AUTOMATION OF HOLLOW FIBER MEMBRANE FABRICATION SYSTEM AND OPTIMIZATION OF SPINNING PARAMETERS FOR POLYMERIC MEMBRANE IN GAS SEPARATION

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DEDICATION

This thesis is lovingly dedicated to my supportive father who taught me that the best kind of knowledge to have is learned for its own sake. He is the person to hand me the first brick of my engineering and technical life. It is also dedicated to my selfless mother, who taught me that love is the source for the solution of any problem even the toughest one. I also should thank my guiding Brother for his role and generous help, and last but not least, I dedicate this thesis to my dear companion wife, who was with me since the beginning of our couple life with patience and kindness.

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ABSTRACT

The applications of the membrane in various types and shapes are crucial and vital especially in gas separation applications. The membrane fabrication process is complex and very delicate, but most of the existing fabrications are not fully automated which focusing only on one parameter at a time without considering the correlation between parameters involved. Therefore, a fully automated system needs to be developed with a fast and reliable fabrication procedure for determining the performance of hollow fiber membrane (HFM). The main objective of this work was to develop an automated fabrication that can be used to produce a highly producible membrane and to identify the optimum properties of HFMs for specific applications. This research was started with development of automatic fabrication machine using programmable logic controller and related sensors and actuators. Next, a mathematical model was developed and used in optimization procedure to predict the properties of HFM for gas separation application. A full factorial model was developed using a systematic experimental strategy based on the design of experiments that emphasized on tuning and simultaneous optimization of fabrication parameters of polysulfone membrane under various controllable and uncontrollable conditions. The tuning parameters for HFM fabrication were dope solution flowrate, bore fluid flowrate, air gap distance and collection speed, while CO_2 flux with CO_2/CH_4 selectivity and diameter of membrane are the optimized membrane properties. The cause and effect relationship between all parameters were observed to obtain an optimum gas separation performance based on scanning electron microscope and gas permeation test. Scanning electron microscope and gas permeation test were also utilized in determining the performance. Finally, experimental verification was conducted to seal the reliability of the model by comparing values predicted by the model and real experimental data for new random spinning condition. Moreover, an acceptable physical morphology and structure revealed by observing and discussing the scanning electron microscope. The mathematical model was able to predict the performance of manufactured membranes based on fabrication parameters during the fabrication of hollow fiber membranes accurately with less than 5 percent error and it shows that this model is well defined and described the phenomena within range reliability for production. As a result, optimum fabrication properties of 1.27 ml/min dope flowrate, 0.33 ml/min bore flowrate, 9.1 m/min collection speed and 4 cm air gap were introduced to obtain 33 and 38.28 gallon per unit as selectivity and CO_2 permeance by this study, respectively. A quality prediction model for HFM has successfully developed with 95% reliability for prediction of responses and 82.7% desirability of the performance for the indicated optimum point mentioned. This model allows membrane quality to be predicted before the fabrication stage thus prevents waste during fabrication. Additionally, variation of HFM (size and shape) can be designed and studied using this model.

