

FABRICATION OF ASYMMETRIC NANOFILTRATION MEMBRANE
USING BINARY, TERNARY, AND QUATERNARY COMPONENT

NORLISA BINTI AHMAD SUBKI

FAKULTI SAINS DAN TEKNOLOGI
UNIVERSITI SAINS DAN TEKNOLOGI MALAYSIA

2005

LF
90
FDN
91
2005

FABRICATION OF ASYMMETRIC NANOFILTRATION MEMBRANE USING
BINARY, TERNARY AND QUATERNARY COMPONENT

By

Norlisa binti Ahmad Subki

Research Report submitted in partial fulfillment of the requirements for the degree of
Bachelor of Technology (Environmental)

Department of Engineering Science
Faculty of Science And Technology
KOLEJ UNIVERSITI SAINS DAN TEKNOLOGI MALAYSIA
2005

Name: Norlisa binti Ahmad Subki

Copy Name:

LP
24
FST
5
2005

1100036916



PENGAKUAN DAN PENGESAHAN LAPORAN
PROJEK PENYELIDIKAN I DAN II

Adalah ini diakui dan disahkan bahawa laporan penyelidikan bertajuk:

FABRICATION OF ASYMMETRIC NANOFILTRATION MEMBRANE USING
BINARY, TERNARY AND QUATERNARY COMPONENT

oleh Norlisa Bt Ahmad Subki No. Matrik UK 6583 telah diperiksa dan semua
pembetulan yang disarankan telah dilakukan. Laporan ini dikemukakan kepada
Jabatan Sains Kejuruteraan sebagai memenuhi sebahagian daripada keperluan
memperolehi Ijazah Sarjana Muda Teknologi (Alam Sekitar), Fakulti Sains dan
Teknologi, Kolej Universiti Sains dan Teknologi Malaysia.

Disahkan oleh:

Penyelia Utama

Nama: Dr Nora'aini bt Ali

Cop Rasmi: DR. NORA'AINI BINTI ALI
Pensyarah

Jabatan Sains Kejuruteraan
Fakulti Sains dan Teknologi
Kolej Universiti Sains dan Teknologi Malaysia
21030 Kuala Terengganu

Tarikh: 20.4.05

Penyelia Kedua (jika ada)

Nama: En Asmadi bin Ali

Cop Rasmi:

ASMADI BIN ALI @ MAHMUD
Pensyarah

Jabatan Sains Kejuruteraan
Fakulti Sains dan Teknologi
Kolej Universiti Sains dan Teknologi Malaysia
21030 Kuala Terengganu

Tarikh: 20.4.05

Ketua Jabatan Sains Kejuruteraan

Nama: PM Ir Ahmad bin Jusoh

Cop Rasmi:

Tarikh: 20.4.05

ACKNOWLEDGEMENT

Syukur alhamdulillah in the name of ALLAH S.W.T, the most Gracious and Merciful I finally finished my thesis. After all hard work and hard task that I have been through, by hook or by crook, I finally managed to finish it in time. First of all, I would like to express my appreciation and thanks to my supervisor, Dr Nora'aini Ali for her courage and guidance, to En Asmadi Ali as my co-supervisor who always supported me and also to En Zul Hassan in completing this task. Million of thanks I dedicated specially to the head department Faculty of Science and Technology(FST), Prof Madya Ir Md Jusoh. Besides that, to all KUSTEM lecturers and staffs for their cooperation and understands my needs in finishing this project.

My deepest love and gratitude for my dearest parents, Ahmad Subki Haji Ahmad and Nor Sulaiman for their warmest love and attention they gave to me. To all my classmates and close friends, a bunch of thanks I give to you all. Last but not least, to all the people I have mentioned above and for those I did not, my deepest love and appreciation I specially dedicated for them. Thank you.

