

OPTIMIZING ISOTACTIC POLYPROPYLENE MEMBRANE  
PREPARATION CONDITION VIA TIPS FOR CARBON DIOXIDE AND  
NITROGEN SEPARATION

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OPTIMIZING ISOTACTIC POLYPROPYLENE MEMBRANE PREPARATION  
CONDITION VIA TIPS FOR CARBON DIOXIDE AND NITROGEN SEPARATION

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Thesis submitted in fulfillment of the requirements for the award of the degree of Master of  
Engineering in Chemical

Faculty of Chemical & Natural Resources Engineering  
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## LIST OF SYMBOLS

$A$	Isotactic polypropylene concentration (wt. %)
$B$	Immersion time in methanol (hour)
$C$	Dry Temperature ( $^{\circ}\text{C}$ )
$D$	Dry Time (minutes)
$K$	Degrees of freedom associated with SSR
$R^2$	Coefficient of determination
$X_i$	Coded value of the $i$ th independent variable
$X_i$	Uncoded value of the $i$ th independent variable
$X_i^*$	Uncoded $i$ th independent variable at the center point
$\Delta X_i$	Step change value
$Y$	Response

### Greek symbols

$\beta_0$	Constant coefficient
$\beta_1/\beta_2$	Linear coefficient
$\beta_{11}/\beta_{22}$	Quadratic coefficients
$\beta_{12}$	Quadratic interaction coefficients
$\varepsilon$	Approximation error

## LIST OF ABBREVIATIONS

IPP	Isotactic polypropylene
RSM	Responds Surface Methodology
CCD	Centre Composite Design
TIPS	Thermally Induced Phase Separation
SEM	Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
DPE	Dipenyl Ether
PE	Polyethylene
PTFE	Polytetrafluorethylene
PVDF	poly (vinylidene fluoride)
N <sub>2</sub>	Nitrogen
CO <sub>2</sub>	Carbon Dioxide



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## ABSTRACT

Over the past few decades, the emission of the flue gases into the atmosphere has increased and warms the earth's surface. One of the options to minimize this problem is by recovering carbon dioxide (CO<sub>2</sub>) from flue gas before releasing it to surrounding. Gas separation technology using membrane is a simple system that can effectively remove the CO<sub>2</sub> from flue gas as well as offering low installation cost. However, in the flue gas, there is water vapour content that causes the membrane to swell. Thus, for this study, in order to increase the durability and performance of membrane for CO<sub>2</sub> and nitrogen (N<sub>2</sub>) separation, the hydrophobic membrane is proposed. This study focuses on the effect of the addition of adipic acid on the hydrophobicity of the isotactic polypropylene (iPP) membranes. Response Surface Method (RSM) consists of 2-Level Factorial (FFD) and Centre Composite Design (CCD) for screening and optimization processes in the production of iPP microporous membrane were performed respectively. From this study, it was found that there are no such work has been reported in the open literature on the optimization of iPP membrane for gas separation. For morphology by using Scanning Electron Microscopy (SEM), observation on the iPP- DPE membranes exhibited smaller pore size compared to iPP-Methyl Salicylate (MS) membrane. Meanwhile, increasing adipic acid showed decreasing membrane pore size distribution. The result obtained by Fourier Transform Infrared Spectroscopy (FTIR) showed that there is no bonding changes between raw iPP and membrane fabricated by Thermally Induced Phase Separation Method (TIPS). Membrane prepared by Dipenyl Ether (DPE) and without addition of adipic acid produced the highest contact angle, CO<sub>2</sub> permeability and selectivity which are 112°, 22.01 GPU and 1.59 respectively. Analysis of variance of FFD showed that the impact of drying temperature and drying time were important than polymer concentration and immersion time in methanol toward contact angle and selectivity. The optimum membranes prepared in this experiment were membranes with the contact angle value and selectivity of 106.49 ° and 1.96 respectively at drying temperature of 54.96 °C and drying time of 18.66 min. These drying temperature and time are adequate for total methanol evaporation from pores which contributed to high selectivity and high contact angle value. The low error (below 30%) between predicted and actual value indicating that regression equations obtained from the FFD and CCD were expected to apply in the preparation of iPP membranes, can reasonably predict and optimize the performance of the iPP membranes.

## ABSTRAK

Sejak beberapa dekad yang lalu, pelepasan gas serombong ke udara telah meningkat dan memanaskan permukaan bumi. Salah satu cara untuk mengurangkan masalah ini adalah dengan menangkap karbon dioksida (CO<sub>2</sub>) daripada gas serombong sebelum ia di bebaskan ke sekeliling. Teknologi pemisahan gas yang menggunakan membran ialah satu sistem ringkas yang boleh memisah CO<sub>2</sub> daripada asap serombong selain dari menawarkan satu kos pemasangan yang rendah. Walau bagaimanapun, dalam gas serombong, terdapat kandungan wap air yang menyebabkan membran membengkak. Jadi, untuk kajian ini, usaha meningkatkan ketahanan dan prestasi membran untuk pemisahan CO<sub>2</sub> dan nitrogen (N<sub>2</sub>), membran yang bersifat hidropobik telah dicadangkan. Kajian ini memberi tumpuan kepada kesan penambahan asid adipik pada sifat hidrofobik daripada polipropilena isotactic (iPP) membran. Kaedah Gerak Balas Permukaan (RSM) terdiri daripada Dua Peringkat Reka Bentuk Faktorial (FFD) dan Reka Bentuk Komposit Berpusat (CCD) masing-masing telah digunakan untuk penelitian dan pengoptimuman proses pengeluaran iPP membran. Dengan menggunakan Mikroskopi Imbasan Elektron (SEM), pemerhatian pada membran struktur iPP-DPE mempamerkan saiz liang yang lebih kecil berbanding dengan membran iPP-Metil Salicilit (MS). Dengan peningkatan adipik asid, saiz liang pada membran semakin mengecil. Hasil yang didapati oleh Fourier Transform Infrared Spektroskopi (FTIR) menunjukkan bahawa tiada sebarang perubahan ikatan di antara iPP asal dan membran yang terhasil dari Aruhan Pemisahan Fasa Secara Pemanasan (TIPS). Membran yang disediakan oleh Dipenil Eter (DPE) dan tanpa penambahan asid adipik menghasilkan sudut sesentuh, ketelapan karbon dioksida dan pemilihan yang tertinggi dimana masing-masing ialah 112°, 22.01 GPU dan 1.59. Analisis varians dari FFD, menunjukkan bahawa kesan pengeringan suhu dan masa pengeringan adalah lebih penting dari kepekatan polimer dan masa rendaman dalam metanol terhadap sudut sesentuh dan kemilihan gas. Membran paling optimum disediakan dalam eksperimen ini adalah membran dengan nilai sudut sesentuh dan pemilihan masing-masing adalah 106.49 ° dan 1.96 pada suhu pengeringan 54.96 °C dan masa pengeringan 18.66 min. Suhu dan masa pengeringan ini adalah mencukupi untuk semua metanol untuk meruap dari liang dimana ia menyumbang pemilihan dan sudut sesentuh yang tinggi. Ralat yang rendah (bawah 30%) di antara nilai yang dijangka dan nilai sebenar adalah di menyatakan bahawa persamaan diperolehi daripada FFD dan CCD telah dijangka akan digunakan dalam penyediaan membran IPP, munasabah boleh meramalkan dan mengoptimumkan persembahan membran IPP.