EFFECT OF OPERATING PARAMETER ON MEMBRANE DISTILLATION PERFORMANCE USING KAPOK FIBRE FOR HUMIC ACID TREATMENT

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A thesis submitted in fulfilment of the requirement for the award of the Degree of Master of Mechanical Engineering

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MAY 2021

Special for my parent, Zulkefli bin Baharuddin and Noraidah binti Selamat

To my dearest husband,

Muhammad Adrian Thomas

To my beloved sibling

ACKNOWLEDGEMENT

Praise is upon Allah SWT, for without His grace and compassion, none of this would have been possible. I want to take this opportunity to extend my appreciation and thankfulness to my supervisor, Dr. Muhamad Zaini Bin Yunos for his kind guidance and support toward the accomplishment of this study.

The appreciation also goes to all parties whom and which are involved in completing this master project, especially to AMMC laboratory and University of Tun Hussein Onn Malaysia (UTHM). My sincere appreciation also extends to my lovely family, especially to my beloved father, Zulkefli bin Baharuddin and my beloved mother, Noraidah Binti Selamat, my beloved mother in-law Antipolo R. Sulbiano also to my dearest husband, Muhammad Adrian Thomas as with their understanding, support and prayer during the completion of this project. Last but not least, I would like to thank also to all my UTHM comrades.

With Allah's willing, such enduring aids and advices have given by all of you definitely had contributed a lot toward building up my confidence, to produce the best in this study.

ABSTRACT

There is no doubt that in finding solution to the problem of fresh water scarcity and its high demand. Membrane distillation have been shown to be an effective alternative to replace conventional technologies which found uneconomical. As agricultural waste is abundantly available, kapok fibre has been chosen an alternative solution for synthetic membranes which not environmentally friendly. This study aims to determine the physical properties of raw kapok fibre in the membrane distillation system, to investigate the effect of operating parameters in membrane distillation for humic acid in terms of water permeability and rejection rate, and finally to determine the reusability of kapok fibre in the membrane distillation system. The physical properties of the raw kapok fibre have been determined with respect to morphology, absorption test, and water contact angle method. Then, it followed by investigating the performances of the kapok fibre through membrane distillation process with three different operating parameters. Next, reusability test was conducted through the membrane distillation system for about 20 hours. Characterisation test revealed the linkage structure of kapok fibre, the low increment of moisture content in the raw kapok fibre and the water contact angle exhibits 152.704°. Meanwhile, the first operating parameter used in membrane distillation system, which is the temperature of feed humic solution at 60°C has produced the highest permeate flux of 0.629 kg/m²h followed by 98.753 % of rejection at 40°C. Then, the lowest feed concentration of humic acid solution which 0.1 g/L showed the highest permeate flux of 0.634 kg/m²h followed by 97.47 % rejection rate. The 5 g of kapok fibre shows the highest permeate flux with 0.803 kg/m²h and 35g of kapok fibre shows highest rejection rate which 98.91%. The long-time operation has resulted in a slow decrease of permeate flux from 0.321 kg/hm² to 0.089 kg/m²h after 20 hours. Therefore, the excellence hydrophobic property of the kapok fibre has successfully created an alternative solution for synthetic membrane studies in the membrane distillation process.