CONTENT

	PAGE
TITLE	i
CONFIRMATION FORM AND THESIS APPROVAL	ii
ACKNOWLEDGEMENT	iii
CONTENT	iv
LIST OF TABLE	vii
LIST OF FIGURES	viii
LIST OF SYMBOLS	x
LIST OF APPENDICES	xi
ABSTRACT	xii
ABSTRAK	xiii
CHAPTER 1	INTRODUCTION AND OBJECTIVE
1.1	Problem Statement 2
1.2	Objective Research 3
1.3	Scope Of Research 3
CHAPTER 2	LITERATURE REVIEW
2.1	Asymmetric Nanofiltration Membrane 5

2.2	Membrane Filtration Configuration	7
2.3	The Effect of Additives In The Casting Solution	8
2.4	The Factors That Effect Membrane Structure	10
2.5	The Factors That Effect Permeate Water Flux	11

CHAPTER 3 METHODOLOGY

3.1	Material Selection	13
3.2	Membrane Preparation	15
	3.2.1 <i>Turbidimetric Titration Method</i>	15
	3.2.2 <i>Dope Preparation</i>	17
	3.2.3 <i>Membrane Casting</i>	20
3.3	Membrane Characterization	22
3.4	Membrane Performance Measurement	23
	3.4.1 <i>Pure Water Flux Permeation</i>	24
	3.4.2 <i>Measurement of Sodium Chloride</i> <i>(NaCl) Performance</i>	26

CHAPTER 4 RESULT AND DISCUSSION

4.1	Membrane Structure Characteristics	31
4.2	Pure Water Flux Permeation	34
	4.2.1 <i>Comparison Between B23, T23 and Q23</i>	34
	4.2.2 <i>Comparison Between B23, T21 and Q16</i>	36
4.3	Measurement of Sodium Chloride (NaCl) Performance	38
	4.3.1 <i>Comparison Between B23, T23 and Q23</i>	38

CHAPTER 5	CONCLUSION AND RECOMMENDATION	
5.1	Conclusion	44
5.2	Recommendations	45
REFERENCES		47
APPENDICES		50
VITAE		62

LIST OF TABLE

Table No.	Title	Page
3.1	Material Selection for Binary, Ternary and Quaternary Component	13
4.1	The first set of formulation and membrane ID	30
4.2	The second set of formulation and membrane ID	31
4.3	Rejection(%) and flux range for NaCl for B23, T23 and Q23	43
4.4	Rejection(%) and flux range for NaCl for B23, T21 and Q16	43

LIST OF FIGURES

Figure No	Title	Page
2.1	Flow direction of feed solution of a dead-end filtration	7
2.2	Schematic diagram of dead-end permeation cell	8
2.3	Flow direction of feed solution of a cross-flow filtration	8
3.1	Structural formula of polyethersulfone (PES)	14
3.2	Three steps in membrane preparation	15
3.3	Apparatus setup for turbidimetric titration method	16
3.4	GR-200 digital scale for weighting PES, NMP, H ₂ O	18
3.5	Apparatus setup for making dope solution	19
3.6	Semi Automated Electrical Casting Machine Model FC 240	20
3.7	Membrane film was hanged up (drying process) for one day	21
3.8	Scanning electron microscopy (SEM) model JSM 630LA	22
3.9	The principle of Scanning Electron Microscopy (SEM)	23
3.10	The apparatus setup for membrane performance measurement	24
3.11	The essential parts of dead-end permeation cell	25
3.12	Calibration curve for 0 to 0.1 M NaCl solution	27
3.13	Calibration curve for 0 to 0.01 M NaCl solution	27
4.1	Cross section of membrane structure for binary, ternary and quaternary component, B23, T21, T23, Q16 and Q23	32

Figure No	Title	Page
4.2	Flux ($\text{m}^3/\text{m}^2\text{sec}$)[$\times 10^{-6}$] versus pressure (MPa) for binary, ternary and quaternary component, B23, T23 and Q23	35
4.3	Flux ($\text{m}^3/\text{m}^2\text{sec}$)[$\times 10^{-6}$] versus pressure (MPa) for binary, ternary and quaternary component, B23, T21 and Q16	37
4.4	Rejection versus pressure graph and flux versus pressure graph (NaCl) for B23, T23 and Q23	40
4.5	Rejection versus pressure graph and flux versus pressure graph for (NaCl) for B23, T21 and Q16	42