ABSTRAK

Aplikasi membran dalam pelbagai jenis dan bentuk adalah penting terutamanya dalam aplikasi pemisahan gas. Proses fabrikasi membran adalah kompleks dan sangat rumit, namun kebanyakan fabrikasi semasa tidak automatik sepenuhnya yang mana hanya menumpukan kepada satu parameter pada satu masa tanpa mengambil kira perkaitan antara parameter yang terlibat. Oleh itu, sebuah sistem automatik perlu dibangunkan dengan satu prosedur yang pantas dan boleh dipercayai untuk menentukan prestasi membran gentian berongga (HFM). Objektif utama kajian ini adalah untuk membangunkan satu sistem fabrikasi automatik yang boleh digunakan untuk menghasilkan membran yang sangat mudah dihasilkan dan mengenal pasti sifat optimum HFM untuk aplikasi tertentu. Penyelidikan ini bermula dengan pembangunan mesin fabrikasi automatik menggunakan pengawal logik pengaturcaraan dan penderia dan penggerak yang berkaitan. Seterusnya, model matematik telah dibangunkan dan digunakan dalam prosedur pengoptimuman untuk meramalkan sifat HFM untuk aplikasi pengasingan gas. Model faktorial penuh dibangunkan menggunakan strategi eksperimen sistematik berdasarkan reka bentuk eksperimen yang menekankan pada penalaan dan pengoptimuman serentak parameter fabrikasi membran polisulfon di bawah pelbagai keadaan yang boleh dikawal dan tidak terkawal. Parameter penalaan untuk fabrikasi HFM adalah kadar alir larutan dop, kadar alir bendalir gerek, jarak jurang udara dan kelajuan pengumpulan, manakala fluks CO_2 dengan pemilihan CO_2/CH_4 dan diameter membran adalah sifat membran yang dioptimumkan. Hubungan sebab dan akibat antara semua parameter diperhatikan untuk mendapatkan prestasi pemisahan gas yang optimum berdasarkan pengimbasan mikroskop elektron dan ujian resapan gas. Pengimbasan mikroskop elektron dan ujian resapan gas juga digunakan dalam menentukan prestasi. Akhirnya, pengesahan eksperimen telah dijalankan untuk menentukan kebolehpercayaan model dengan membandingkan nilai yang diramalkan oleh model dan data eksperimen sebenar untuk keadaan putaran rawak baharu. Selain itu, morfologi dan struktur fizikal yang boleh diterima dijelaskan dengan memerhati dan membincangkan mikroskop elektron pengimbasan. Model matematik dapat meramal prestasi membran yang dihasilkan berdasarkan parameter fabrikasi semasa fabrikasi membran gentian berongga dengan tepat dengan ralat kurang daripada 5 peratus dan ia menujukkan bahawa model ini ditakrifkan dengan baik dan menerangkan fenomena dalam julat kebolehpercayaan untuk pengeluaran. Hasilnya, sifat fabrikasi optimum kadar alir dop 1.27ml/min, kadar alir gerek 0.33 ml/min, kelajuan pengumpulan 9.1 m/min, dan jurang udara 4 cm telah diperkenalkan untuk mendapatkan 33 dan 38.28 gelen seunit, masing-masing sebagai pemilihan dan ketelapan CO_2. Model ramalan kualiti untuk HFM telah berjaya dibangunkan dengan kebolehpercayaan 95% untuk ramalan tindak balas dan 82.7% kebolehinginan prestasi untuk titik optimum yang dinyatakan. Model ini membolehkan kualiti membran diramal sebelum peringkat fabrikasi sekali gus mengelakkan pembaziran semasa fabrikasi. Selain itu, variasi HFM (saiz dan bahan) boleh direka bentuk dan dikaji menggunakan model ini.

TABLE OF CONTENTS

TITLE **PAGE**

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AG	-	Air Gap
ANOVA	-	Analysis of Variance
BFR	-	Bore Flow Rate
CS	-	Collection Speed
DER	-	Dope Extrusion Rate
DMAc	-	N,N-Dimethylacetamide
DOE	-	Design of Experiment
EDA	-	Ethanediamine
EOR	-	Enhanced Oil Recovery
EtOH	-	Ethanol
HFM	-	Hollow Fiber Membrane
HFMs	-	Hollow Fiber Membranes
HMI	-	Human Machine Interface
JS	-	Jet-Stretch
MC	-	Methylcarbamate
MEA	-	Monoethanolamine
MIEC	-	Mixed Ionic-Electronic Conducting
MM-HFMs	-	Mix Matrix Hollow Fiber Membranes
MOFs	-	Metal-Organic Frameworks
MSCS	-	Melt Spinning and Cold Stretching
NGLs	-	Natural Gas Liquids
NIPS	-	Non-Solvent Induced Phase Inversion
PDMS	-	Polydimethylsiloxane
PEG	-	Polyethylene Glycol
PEI	-	Polyetherimide
PG	-	Piperazine Glycinate
PI	-	Polyimide
PIP	-	Piperazine
PLC	-	Programmable Logic Controller
PP	-	Polypropylene