ABSTRAK

Kekurangan sumber air bersih dan mempunyai permintaan yang tinggi pasti mempunyai jalan penyelesaian. Penyulingan membran telah terbukti menjadi alternatif yang berkesan untuk menggantikan teknologi konvensional yang didapati memerlukan kos yang tinggi. Oleh kerana terdapat banyak sisa pertanian yang tidak digunakan, serat kapok telah dipilih sebagai penyelesaian alternatif untuk membran sintetik yang bersifat tidak mesra alam. Kajian ini bertujuan untuk mengetahui sifat fizikal serat kapok asli dalam sistem penyulingan membran, bagi mengkaji kesan parameter dari segi kebolehtelapan air dan pengasingan asid humik, dan akhir sekali untuk menentukan kebolehgunaan semula serat kapok dalam sistem penyulingan membran. Sifat fizikal serat kapok asli telah dikaji berdasarkan morfologi, ujian penyerapan, dan ujian sifat hidrofobik. Kemudian, diikuti dengan penyiasatan prestasi serat kapok melalui proses penyulingan membran dengan tiga parameter yang berbeza. Seterusnya, ujian penggunaan semula serat kapok dijalankan melalui proses penyulingan membran selama 20 jam. Ujian ciri fizikal menunjukkan struktur hubungan serat kapok, kenaikan yang rendah pada kadar kelembapan serat kapok asli dan sudut kontak air yang menunjukkan 152.704°. Sementara itu, parameter pertama pada 60°C menghasilkan wap air paling tinggi iaitu 0.629 kg/m²h diikuti dengan 98.753% pengasingan pada 40°C. Parameter kedua, wap air tertinggi iaitu 0.634 kg/m²h diikuti dengan 97.47% kadar pengasingan pada 0.1g/L asid humik. 5g serat kapok menunjukkan wap air paling tinggi dengan 0.803 kg/m²h diikuti dengan pengasingan paling tinggi iaitu 98.91% pada 35g serat kapok. Ujian penggunaan semula serat kapok menghasilkan penurunan wap air yang perlahan dari 0.321 kg/m²h kepada 0.089 kg/m²h selepas 20 jam. Oleh itu, sifat hidrofobik yang ada pada serat kapok telah menghasilkan penyelesaian alternatif kepada membran sintetik di dalam proses penyulingan membran.



CONTENTS

TITLE			i	
DECLARATIO	ON		ii	
DEDICATION			iii	
ACKNOWLEDGMENT				
ABSTRACT				
LIST OF TABLES				
LIST OF FIGURES				
LIST OF SYMBOL AND ABBREVIATIONS				
LIST OF APPENDICES CHAPTER 1 INTRODUCTION			XV	
CHAPTER 1 I	NTRO	DUCTION	1	
	1.1	Background of study	1	
	1.2 5	Problem statement	2	
DERM	1.3	Objective	4	
	1.4	Scope of the study	4	
1	1.5	Significant of the study	5	
CHAPTER 2 I	LITEF	RATURE REVIEW	6	
	2.1	Membrane distillation	6	
2	2.1.1	Membrane distillation configuration and module	8	
2	2.1.2	Membrane material used in the membrane		
		distillation system	14	
2	2.1.3	Operating parameter of membrane distillation	17	
2	2.1.4	Application in membrane distillation	19	
	2.2	Natural fibre	20	

	2.2.1	Kapok fibre	22
	2.2.2	Application of kapok fibre	25
	2.3	Humic acid	27
	2.4	Related works	29
CHAPTER 3	8 METH	IODOLOGY	34
	3.1	Research method	31
	3.2	Preparation of raw materials	36
	3.3	Characterisation of kapok fibre	36
	3.3.1	Morphology test	37
	3.3.2	Water absorption test	39
	3.3.3	Water contact angle test	39
	3.4	Effect of operating parameter toward kapok fibre	
		in membrane distillation process	40
	3.4.1	Effect of feed temperature on permeate flux	43
	3.4.2	Effect of feed solution concentration on permeate	44 A H
		flux	44
	3.4.3	Effect of the amount of kapok fibre on permeate	
		flux	45
	3.4.4	Humic acid rejection test	46
	3.5	Reusability test	48
CHAPTER 4	DATA	ANALYSIS AND RESULTS	50
	4.1	Introduction	50
	4.2	Characterisation of kapok fibre	51
	4.2.1	Morphology test	51
	4.2.2	Absorption test	53
	4.2.3	Water contact angle test	54
	4.3	Effect of operating parameter towards membrane	
		distillation process	55
	4.3.1	Effect of feed temperature in membrane distillation	
		process	56
	4.3.2	Effect of feed concentration in membrane	
		distillation process	65

viii

	4.3.3	Effect of kapok fibre mass in membrane distillation	on
		process	73
	4.4	Reusability Test	80
	4.4.1	Effect of kapok fibre in reusability test towards	
		morphology surface	81
	4.4.2	Effect of kapok fibre in reusability test towards	
		contact angle	83
	4.5	Summary	83
СНАРТЕ	CR 5 CONC	CLUSION AND RECOMMENDATION	85
	5.1	Conclusion	89
	5.2	Recommendations	87
REFEREN	NCES		89
APPEND	ICES		96

