

PVDF-ALUMINA AND PVDF-BENTONITE  
HOLLOW FIBER MEMBRANE FOR CO<sub>2</sub>  
ABSORPTION VIA MEMBRANE CONTACTOR

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## **SUPERVISOR'S DECLARATION**

We hereby declare that We have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science in Chemical Engineering.

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## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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## ABSTRACT

PVDF hollow fiber membrane contactor system was improved by the addition of alumina and bentonite in terms of membrane structure and wetting resistance. Polyvinylidene fluoride (PVDF) polymer was modified by varying different concentration of alumina and bentonite (1 wt%, 3wt% and 5 wt%). Polymer dope solution (18 wt%) with various concentration of (alumina and bentonite) had been prepared and the hollow fiber membrane was fabricated using wet spinning process. CO<sub>2</sub> absorption was conducted using 1 M and 2 M of Monoethanolamine (MEA) as liquid absorbent. The hollow fiber membranes were characterized by using contact angle measurement, gas permeation test and tensile strength test to investigate the mechanical properties and membrane structure. Scanning electron microscope (SEM) and atomic force microscope (AFM) were used to investigate the membrane morphology and surface roughness of the membrane. The introduction of alumina and bentonite to polymer dope solution had improved the PVDF hollow fiber membrane characteristic, including membrane hydrophobicity and CO<sub>2</sub> absorption flux. The formation of sponge-like structure and finger-like structure in the membrane was developed by ranging different concentration of alumina and bentonite. Increasing the alumina and bentonite concentration in PVDF dope solution produced more sponge-like structure. The membrane with more sponge-like structure produced high hydrophobic membrane, reasonable mechanical strength, good gas permeability, high surface porosity, small in mean pore size and high absorption flux. The membranes surface roughness analyzed via atomic force microscope (AFM) showed that the mean surface roughness of the outer membrane was lower than inner membrane as the concentration of alumina and bentonite increased. PA5 and PB5 hollow fiber membranes possessed the highest contact angle values of 97.39° and 95.79°. PA5 and PB5 hollow fiber membrane showed the highest absorption flux in 1 M MEA of  $3.89 \times 10^{-2}$  mol/m<sup>2</sup>.s and  $4.08 \times 10^{-2}$  mol/m<sup>2</sup>.s at flow rate 200 ml/min, respectively. The CO<sub>2</sub> absorption was repeated by using 2 M MEA as absorbent. The absorption flux of PA5 and PB5 hollow fiber membrane had increased up to  $6.12 \times 10^{-2}$  mol/m<sup>2</sup>.s and  $6.35 \times 10^{-2}$  mol/m<sup>2</sup>.s, respectively. The long-term absorption test was conducted with PA5 and PB5 hollow fiber membranes in 2 M MEA for 60 hours. From the long-term CO<sub>2</sub> absorption flux, PA5 hollow fiber membrane showed much better performance than PB5 hollow fiber membrane. Therefore, PB5 membrane possessed a more sponge-like structure, high water contact angle, good mechanical strength, great gas permeability, high surface porosity with small mean pore size and demonstrated a higher absorption CO<sub>2</sub> flux is preferred for CO<sub>2</sub> membrane contactor application.