PSA	-	Pressure Swing Adsorption
PSF/PS	-	Polysulfone
PVA	-	Poly Vinyl Alcohol
PVAm	-	Polyvinylamine
PVDF	-	Polyvinylidene Fluoride
PVF	-	Polyvinyl Fluoride
SEM	-	Scanning Electron Microscope
TFC-HFMs	-	Thin Film Composite Hollow Fiber Membranes
THF	-	Tetrahydrofuran
TIPS	-	Thermal Induced Phase Inversion
TSA	-	Raw Natural Gas
UF	-	Ultra-Filtration
VIPS	-	Vapor-Induced Phase Inversion

LIST OF SYMBOLS

J - Diffusion coefficient [cm^{2}s^{-1}]

D, d - Diameter

l - Membrane thickness [cm]

v - Velocity

p - Pressure

P - Permeability [Barrer]

S - Solubility coefficient [cm3(STP)cmHg-1]

α - Ideal selectivity [-]

V_o - Dope extrusion velocity from spinneret

V_f - Take-up speed

$\mathrm{V_{bore}}$ - Bore fluid rate

P_i/l - Gas permeance of a membrane [GPU]

Q - Volumetric flow rate of the permeated gas [cm^3/s, STP]

A - Effective membrane area [cm^2]

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LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

The word membrane was derived from the Latin word "membrana," which means "skin" or "thin film." Membrane is a term used in science to describe a semipermeable solid or fluid layer that acts as a selective barrier or a boundary between two phases or mixtures. In other terms, a membrane is a selective barrier to the passage of molecules and ionic species in the liquid and vapor phases. When one component of a combination goes quicker across the membrane than the other, separation occurs.

Nowadays, the application of the membrane in various types and shapes is crucial and viral. In fact, new modern life is no longer possible without consideration of role membrane in domestic applications, industries, and even medical usages. All these have not been achieved if a pioneer discovery has not achieved during the 18th century. A French priest who was a physicist named Jean-Antoine Nollet (1700-1770) has noticed a recent named phenomenon as osmosis. His passion in 1747 was followed by a description for permeation of water through a semipermeable membrane (Sanders et al., 2013).

Membranes are used in a wide range of fields in science and industry, including gas separation applications for acid gas removal from natural gas, oxygen, and nitrogen production from air, and ethylene separation from emissions in polyethylene production units), environmental optimization, seawater desalination, hemodialysis, filtration process for ground and underground waters, filtration of industrial wastes water, and even health care industry. A membrane is a semi-permeable barrier that separates fluid mixtures by modulating the rate of movement of specific components from the mixture through the membrane, as stated previously. Membrane technology has been around since the 1960s. The importance and application of membrane in gas separation have been noticed earlier. Furthermore, gas permeation using membrane was conducted by J.K. Mitchell back in 1831 (Qian et al., 2022; Wisniak, 2013).

Membrane-based separation techniques have several advantages over traditional separation methods, including energy savings due to the lack of phase shifts known as low energy consumption (Waqas et al., 2020). Membrane separation does not need any high energy-consuming processes such as heating or changing phase processes; therefore, less energy is consumed in this process. (Tsai et al., 2019). More membrane separations do not require any chemical reaction, thus it leads to simplicity. (Kurniawan et al., 2021; Yuliwati et al., 2011) The ability to separate temperature-sensitive solutions is another key feature of membrane separations because the separation is being performed at room temperature (Huang et al., 2021). The physical and physicochemical features of membranes are very changeable and can be tailored to meet specific requirements (Goh et al., 2015; Turken et al., 2019). Removal of a wide spectrum of pollutants from particles, from viruses to ionic species (Ashrafizadeh & Khorasani, 2010; Tsai et al., 2008; Wei et al., 2013). Cost-effectiveness, high selectivity, simplicity of scaling up, and capacity to combine with other processes are all known advantages at the moment (Feng et al., 2013; Hewawasam et al., 2018). Low time consumption, ease of operation, a simple and efficient device, and a small footprint (Haase et al., 2017; Zhu et al., 2017) are some of the other advantages of membrane separation that have been studied extensively by academics and continue to pique their interest.