LIST OF SYMBOLS

C_b	concentration of bulk solution (molm^{-3})
C_p	concentration of permeate solution (molm^{-3})
C_w	wall concentration
$D_{\text{eff},\infty}$	effective bulk diffusivity (m^2s^{-1})
H_2O	Water
J_v	flux (m^3/s)
k	mass transfer coefficient
MWCO	molecular weight cut off
NMP	N-Methyl-2-Pyrrolidone
PEG	polyethylene glycol
PES	Polyethersulfone
P_m	permeability coefficient
PSf	Polysulfone
PVP	Polyvinylpyrrolidone
r	radius of influence (cm)
TEG	tetra ethylene glycol
ν	kinematic viscosity (m^2s^{-1})
ω	stirring speed (rads^{-1})

LIST OF APPENDICES

Appendix	Title	Page
A	Calculation of Turbidimetric Titration Method	50
B	Calculation for the preparation of NaCl stock solution	54
C	Conductivity value for NaCl solution	56
D	Raw data for water flux and NaCl rejection	57

ABSTRACT

Membranes can be built from binary (polymer and solvent), ternary (polymer, solvent and non-solvent) and quaternary component (polymer, solvent, non-solvent and additive). This study is to investigate the asymmetric nanofiltration membrane performance in those three components. Two sets of formulation were prepared using polyethersulfone (PES) as the polymer, N-methyl-2-pyrrolidone (NMP) as the solvent, water (H₂O) as the non-solvent and polyvinylpyrrolidone (PVP) as the additive. The first formulation has the same PES percentage (23%) coded as B23, T23 and Q23 while the second formulation has different PES percentage (binary 23%, ternary 21% and quaternary 16%) coded as B23, T21 and Q16. The flat sheet membrane were prepared using dry-wet phase inversion technique and cast on semi-automated electrical casting machine. The liquid separation performance was measured using pure water and sodium chloride (NaCl) permeation test and the membrane structure was analyzed using scanning electron microscopy (SEM). The experimental result gives B23 as the highest pure water permeability ($21.24 \times 10^{-6} \text{ m}^3 / \text{m}^2 \text{sec.MPa}$) with finger-like structure. Q16 exhibits the best performance among other components, where it gives moderate flux (from 0 to $6.4 \text{ m}^3 / \text{m}^2 \text{sec}$) and high in rejection (from 0 to 26%). From the findings, it can be concluded that the introduction of additives in the casting solution will increase the membrane performance with sponge structure (small voids in the upper layer of the membrane)

ABSTRAK

Membran boleh dibina daripada komponen binari (polimer dan pelarut), ternari (polimer, pelarut dan bukan pelarut) dan kuaternari (polimer, pelarut, bukan pelarut dan aditif). Kajian ini dijalankan untuk membandingkan prestasi membran asimetrik penuras nano dalam ketiga-tiga komponen tersebut. Dua set formulasi disediakan menggunakan polyethersulfone (PES) sebagai polimer, N-methyl-2-pyrrolidone (NMP) sebagai pelarut, air (H₂O) sebagai bukan pelarut dan polyvinylpyrrolidone (PVP) sebagai aditif. Formulasi yang pertama mempunyai peratus polimer yang sama untuk setiap komponen (23%) yang dikodkan sebagai B23, T23 dan Q23. Formulasi yang kedua pula menggunakan peratus polimer yang berbeza bagi setiap komponen (binari 23%, ternari 21% dan kuaternari 16%) yang dikodkan sebagai B23, T21 dan Q16. Membran kepingan rata disediakan menggunakan teknik fasa basah-kering dan diacuankan menggunakan mesin acuan separa automatik. Prestasi pengasingan cecair dijalankan menggunakan air dan larutan garam (NaCl) dan struktur membran dianalisa menggunakan *scanning electron microscopy* (SEM). Keputusan eksperimen yang didapati ialah B23 menunjukkan pemalar kebolehtelapan air yang tertinggi ($21.24 \times 10^{-6} \text{ m}^3 / \text{m}^2 \text{sec.MPa}$) dengan struktur membran berbentuk jari. Q16 pula menunjukkan prestasi terbaik jika dibandingkan dengan komponen ternari dan binari, di mana ia memberikan lingkungan fluks yang sederhana (daripada 0 to $6.4 \text{ m}^3 / \text{m}^2 \text{sec}$) dan lingkungan penolakan ion garam yang tinggi (daripada 0 to 26%). Daripada kajian, boleh disimpulkan bahawa penambahan aditif ke dalam larutan dop akan meningkatkan prestasi membran dengan struktur berspan (kelompok kecil pada permukaan atas membran)