ix

LIST OF TABLES

2.1	1 Advantages and disadvantages of membrane distillation for		
	each configuration	12	
2.2	Configuration of membrane distillation	13	
2.3	Morphologies of commercially available membrane distillation		
	membrane	16	
2.4	Effect of temperature on permeate flux	18	
2.5	Effect of concentration on permeate flux	19	
2.6	Application of membrane distillation towards wastewater treatment	20	
2.7	Physical properties of raw kapok fibre	24	
2.8	Summary of related work from previous researchers	32	
3.1	Humic acid solution concentration	36	
3.2	Test conditions for the effect of feed temperature on permeate flux	44	
3.3	Test conditions for the effect of feed solution concentration on		
	permeate flux	44	
3.4	Test conditions for the effect of kapok fibre amount on permeate		
	flux	46	
4.1	Optical microscope image of kapok fibre surface layer at		
	various feed temperature after membrane distillation process	64	
4.2	Optical microscope image of kapok fibre surface layer at		
	various feed concentration after membrane distillation process	51	
4.3	Optical microscope image of kapok fibre surface layer at		
	various kapok fibre mass after membrane distillation process	57	

LIST OF FIGURES

2.1	Schematic diagram of membrane distillation operation	7
2.2	Configuration of membrane distillation	8
2.3	Schematic diagram of DCMD experimental set-up	9
2.4	Schematic diagram of AGMD experimental set-up	9
2.5	Schematic diagram of VMD experimental set-up	10
2.6	Schematic diagram of SGMD experimental set-up	11
2.7	Type of membrane material	15
2.8	Classification of natural fibre	22
2.9	Image of kapok (a) tree (b) leaf (c) immature and (d) mature pod	23
2.10	SEM images of (a) single and (b) cluster kapok fibre	25
2.11	Representative relationship between structure properties and	
	applications of kapok fibre in membrane distillation system	27
3.1	Overall research methods	35
3.2	JSM 6380LA Scanning Electron Microscope	37
3.3	Fision SEM coating system	38
3.4	Nikon Eclipse LV150 Microscope function to magnify surface	
	layer on used kapok fibre.	38
3.5	Experimental setup of the distillation process	41
3.6	Experimental setup of kapok fibre distillation modules	42
3.7	Hot feed tank at various temperatures	43
3.8	Hot feed tank at various feed solution concentration	45
3.9	Distillation module at various mass of kapok fibre	46
3.10	GENESYS 10S UV-Vis Spectrophotometer	48
4.1	SEM image of single raw kapok fibre	51
4.2	SEM image on surface of raw kapok fibre	52

4.3	Image of optical microscope of raw kapok fibre	53
4.4	Water absorption percentage of kapok fibre	54
4.5	Image of water droplet of raw kapok fibre	55
4.6	Effect of feed temperature at 40°C, 50°C and 60°C on permeate	
	flux at 0.1g/l of feed humic acid solution	57
4.7	Schematic diagram of humic acid and vapour molecular	
	movement toward the distillation process	58
4.8	Percentage of rejection rate of humic acid solution at 40°C, 50°C,	
	and 60°C of feed temperature	59
4.9	Schematic diagram of arrangement kapok fibre in distillation	
	module in (a) top view and (b) side view	60
4.10	Schematic diagram of the affected area of feed temperature toward	
	humic acid retention from (a) top view (b) side view	61
4.11	Contact angle measurement of kapok fibre surface after membrane	
	distillation process at various feed temperature	63
4.12	Effect of feed concentration of humic acid on permeate flux	
	with a constant temperature of 60°C	66
4.13	Concentration profile with concentration polarization	67
4.14	Percentage of rejection rate of humic acid solution at 0.1 g/L,	
	0.2g/L, 0.3g/L and 0.4g/L of feed concentration.	68
4.15	Schematic diagram effect of feed concentration toward humic	
	acid retention at (a) high concentration (b) low concentration	69
4.16	Contact angle measurement of kapok fibre surface after	
	membrane distillation process at various feed concentration of	
	humic acid solution	71
4.17	Effect of mass of kapok fibre on permeate flux at 0.1g/l and 60°C	74
4.18	Percentage of rejection rate of humic acid solution at 5g, 15g, 25g	
	and 35g of kapok fibre	75
4.19	Schematic diagram of effect of (a) high and (b) low kapok fibre	
	Amount towards humic acid retention	76
4.20	Contact angle measurement of kapok fibre surface after membrane	
	distillation process on various kapok fibre mass	78