One of the most important advancements in the membrane separation process is the analysis and investigation of the physicochemical parameters and performances of a membrane. To achieve the best separation results, the membrane must have physical qualities that allow for optimal interactions with the solution in the process stream. Microscopic observation is the most effective method of investigating physical qualities. Surface morphology, the minimum attachment of solutes, and scattered components to the membrane surface are all factors that influence membrane fouling.

All mentioned properties are highly dependent on the conditions and stability features of the fabrication of membrane. In other words, to obtain high quality and stable membrane, a reliable machine is needed to produce the membrane, which was emphasized by all researchers since the discovery of membrane.

In modern categories for membrane science, there are two types: flat sheet and hollow fibers. Both types have their application based on the properties in various fields, science, and industries along with their advantages and their disadvantages. Until the first synthetic nitrocellulose membranes were successfully fabricated in laboratories in the middle of the 1900s, researchers usually interacted with biological membranes made of animal parts such as bladders of pigs, cattle, or fish, and sausage casings derived from animal gut, and plant origin such as onion. (Baker, 2012; Dai et al., 2016)

Membrane-based gas separation technology, in general, has numerous advantages over traditional unit operation systems. Because capital expenditure may be dependent on flowrate or throughput, membrane-based gas separation systems have proven to be affordable and significantly less expensive to operate. Because the system is modular and compact, it requires little room and can occupy a larger area. The equipment is lightweight and easy to transport and install due to its modularity, making it ideal for isolated places such as offshore installations. The operational system is way less complicated. Because there is no phase change and no movable or rotary in the system, it requires minimum oversight and maintenance. Furthermore, it consumes very little energy, as it does not require the use of electricity or combustion fuels. Except for membrane contactor systems, the system does not need any solvents or solid sorbents. Last but not least, the system has been demonstrated to be safe, with minimum environmental impact due to the absence of hazardous or toxic wastes. Air separation and carbon dioxide removal are two important challenges in membrane-based gas separation.

Cryogenically liquefying ambient air is a common method of obtaining widely used commodity gases like nitrogen, oxygen, and argon. A typical nitrogen separation from air by membrane process uses an 8-10 atm feed that passes through a series of hollow-fiber modules. The feed is routed via a membrane that preferably allows oxygen to flow through. Purified nitrogen in the residual can have a purity of up to 99.5 percent (Wallace et al., 2006). The biggest disadvantage of nitrogen enrichment from air, on the other hand, is the high expense of compressor operations to achieve improved nitrogen purity, pressurize the air stream. If the membrane employed has a better selectivity, the compressor's size can be lowered. The implementation of a membrane system will significantly reduce the operational costs of compression. In other words, increasing the selectivity of the membrane and improving its quality and performance by controlling the production parameters can achieve a higher rate of acceptance and application in low-cost demands.

Carbon dioxide removal is required in a variety of industries, including pipeline grade natural gas production, enhanced oil recovery (EOR), methane retrieval from landfills and biogas, carbon dioxide recovery from the steam reforming process and flue gases. Most membrane scientists across the world are particularly interested in removing carbon dioxide in natural gas streams. Hydrogen sulfide and water are also impurities in natural gas resources, the elimination of carbon dioxide and hydrogen sulfide is the most important goal in natural gas processing. Carbon dioxide is very undesirable in most gas streams since it might severely damage pipelines in the contact with water, whereas hydrogen sulfide is also mortally deadly. Simultaneously, high-value gas fuel can be reclaimed while gas impurities are reduced to the desired level. Raw natural gas is mostly made up of methane and is extracted from underground sources. Natural gas also contains higher hydrocarbons, or natural gas liquids (NGLs), which are referred to as "the heavies" in the natural gas processing industry. Ethane (C_2H_6), propane (C_3H_8), butane (C_4H_{10}), and natural gasoline are the most common heavier hydrocarbons. These gases have been shown to have a negative impact on membrane separation efficiency and membrane life span. Absorption processes, such as the BenfieldTM process (hot potassium carbonate solutions) and amine scrubbing; cryogenic processes; and adsorption processes, such as pressure swing adsorption (PSA), thermal swing adsorption, have all been used in the past to remove carbon dioxide from raw natural gas (TSA). These traditional unit processes used a lot of energy for gas processing, solvent regeneration, and absorbent material replacement, as well as transportation costs. The world's largest membrane unit, able to process 680 MMscfd of gas, was deployed offshore Malaysia in March 2007. In comparison to traditional separation systems, this installation provides for a significant cost, weight, and footprint savings (Zulhairun et al., 2015).

