.967)

Indexed Journal

1. Sean, N. A., Leaw, W. L., Abouzari-lotf, E., & Nur, H. (2020). Ferrocene-modified polybenzimidazole membrane with enhanced proton conductivity. *Malaysian Journal of Chemistry*, 22, 20-27.

Conferences

1. Participant at the Nanomite Annual Symposium 2017 (NMAS 2017), Universiti Putra Malaysia. 14th – 15th November 2017.

2. Participant at the 7th International Conference and Workshop on Basic and Applied Sciences (ICOWOBAS 2019), KSL Resorts, Johor Bahru. 14th – 18th July 2019.

3. Participant at the Nanomite Annual Symposium 2021 (NMAS 2021), Kuala Lumpur, Malaysia & Cisco Webex. 11th – 12th March 2021.