xii

4.21	Reusability test of kapok fibre in membrane distillation	81
4.22	Optical microscope image of used kapok fibre in reusability test	
	of membrane distillation; (a) bottom (b) middle and (c) upper layer	
	of kapok fibre	82
4.23	Image of water droplet of used kapok fibre through reusability test	83

xiii

LIST OF SYMBOLS AND ABBREVIATIONS

CO_2	-	Carbon Dioxide
MD	-	Membrane distillation
RO	-	Reverse osmosis
DCMD	-	Direct contact membrane distillation
AGMD	-	Air gap membrane distillation
VMD	-	Vacuum membrane distillation
SGMD	-	Sweeping gas membrane distillation
F-TNF	-	Fluorinated-Titania nanofibre
SDS	-	Sodium dodecyl surface
NaCl	-	Sodium Chloride
PTFE	-	Polyetrafluoroethylene
TEG	-	Triethylene glycol
PP	-	Polypropylene
PVDF	-	Polyvinylidene fluoride
HNO3	<u>Y</u>	Nitric acid
HA	-	Humic acid
NOM	-	Natural organic matter
SEM	-	Scanning electron microscope
<i>CO2</i> +	-	Cobalt ions



LIST OF APPENDICES

APPENDIX	TITLE	PAGE
А	Result and example of calculation for water absorption test.	100
В	Result and example of calculation for effect of feed temperature in membrane	102
С	distillation process. Result and example of calculation for effect of feed concentration in membrane distillation process.	105
DRP	Result and example of calculation for effect of kapok fibre mass in membrane distillation process	108

CHAPTER 1

INTRODUCTION

1.1 Background of study

The scarcity for fresh and clean water is due to the heavy polluted water systems that has also impacted the climate change recently. Accordingly in 2019, a wildfire at Amazon, which is the largest tropical rainforest has burned [1]. The Amazon absorbs tons of Carbon Dioxide (CO₂) every year which tends to slow the global warming [2] as it can bring effect on biodiversity, oceans, human and weather with high content of CO₂ in the atmosphere. The significant impact of weather that brings drought and heatwaves will increase the demand for clean water continuously for the coming generations [3]. Therefore, as the population growth increases, this greatly impact the demand for quality and quantity of water resources. In addition, 70 % of the earth is covered with water and human beings think that is always more than enough for daily uses. However, 3 % of the water available on the earth is pure water and the remaining is unavailable to be consumed [4]. Thus, humanity faces challenges as they need to deal with polluted drinking water sources, which contain hazardous contaminants and faeces. Accordingly, these matters become an interest to many researchers to explore for different approaches for efficient water production technology.

The advanced technology of wastewater treatment is possible to discharge all contaminants in wastewater before it can be used as drinking water. However, the proposed advanced technology is expensive. Hence, membrane distillation (MD) is another reliable technology to enhance the treatment process of extremely polluted



water sources. Membrane distillation is a separating technology between two distinct types of miscible fluids at different temperatures through a hydrophobic membrane [5]. Simultaneously, the membrane is a thin barrier for a gas-liquid state that avoids the liquid to pass on the interface of the membrane. The membrane distillation process is operated with small and compact apparatus and also only low pressure required. Hence, the membrane distillation technology is more convenient and economical compared to other advanced and conventional technology [6].

In the membrane distillation process, the hydrophobic properties of the material or membrane used need to be stressed to avoid the liquid penetrating into the membrane but only water vapours are able to pass through the hydrophobic membrane [7]. This ensures the resulted permeate flux is clean and only pure water is produced. In this work, Ceiba Pentandra, or known as kapok fibre, has been proposed as a natural biopolymer for the distillation system. Additionally, the kapok fibre is well-known for its excellence hydrophobic and oleophilic properties [8]. These are the reason why the kapok fibre has been chosen as an alternative material to be applied for the membrane distillation system. Furthermore, humic acid was chosen to act as wastewater due to the distribution system of humic acid in the surface water bodies that can lead to the growth of bacteria. Hence, the membrane distillation process was conducted to produce pure and clean water.