Membrane separation methods have become one of the new technologies that have seen remarkable expansion over the last few decades. It has suffocated global attention, particularly in the field of filtration technologies. Determination of fabrication conditions such as polymer flowrate bore flowrate and other factors are the most significant approach to obtain higher quality and reliable hollow fiber membranes (HFMs). Various investigations have been conducted. Performance tests mostly study the permeance and selectivity of the membrane for different gases. For characterizing the structure and morphology of HFMs, the scanning electron microscope (SEM) is the most extensively used method. Many techniques are available for the fabrication of the hollow fiber membrane, and also many types of analysis attempted with the previous researcher through the history form trial and error approaches, random test to analytical and simulation.

Recently, automation and increasing the accuracy of the production is the key plan and only solution in most industries. Conducting this can be followed by less human error and consistency in the performance and quality of the product. It is not hidden from anyone that automated fabrication and production line have increased the productivity and decreasing cost of the production significantly in the last decades and still improving.

In general, creating a high-performance membrane entail using trial-and-error approaches to evaluate models, which include determining the best material composition, selecting essential operating factors, and ensuring optimum fiber spinning conditions. Besides these facts, a reliable fabrication system, which is able to control and monitor the parameters during the production of hollow fiber membranes, is necessary, which can be so effective and a great help to approaching higher performances in membranes. Biogas, which contains 60 to 70 percent CH_4 and 30 to 40 percent CO_2, is considered an essential renewable power supply due to the finite nature of fossil fuels. Nonetheless, in order to improve the energy grade, minimize pipeline corrosion, and decrease climate change, the unavoidable impurities of biogas, particularly known as CO_2, should be eliminated (Gong et al., 2020). Despite the fact that a lot of work has been done in the past, evaluating membrane performance for a specific application is still very challenging, and there are so many factors that should be considered during membrane fabrication and a great deal of assumptions can be assumed for this evaluation.

The membrane performance evaluation in this study was focused on the fabrication conditions of the membrane during the process of manufacturing in order to gain higher performances and optimized product in industry-grade and reproducibility of the same properties in membrane fabrication. Several factors involve in this property as mentioned influence the performance and properties of the membrane. In this study, an experiment was conducted to find effective factors and develop an instrumentation and control system to obtain highly reproducible membrane fiber with consistent properties for gas separation usage.

1.2 Problem Statement

As reported in most previous researches on membrane manufacturing systems, these usually involve the use of one process factor at a time in the experimental method, for example, the effects of factors are investigated individually, which is not only time-consuming, but also expensive, and does not guarantee the characteristics of the manufactured product. Furthermore, a fully automated system is highly beneficial in order to control the hollow fiber spinning manufacturing process conditions and to be sure the parameters are always as same as before in order to have a reliable test condition and easier to compare the results. Therefore, having an instrumented and automated system is so crucial.

