

DEVELOPMENT OF POLYETHER BLOCK  
AMIDE (PEBAX) NANOCOMPOSITE  
MEMBRANES USING NANOADSORBENT  
FROM AGRICULTURAL WASTES AS  
FILLERS FOR GAS SEPARATION

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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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ALIA AQILAH BINTI GHAZALI

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## ABSTRAK

Sepanjang dekad yang lalu, proses membran untuk pemisahan gas semakin mendapat penerimaan dalam industri dan pasaran dan mereka bersaing dengan operasi sedia ada seperti tekanan ayunan penyerapan dan penyulingan kriogenik. Baru-baru ini, terdapat usaha yang intensif dalam pembangunan bahan membran yang lebih baik dan lebih kuat yang dipanggil membran nanokomposit yang melibatkan penambahan nanoadsorben seperti zeolit dan silika dalam polimer matriks. Minat yang mendalam telah diberikan kepada nanoadsorben yang disediakan daripada sisa pertanian kerana jumlahnya yang amat tinggi, ciri-ciri yang menjanjikan, dan prestasi penjerapan yang munasabah berbanding dengan nanoadsorben konvensional. Oleh itu, tujuan kajian ini adalah untuk membina, mencirikan, menilai dan mengoptimumkan membran nanokomposit yang terdiri daripada polyether block amide (Pebax) bergabung dengan dua jenis nanoadsorben yang berlainan (kulit nenas dan pelepah kelapa sawit) disalut atas polyvinylidene fluoride (PVDF) komersil bagi menyokong membran.  $2^4$  Full Factorial Design (FFD) telah digunakan dalam kajian ini untuk meminimumkan faktor-faktor yang mempengaruhi semasa penyediaan membran nanokomposit. Sebanyak empat faktor telah dipilih untuk dijalankan secara serentak dan respons adalah berdasarkan kebolehtelapan gas ( $\text{CO}_2$ ,  $\text{CH}_4$ ,  $\text{N}_2$ ) dan selektiviti ( $\text{CO}_2/\text{CH}_4$ ,  $\text{CO}_2/\text{N}_2$ ). Dalam penyediaan membran, faktor-faktor yang terlibat adalah kepekatan Pebax (3 dan 6 wt%), jenis nanoadsorben (kulit nenas dan OPF), kepekatan nanoadsorben (0.5 dan 5 wt% berdasarkan berat Pebax) dan masa sonikasi (15 dan 30 minit). Dua faktor yang paling berpengaruh terhadap kecekapan pemisahan gas akan dijalankan kajian lanjut untuk pengoptimuman menggunakan Central Composite Design (CCD). Molekul permukaan membran nanokomposit yang baru disintesis dianalisis dengan menggunakan Field Emission Scanning Electron Microscope (FESEM), kewujudan kumpulan fungsi menggunakan Fourier Transform Infrared Spectroscopy (FTIR), pengukuran kuasa dan pengimejan menggunakan kaedah Atomic Force Microscopy (AFM), dan kristaliti menggunakan instrumen X-Ray Diffraction (XRD). Penemuan dari pemeriksaan tersebut mencadangkan bahawa faktor yang paling mempengaruhi kebolehtelapan dan selektiviti adalah kepekatan Pebax dan jenis nanoadsorben. Keadaan terbaik diketahui memaksimumkan kebolehtelapan dan selektiviti. Keadaan yang dikenalpasti adalah kepekatan Pebax pada 5.5 wt% menggunakan 4.5 wt% nano nenas karbon aktif dan 15 minit sonikasi yang memberikan ketelapan  $\text{CO}_2$ , selektiviti  $\text{CO}_2/\text{CH}_4$  dan selektiviti  $\text{CO}_2/\text{N}_2$  1537.08 Barrer, 40.21 dan 41.39. Hasil kajian menunjukkan bahawa FFD sesuai untuk meminimumkan faktor-faktor dengan mempertimbangkan interaksi antara faktor-faktor yang terlibat dalam sintesis filem membran untuk prestasi pemisahan gas yang sangat baik. Selain itu, penggabungan nanoadsorben kos rendah ini dilaporkan mengubah struktur membran polimer dan sifat-sifat kimia yang membawa kepada penambahbaikan prestasi membran yang secara signifikan berada hampir dan melepasi garisan limit Robeson, serta menunjukkan nilai yang tinggi buat kedua-dua kebolehtelapan gas dan selektiviti.