1.2 Problem Statement

Throughout the world, it is assumed almost two million of people who depend on the source of drinking water is polluted that can spread various diseases and cause diarrhoeal deaths every year [9]. The availability of freshwater from river, streams and lake could not be rely on fully due to the water hygiene level since there are many contaminants specifically humic acid in the freshwater One of the pollution control measure is to remove all the contaminants specifically humic acid in the freshwater. A lack of clean water, which is free from contaminants that can be used has interfered in human beings' healthy daily life. Conventional methods such as gravity separation, dissolved air flotation, coalescence, centrifugation, flocculation and coagulation either fail to remove the contaminants cost-effectively to meet discharge standards. Over the

years, innovation surrounding water treatment systems has intensified, beginning with conventional media filters to high-efficiency centrifuge filters, disk filters, membrane filtration including micro and ultrafiltration and non-membrane filtration systems. Membrane distillation, are in fact a standout among the best options for wastewater treatment. However, the traditional materials employed, and conventional wastewater treatments have been unable to meet the environmental standards and hence, have been ineffective in addressing the removal of contaminants in some instances. Moreover, most of the materials used to produce the absorbents were produced from polymeric materials and industrial by-products which are quite expensive. Then again, the costly capital and working expense has been the prevention to the wide application of the membrane distillation advances. Regardless of the possibility that the application of membrane distillation of wastewater is reasonable, pre-treatment of the wastewater is frequently needed for forestalling untimely film fouling.

The interest in using natural fibres as raw materials has been growing rapidly due to the increasing awareness towards sustainability of the environment. Recently, over the last few years, bio-based materials have secured high demand in the market and industries. In this study, the use of kapok fibre in the membrane distillation process is proposed to improve the quality of freshwater for drinking. However, studies of kapok fibre in the membrane distillation process is yet to be focused on more. Therefore, the investigation by means of physical properties of the kapok fibre is crucial to ensure the material's suitability is feasible in the process. Kapok fibre shows great water repellence behaviour which suitable to be used in membrane distillation process and also well reusable trademark. Additionally, this membrane distillation process can separate the humic acid in freshwater and obtain clean water. The hydrophobic properties of kapok fibre ultimately prevent the humic acid from mixing with the clean water. Therefore, the separation of humic acid wastewater through the membrane distillation system via kapok fibre is carried out in various operating parameters to achieve the quality pure water.



1.3 Objective

The objectives of this research were stated as below:

- To determine the physical properties of raw kapok fibre as membrane in the membrane distillation system.
- (ii) To investigate the effect of operating parameters in membrane distillation for humic acid separation with respect of water permeability and humic acid rejection.
- (iii) To evaluate the reusability of kapok fibre as membrane in the membrane distillation system.

1.4 Scope of the Study

The scopes of the study were stated as below:

- (a) The morphology of raw kapok fibre before the membrane distillation process was observed through a scanning electron microscope (SEM) and optical microscope (OM).
- (b) The capability of kapok fibre to repel water was observed through the absorption test in humic acid solution and distilled water for 120 hours.
- (c) The water contact angle of kapok fibre before and after membrane distillation process was determined by using the Dino-Lite Digital Microscope.
- (d) The performance of the membrane distillation system by using kapok fibre as a barrier was evaluated by using various feed temperature of humic acid at 40°C, 50°C and 60°C.

- (e) The effect of different concentrations of humic acid was evaluated at 0.1 g/L, 0.2 g/L, 0.3g/L and 0.4 g/L in the distillation system performance toward permeate flux and percentage of rejection.
- (f) The amount of kapok fibre was varied at 5g, 15g, 25g and 35g in the membrane distillation module.
- (g) The reusability of kapok fibre during membrane distillation was conducted within the same membrane distillation unit and experimental procedure.