The development of a fast and reliable procedure for determining the performance and attributes of a hollow fiber membrane has become critical in recent years. The production of HFM material is extremely delicate because the end result must be of satisfactory value and suitable for usage in the potential uses. As a result, a thorough investigation should be carried out to discover the elements that are particularly connected to the properties of HFM during membrane manufacturing, such as dope flowrate, bore flowrate, air gap distance, coagulant bath temperature, and take-up speed. In addition, inequity of hollow fiber physical properties is a factor that should be considered as well. Changes in membrane physical structure (e.g., outer diameter) during spinning are other factors that make complexity, and they should be monitored and finely controlled. Many dependent parameters co-exist during the fabrication of the hollow fiber membrane, and the effects of each parameter are not totally understood until now. Controlling these parameters may result in membranes with the appropriate performance and physical features, hence knowing the key mechanisms in the development of hollow fiber membranes is of tremendous importance. In the absence of automation and quality control, the challenges of HFM manufacturing are restrictedly centered on the membrane performance and reliability of the system (Park & Kim, 2008). The modeling of the membrane is another era, which is attempted by various methods in order to predict the performance before the production of membrane. Achieving a simple model able to show effects of all parameters simultaneously on performances of membrane to make an easier illustration for depended parameters. These parameters are monitored and checked throughout the manufacturing process, allowing us to develop a variety of hollow fiber membranes with varying features for various applications with the appropriate attributes and performance.

Moreover, in any kind of manufacturing and fabrication system, there is a basic need to have very high reproducibility. It is the other factor that should be considered deeply and accurately to fulfill the aim of the system to achieve a high-performance membrane at any time and in any conditions.

To develop and build a membrane for a particular application, a method of learning by making tries and learning from the outcomes, trial-and-error, and error process, has traditionally been used. Membrane manufacture and membrane testing or characterization are the two processes in the iteration. The first step is to prepare a membrane sample. The second step examines and interprets membrane performance using theoretical, mathematical, equation, and experimental data. After the performance parameters have been determined, the actual and target membrane performances are compared. Typically, the results differ greatly from one another. As a result, a fresh membrane sample with unique properties must be created and examined. These two stages are repeated until the membrane characteristics and performance goals are met. Because large quantities of experimental data are necessary, such a technique is time demanding. Furthermore, due to material waste, it is very expensive.

One of the goals of this research is to reduce the number of iterations involved in membrane production for a specific application. Theoretically, the newly developed system is able to control the manufacturing parameters to reach the desired properties such as outer diameter, permeance, selectivity, jet ratio. Therefore, the number of iterations will be reduced considerably. The proposed fabrication system will be developed with the ability to produce the membrane with the same specifications with desired quality.

In this studied approach of membrane design and fabrication, an automated system is designed for controlling the parameters during fabrication to gain a satisfactory level in performance and significant characteristics of the hollow fiber membrane. The manufacturing of the membrane is refined such that parameters of the membrane fabrication keep stable and remain constant reliably. A mathematical model and the predicted membrane parameters are used to predict membrane performance, in this study: gas flux and selectivity. Design of experiment (DOE) have been successfully used for solving a wide variety of problems; Hence, an iterative procedure is not required in the design of the membranes for specific application. A full factorial DOE also shows all relations between the parameters. For each input parameter, a model is obtained for its effect of performance and an optimized condition for the specific membrane is achieved. The advantages of this approach over the traditional approach are as the following: Ideally, only one iteration is required; less cost and waste on material; and, the design of an optimum membrane for a specific application can be achieved in considerably less time. To summarize, the aim is to develop a high-precision spinning machine for producing highly reproducible membranes with constant desirable properties.

1.3 Research Objectives

The overall purpose of this study is to model the fabrication of hollow fiber membrane and to design and develop an automated fabrication system for producing highly reproducible membrane, which will be covered by the following objectives:

(1) To design and develop a precision automated hollow fiber spinning machine equipped with a controller and an instrumentation system integrated with sensors and actuators for producing polymeric membrane.

(2) To optimize spinning parameters for non-porous membrane for gas separating applications by tuning its parameters based on the design of experiments (DOE) methodology to determine the suitable model and index for best performance based on the fabrication conditions.

(3) To compare the performance of the optimized spinning parameters using the automated procedure as compared to literatures in terms of performance improvement and reduction of extensive iteration needed to achieve the result more efficiently.

1.4 Research Scopes and Limitations

Any study must be constrained by certain sufficiently defined scopes in order to fulfill its aims. This study is also not an exception to the rule. The scopes 1 to 3, 4 to 9 and scopes 10 and 11 are here to fulfil objectives 1 to 3 respectively. The following are this study's scopes and limitations:

(1) Programmable logic controller and sensors which are temperature, humidity, pressures and flow rate only are integrated within the testing.