## ABSTRAK

Pembasahan membran adalah masalah utama dalam aplikasi penyentuh bermembran untuk penyerapan karbon dioksida. Untuk mengatasi masalah ini, membran serat hidrofobik gabungan komposit telah dibangunkan. Polimer polivinilidena fluorida (PVDF) telah diubahsuai dengan memperkenalkan pelbagai jenis tanah liat organik termasuk alumina dan bentonit (1% berat, 3% berat dan 5% berat) dimasukkan ke dalam larutan polimer untuk menghasilkan membran hidrofobik. Larutan polimer (18 wt%) dengan pelbagai kepekatan tanah liat organik (alumina dan bentonit) telah disediakan dan membran serat berongga dibuat dengan menggunakan proses berputar basah. Penyerapan CO<sub>2</sub> dilakukan menggunakan 1M dan 2M Monoethanolamine (MEA) sebagai cecair penyerap. Membran serat berongga dicirikan menggunakan pengukuran sudut sesentuh, ujian permeasi gas dan ujian kekuatan tarik untuk menyiasat sifat mekanik dan struktur membran. Pengimbasan mikroskop elektron (SEM) dan mikroskop tenaga atom (AFM) digunakan untuk menyiasat morfologi membran dan kekasaran permukaan membran. Pengenalalan alumina dan bentonit kepada penyelesaian larutan polimer telah meningkatkan ciri PVDF membran serat berongga termasuk membran hidrofobisiti dan penyerapan CO<sub>2</sub>. Pembentukan struktur seperti span dan jejari dalam membran telah dihasilkan oleh kepelbagai kepekatan alumina dan bentonit yang digunakan. Meningkatkan kepekatan alumina dan bentonit dalam larutan PVDF menghasilkan lebih banyak struktur span. Membran yang mempunyai lebih banyak struktur span telah menghasilkan membran yang tinggi hidrofobik, kekuatan mekanikal yang bagus, kebolehtelapan gas yang baik, keliangan permukaan yang tinggi, saiz liang yang kecil dan fluks penyerapan yang tinggi. Kekasaran permukaan membran yang dianalisis melalui mikroskop tenaga atom (AFM) menunjukkan bahawa kekasaran permukaan rata dari membran luar adalah lebih rendah daripada membran dalaman kerana kepekatan alumina dan bentonit meningkat. Membran serat berongga bagi PA5 dan PB5 mempunyai nilai sudut sesentuh tertinggi iaitu 97.39° dan 95.79°. Membran serat berongga bagi PA5 dan PB5 menunjukkan fluks penyerapan tertinggi dalam 1M MEA iaitu  $3.89 \times 10^{-2}$  mol / m<sup>2</sup>.s dan  $4.08 \times 10^{-2}$  mol / m<sup>2</sup>.s pada kadar aliran 200 mL/min. Penyerapan CO<sub>2</sub> diulang dengan menggunakan 2M MEA sebagai penyerap. Fluks penyerapan membran serat berongga bagi PA5 dan PB5 meningkat sehingga  $6.12 \times 10^{-2}$  mol / m<sup>2</sup>.s dan  $6.35 \times 10^{-2}$  mol / m<sup>2</sup>.s. Ujian penyerapan jangka panjang dilakukan dengan menggunakan membran serat berongga PA5 dan PB5 dalam 2M MEA selama 60 jam. Dari fluks penyerapan CO<sub>2</sub> bagi jangka masa yang panjang, membran serat berongga PA5 menunjukkan prestasi yang lebih baik daripada membran serat berongga PB5. Oleh itu, membran PB5 mempunyai lebih banyak struktur span, sudut sesentuh air yang tinggi, kekuatan mekanikal yang baik, kebolehtelapan gas yang hebat, keliangan permukaan yang tinggi dengan purata saiz liang yang kecil dan menunjukkan penyerapan CO<sub>2</sub> fluks yang lebih tinggi diberikan keutamaan dalam aplikasi penyentuh bermembran khas dalam penyerapan CO<sub>2</sub>.

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## LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
AMP	-	2-amino-2-methyl-1-propanol
CO <sub>2</sub>	-	Carbon Dioxide
DEA	-	Diethanolamine
FAS		1H, 1H, 2H, 2H-perfluorooctyltriethoxysilane
KSar	-	Potassium sarcosinate
KOH	-	Potassium hydroxide
LiCl	-	Lithium chloride
LiClO <sub>4</sub>		Lithium perchlorate
LEP <sub>w</sub>	-	Liquid entry pressure of water
MEA	-	Monoethanolamine
MDEA	-	Methyldiethanolamine
MIP	-	Mercury intrusion porosimetry
MMMs	-	Mixed matrix membranes
MMT	-	Montmorillonite
NaOH	-	Sodium hydroxide
NMP	-	N,N-dimethyl pyrrolidone
PVDF	-	Polyvinyl fluoride
PE	-	Polyethylene
PEG	-	Polyethylene glycol
PEI	-	Polyetherimide
PP	-	Polypropylene
PTFE	-	Polytetrafluorethylene
PSF	-	Polysulfone
PES	-	Polyether sulfone
PAN	-	Polyacrylonitrile
PCN	-	Polyurethane/clay nanocomposite
SAPO	-	Silicoaluminophosphate
SEM	-	Scanning Electron Microscopy
SMM	-	Surface modified micromolecule
THAM	-	2-amino-2-hydroxymethyl-1,3-propanediol

TEA	-	Triethylamine
TEM	-	Transmission Electron Microscopy
TiO <sub>2</sub>	-	Titanium Dioxide

## LIST OF SYMBOLS

$K_{ov}$	-	Overall mass transfer coefficient (m/s)
$d_i$	-	Fiber internal diameter (m)
$d_o$	-	Fiber outer diameter (m)
$d_m$	-	Log mean diameter of the fiber (m)
H	-	Henry's constant (mol/m <sup>3</sup> atm)
E	-	Enhancement factor
$S_h$	-	Sherwood number
$D_L$	-	Diffusion coefficient (m <sup>2</sup> /s)
L	-	Effective length of the membrane (m)
Sc	-	Schmidt number
$K_m$	-	Membrane mass transfer coefficient (m/s)
$D_{g,eff}$	-	Effective diffusion coefficient of gas
Re	-	Reynold number
$\delta$	-	Thickness of membrane (m)
$\tau$	-	Tortuosity of the membrane
$\varepsilon$	-	Porosity of the membrane
$d_h$	-	Hydraulic diameter (m)
$L$	-	Active length of the membrane (m)
$^{\circ}C$	-	Celcius
$J_{CO_2}$	-	CO <sub>2</sub> absorption flux (mol/m <sup>2</sup> s)
$Q_l$	-	Liquid flow rate (m <sup>3</sup> /s)
$A_i$	-	Inner surface of the hollow fiber membranes (m <sup>2</sup> )



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