1.5 Significant of Study

The outcome of this research will contribute to the benefits of the inhabitants around the world bearing in mind that clean water is needed in daily routine especially for drinking. The higher demanding countries which are the most threatened by water shortage such as Libya, Western Sahara, Yemen, Djibouti and Jordan [9] to ensure they have adequate clean water source or supply to support the daily needs of the people and also for the food making sectors [4]. Specifically, food production industries may face a significant challenge in producing clean food if the water supply polluted and thus, this can further affect that the welfare of the community [10]. In short, water crisis contributes to health crisis, which can be reduced when there is a clean water source from each occupant house especially homes where children live. Moreover, for researchers, this research will be very interesting for them to discover a few potential ways to treat the freshwater and wastewater into clean, where more source of clean water can easily be accessed.



CHAPTER 2

LITERATURE REVIEW

2.1 Membrane Distillation

In the early stage of the membrane distillation technology in 1963, an organic membrane proposed in this application was a silicon rubber which has hydrophobic properties that is designed by an inventor, Bruce R Bodell. Then, in 1968, one more membrane distillation patent was introduced as an operation of mass transfer. After that, in 1999, Doig et al. verified that the silicon rubber used in the membrane distillation process does not give good results in the separation process [11]. In the same way, the effect of mass transfer through the hydrophobic silicon rubber membrane was studied. Recently, the study of membrane distillation process becomes an interest from year to year.

There are many conventional methods applied in separating trace elements from polluted water, for instance, coagulation, chemical precipitation and adsorption [12]. Nowadays, membrane distillation has become an interesting research among researchers due to the ability of the membrane distillation process to separate the highly contaminated water source. Membrane distillation is a method or technique that is widely used in the separation and treatment process, which apply temperature differences to produce pure water. The membrane distillation process is not only limited in the separation of water but also oil emulsion [13], liquid food product [14],



and heavy metal [15]. This economical technique only required small equipment for the process set up in ensuring the separation process generate a successful outcome.

Moreover, using low-cost material such as plastics, this system can still operate efficiently and besides, plastic is a durable corrosion-resistant material. Concurrently, the membrane distillation process is a safe process to carry out on account of low pressure and temperature applied during the operations compared to other systems. The range of feed temperature acquired for this system ranges from 40°C to 80°C, which does not to reach a boiling point. A schematic diagram of the membrane distillation process is illustrated in Figure 2.1. The membrane distillation, which is also called as thermal membrane separation operates in such a way that it involved the evacuation process from the hot side of the hydrophobic membrane (feed solution) to the other side of the membrane where the water vapour is collected.

In the membrane distillation system, there is a physical barrier called a membrane, where only water vapour is allowed to pass through it. Indeed, the used membrane has water repellent nature called the hydrophobic properties. The membrane material becomes a tremendous interest to many researchers either in inorganic materials which consists of ceramic, glass and metal or organic materials which consist of the polymer. These materials used as the membrane can result in a different characteristic due to the nature of the membrane itself. The vapour-liquid equilibrium principle has taken into account, where the rising temperature will result to higher vapour pressure or water vapour according to the Raoult's law [16]. However, there are many conditions in the membrane distillation system that can affect the resulted permeate flux, which will be discussed in detail in the next section.



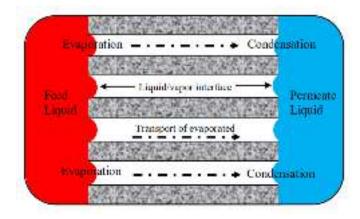


Figure 2.1: Schematic diagram of membrane distillation operation [6].

2.1.1 Membrane distillation configuration and module.

In this part, several arrangements of the membrane distillation are applied to produce vapour molecules through a hydrophobic membrane from feed solution will be discussed. Referring to permeate flux collected, membrane distillation configuration can be categorised into four groups as illustrated in Figure 2.2 which are direct contact membrane distillation (DCMD) [17], air gap membrane distillation (AGMD) [5], vacuum membrane distillation (VMD) [18], and sweeping gas membrane distillation (SGMD) [19]. Every membrane distillation configuration has different ways of permeating the water vapour.