(2) Designing and developing power and signal conditioning boxes for HFM manufacturing according to Advanced Membrane Technology Research

Center (AMTEC) needs base on the available and existed manual machine to be improved.

(3) Designing and developing a fabrication system integrated with sensors and actuators for the manufacturing process.

(4) This research conducted only using polymer dope solution comprised of polysulfone (PSF) Udel P3500, N,N-dimethylacetamide (DMAc), tetrahydrofuran (THF), and ethanol with weight percentages of 30%, 35%, 30%, and 5%, respectively. Fabrication process was only using dry-wet spinning methods.

(5) During the fabrication process, the effective parameters on mechanical properties such as ($x1$) flow rate of dope and ($x2$) bore fluid composition, ($x3$) spinning drum speed and ($x4$) air gap distance are changed manually using the automated system between $1< x1 <2$ ml/min, $0.33< x2 <0.67$ ml/min, $9.1< x3 <11.7$ m/min and $1< x4 <4$ cm respectively. Other parameters are assumed to be constant.

(6) Applying the pure gas flux test, to evaluate the performance of the membranes for CO_2/CH_4 separation.

(7) This work is focused on available membrane performance factors which are selectivity, permeance and diameters.

(8) The model of system is developed using the full factorial method of design of experiment (DOE) to estimate the properties of HFM

(9) Optimization of the dry-jet wet spinning process parameters by varying the air gap distance, force convection flow rate, dope extrusion rate, and the take-up speed for the fabrication of hollow fiber membranes using DOE

(10) Model verified only using the experimental tests and validated by comparing with benchmark of previous literatures.

(11) Analysis, verifying properties of the produced membrane using scanning electronic microscope (SEM) to determine the defects to verify the outcome.

1.5 Significance and Novelty of the Research

The high performance for permeance and selectivity of the membrane are the most crucial factors of the membrane properties. Without an accurate shape, processing other properties of the membrane is useless; therefore, this study is focused on measuring and controlling this factor, which can be the first step of the production of a high-performance membrane. Studying the characteristics and performance of HFM will provide useful clues and insights for designing and developing a revolutionary automated manufacturing system with the goal of increasing the system's productivity, efficiency, and quality of HFM production. The suggested system's mechatronic design and development will unavoidably necessitate the comprehensive integration of numerous mechanisms, sensors, actuators, electrical/electronic circuitries, and controller inclusive programable logic controller (PLC)-based system with a user-friendly interface. The systematic method for study and optimization of the performance and process also is beneficial due to making the process simpler to understand for researchers and giving them ability to manipulate the condition, as they desired to reach what they are looking for. A correlation between the two techniques has never been established or ascertained in literature to the best of the author’s knowledge. In addition, the performance of membrane will be rigorously studied via DOE. In summary, the study aims to achieve the high-precision spinning machine in producing highly reproducible membranes that can satisfy desirable properties and a method to making index and simple model for any type of membrane.

In simple words, it has been tried to find an easier way and a methodology to index and find the performances and characteristics of HFM with fewer iterations and more systematically. Meanwhile, an instrumented hollow fiber fabrications system able to spin all possible variances of other types of membranes will be designed and manufactured for further researchers.

Although many studies have been conducted in the manufacturing and production of membrane, but there is a lack of comprehensive study by using a systematic method in order to optimize its operating process, and there is still a gap that can be filled more and deeply. Meanwhile, the unavailability of an automated control system for producing hollow fiber membranes with more accuracy to gain higher performances on membranes and the ability to set the parameters accurately and reliable is caused to study more in this field.

The following questions remain about membrane manufacturing innovation:

(1) Can manufacturing limitations lead to development and change in membrane performance?

(2) What are the potential performance improvements possible with manufacturing innovation?

(3) Is it able to implement other manufacturing technique from one field and apply it into manufacturer membranes systems to achieve better performances?

(4) Is it reevaluating how to make membrane structures by developing fabricating controls that are as of now inaccessible with regular conventional methods is possible?