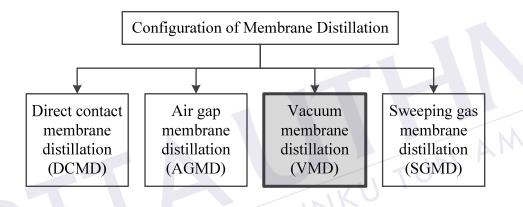


Figure 2.2: Configuration of membrane distillation.

The position of the membrane in the DCMD process is placed directly to the hot feed solution and the cold flow water on two sides of the membranes as shown in Figure 2.3. The changes in temperature of hot and cold stream resulted in pressure drop will produce the vapour [20]. In particular, this pressure difference phenomena causes the tendency for the molecules of the aqueous solution to escape as vapour. The resulted vapour penetrates through the pore of the hydrophobic membrane which then condenses in the cold surface of the membrane module [21]. According to Fan et al., the developed F-TNF membrane through the DCMD process exhibits high permeate flux which is more than 12LMH at 80°C followed by high salt rejection which is more than 99.92% [17].



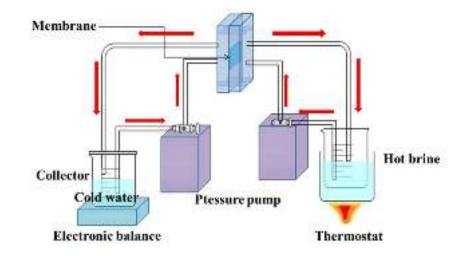


Figure 2.3: Schematic diagram of DCMD experimental set-up [17].

AGMD is a variety of membrane distillation, whereby in the middle of the hydrophobic membrane and a surface of condensation, there is an air gap where it creates a cool area by the flowing cold water. The vapour molecules are passed through the hydrophobic membrane, where it penetrates over the air gap area and then forming condensed vapour in the cold side of the membrane distillation unit [22]. According to Leaper et al., the separation of Sodium dodecyl sulfate (SDS) surfactant and Sodium chloride (NaCl) utilizing a commercial PTFE membrane shows high permeate flux which is 9.6 LMH (Liter/m²/h). The experimental setup for the PTFE membrane in AGMD configuration is illustrated in Figure 2.4 [23].

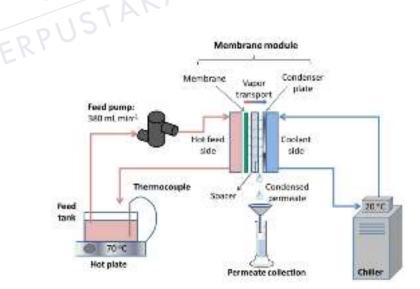


Figure 2.4: Schematic diagram of AGMD experimental set-up [23].

Compared to other configuration of the membrane distillation, the difference in VMD is the utilisation of a vacuum pump. Indeed, the membrane permeates sides which it contains vapour is where the process of suction occurred using the vacuum pump. The formed vapour, which penetrates through the membrane pores is sucked due to the pressure of the vacuum applied is way less than the saturation vapour pressure [20]. Then, the collected vapour is condensed in the condenser which is placed outside of the membrane module. The high-performance membrane used in saline-water distillation for treatment NaCl was investigated by using VMD process as illustrated in Figure 2.5. Thus, the maximum flux reached 27.28 kg/m²h at 75°C and the percentage of rejection was 99.8% [24].

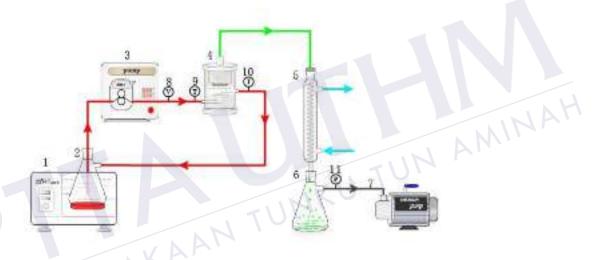


Figure 2.5 : Schematic diagram of VMD experimental set-up [24].

Meanwhile, the main difference in SGMD is the type of inert gas applied such as air or nitrogen. The vapour released at the membrane permeates side is carried by the inert gas to an external chamber which placed outside of the membrane module and the condensation process takes place after [25]. Figure 2.6 shows the SGMD process to determine the ability of intensification process of triethylene glycol (TEG) from binary solutions [19].

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