In other words, by consideration of widespread usages and applications of HFM in industries and progressively increase of the demand for them because of new modern technologies and global energy and resources concerns, having a study in such a way that helps to produce more reliable HFM using recent and automated techniques and modeled and predict the behavior of new combinations using a standard statistical method to achieve sustainability and reproducibility was the motivation for this study base on the literature. This type of approach was not commonly considered by other researchers to study the effect of chemical properties control, instrumentation, and statistical analysis simultaneously.

This research has contributed greatly to the field of membrane in terms of building an automated manufacturing machine to be able to conduct the production easier and more accurate, and testing new statistical approach to obtain a mathematical model for prediction of the performance of membrane without conducting extra actual experiments tests. Last but not least, it evaluates the new optimization method for production of hollow fiber membranes.

1.6 Thesis Organization

Original research and fresh perspectives on gas separation polysulfone membranes with an automated design fabrication technology are described in this thesis. This thesis is organized into five chapters. The first chapter covers the fundamentals of membrane separation processes. The background of membrane technology is discussed, as well as the issues that led to the current inquiry. The research objectives are determined, followed by a scope of work that has been written out in a systematic manner in order to fulfill the study's goal.

In Chapter 2, which addresses the concept of gas separation membranes, basic concepts of gas transport across membranes, materials, structures, and manufacturing procedures, a related evaluation of the scientific literature review is offered.

Chapter 3 presents the materials and experimental methods applied throughout the study. The designed automated fabrication system has been described. The methods for membrane fabrication in hollow fiber configurations are described, followed by relevant characterizations procedures and analysis assumptions.

Chapter 4 focuses on optimizing hollow fiber manufacturing characteristics including effects of dry gap height, force convection flow rate, dope extrusion rate, and take-up speed on selectivity, permeance of gases, and diameter. This followed by finding the mathematical model and experimental confirmation test and comparison of the achieved results with previous findings. The goal of this research is to find the best spinning conditions for hollow fiber membranes that are defect-free and have appealing permeance and selectivity combinations. DOE is used to investigate the effects of varied spinning conditions on membrane performance and gas permeation behavior.

Finally, Chapter 5 concludes the key findings from the present work as well as provides a list of recommendations for future research.

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LIST OF PUBLICATIONS

Indexed Journal

1. Khademi, S., Zaurah, I., Darus, M., Mailah, M., Jye, L. W., & Khademi, A. (2021). Modeling and Optimization of Spinning Parameters on Selectivity of Polysulfone Hollow Fiber Membrane for CO2/CH4 Separation. Makara Journal of Technology, 25(3), 111–118. (Indexed by WOS) https://doi.org/10.7454/mst.v25i3.3865

Journal with Impact Factor

1. Roslan, R. A., Lau, W. J., Sakthivel, D. B., Khademi, S., Zulhairun, A. K., Goh, P. S., Ismail, A. F., Chong, K. C., & Lai, S. O. (2018). Separation of CO2/CH4 and O2/N2 by polysulfone hollow fiber membranes: Effects of membrane support properties and surface coating materials. Journal of Polymer Engineering, 38(9), 871–880. (Q1, IF: 1.367) https://doi.org/10.1515/polyeng-2017-0272

Non-Indexed Conference Proceedings

1. Khademi, S., Zaurah, I., Mailah, M., & Jye, L. W. (2019). Optimization and Modelling of Spinning Parameters on Selectivity of Hollow Fiber Membrane Using Automated Hollow Fiber Fabrication System in Separation of Co2 and Ch4. Intelligent Control and Automation Symposium (ICAS 2018), 1(3), 83–89. https://easychair.org/cfp/icas2018

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4. Abbasgholipourghadim M.,Musa M., Intan Z. M. D., A. F. Ismail, Rezaei dashtarzhandi M., Abbasgholipourghadim M., Khademi S., (2015). Porosity and Pore Area Determination of Hollow Fiber Membrane Incorporating Digital Image Processing. Recent Advances in Mechanics and Mechanical Engineering Intelligent, 118–123. http://www.wseas.us/e-library/conferences/2015/Malaysia/MECH/MECH-18.pdf