## ABSTRACT

Over the past decade, membrane processes for gas separation are gaining a larger acceptance in the industry and the markets are competing with consolidated operations such as pressure swing absorption and cryogenic distillation. There have been intensive efforts in the development of better and more robust membrane material called nanocomposite membranes which involved the incorporation of nanoadsorbents such as zeolite and silica in the polymer matrix. Recently, interest has been given to nanoadsorbents derived from agricultural wastes due to their abundance, promising features, and reasonable adsorption performances compared to the conventional nanoadsorbents. Therefore, the purpose of this study is to develop, characterise, evaluate and optimise the nanocomposite membranes comprised of the coated polyether block amide (Pebax) filled with two different types of nanoadsorbent (pineapple peel and oil palm frond) on the commercial polyvinylidene fluoride (PVDF) support membrane. A  $2^4$  full factorial design (FFD) was utilized in this study to minimize the influenced factors during nanocomposite membrane preparation. A total of four factors were chose to run simultaneously and the responses were based on permeability ( $\text{CO}_2$ ,  $\text{CH}_4$ ,  $\text{N}_2$ ) and ideal selectivity ( $\text{CO}_2/\text{CH}_4$ ,  $\text{CO}_2/\text{N}_2$ ). For the preparation of flat sheet membrane, the factors include Pebax concentration (3 and 6 wt%), types of nanoadsorbent (pineapple peel and OPF), nanoadsorbent concentration (0.5 and 5 wt% based on Pebax weight) and sonication time (15 and 30 min). Two most influenced factors towards the final separation efficiency were further study for optimisation using central composite design (CCD). The surface morphologies of the newly synthesized nanocomposite membranes were analysed using Field Emission Scanning Electron Microscope (FESEM), functional groups using Fourier Transform Infrared Spectroscopy (FTIR), force measurement and imaging using Atomic force microscopy (AFM), and crystallinity using X-Ray Diffraction instrument (XRD). The findings from the screening suggested that the most influential factors that affect the permeability and selectivity are Pebax concentration and types of nanoadsorbent. The best condition was known to maximize the permeability and selectivity. The identified conditions were Pebax concentration at 5.5 wt% using 4.5 wt% nano-pineapple peel activated carbon and sonication time of 15 min which gave  $\text{CO}_2$  permeability,  $\text{CO}_2/\text{CH}_4$  selectivity and  $\text{CO}_2/\text{N}_2$  selectivity of 1537.08 Barrer, 40.21 and 41.39, respectively. The outcome of this study indicates that FFD was suitable to minimize and eliminate factors by considering the interaction among the factors involves in membrane film synthesis for excellent gas separation performance. Besides, the incorporation of this nanoadsorbents were reported to alter the polymer membrane structure and chemical properties which led to an improvement of the membrane's performance that lie significantly near and above the Robeson upper bound limit, which satisfies both high selectivity as well as high permeability.

## TABLE OF CONTENT

|  |            |
|--|------------|
| <b>DECLARATION</b>   |            |
| <b>TITLE PAGE</b>  |            |
| <b>ACKNOWLEDGEMENTS</b>                                    | <b>ii</b>  |
| <b>ABSTRAK</b>   | <b>iii</b> |
| <b>ABSTRACT</b>  | <b>iv</b>  |
| <b>TABLE OF CONTENT</b>                                    | <b>v</b>   |
| <b>LIST OF TABLES</b>                                      | <b>ix</b>  |
| <b>LIST OF FIGURES</b>                                     | <b>xi</b>  |
| <b>LIST OF SYMBOLS</b>                                     | <b>xiv</b> |
| <b>LIST OF ABBREVIATIONS</b>                               | <b>xv</b>  |
| <br>   |            |
| <b>CHAPTER 1 INTRODUCTION</b>                              | <b>1</b>   |
| 1.1 Introduction   | 1          |
| 1.2 Background of Study                                    | 1          |
| 1.3 Problem Statement                                      | 3          |
| 1.4 Research Objectives                                    | 5          |
| 1.5 Scope of Study   | 5          |
| 1.6 Organisation of Thesis                                 | 6          |
| <br>   |            |
| <b>CHAPTER 2 LITERATURE REVIEW</b>                         | <b>8</b>   |
| 2.1 Introduction   | 8          |
| 2.2 Membrane Technology                                    | 8          |
| 2.2.1 Gas Permeation Mechanisms through Polymeric Membrane | 13         |
| 2.2.2 Gas Transport Mechanisms in Nanocomposite Membrane   | 15         |
| 2.3 Nanocomposite Membrane                                 | 16         |



|                              |  |           |
|------------------------------|--|-----------|
| 2.3.1                        | Pebax as the Selective Layer in Composite Membrane Development   | 17        |
| 2.3.2                        | PVDF as the Substrate Layer in Composite Membrane Fabrication  | 18        |
| 2.3.3                        | Role of Fillers in Nanocomposite Membranes   | 19        |
| 2.4                          | Nanoadsorbent from Agricultural Wastes in Composite membrane   | 24        |
| 2.5                          | Potential of Activated Carbon Adsorbents from Agricultural Wastes as Alternative Fillers                   | 29        |
| 2.5.1                        | Pineapple Peel (PP) as a Better Alternative Adsorbent  | 30        |
| 2.5.2                        | Oil Palm Frond (OPF) as a Better Alternative Adsorbent   | 33        |
| 2.6                          | Influence of Various Synthesis Conditions on Nanocomposite Membrane Properties and Separation Performances | 36        |
| 2.6.1                        | Effect of Pebax Concentration  | 36        |
| 2.6.2                        | Effect of Nanoadsorbent Type   | 37        |
| 2.6.3                        | Effect of Nanoadsorbent Concentration  | 38        |
| 2.6.4                        | Effect of Sonication Time  | 39        |
| 2.7                          | Design of Experiments (DOE)  | 39        |
| 2.7.1                        | Full Factorial Design (FFD)  | 40        |
| 2.7.2                        | Central Composite Design (CCD)   | 41        |
| 2.8                          | Chapter Summary  | 42        |
| <b>CHAPTER 3 METHODOLOGY</b> |  | <b>44</b> |
| 3.1                          | Introduction   | 44        |
| 3.2                          | Research Methodology   | 44        |
| 3.3                          | Materials  | 46        |
| 3.4                          | Nanoadsorbents Synthesis and Characterisation  | 46        |
| 3.4.1                        | Preparation of Nanoadsorbents  | 46        |

|   |   |               |
|---|---|---------------|
| 3.4.2                                       | Characterisation of Nanoadsorbents  | 48            |
| 3.5   | Nanocomposite Membrane Formation  | 49            |
| 3.6   | Screening using Full Factorial Design (FFD)   | 50            |
| 3.7   | Optimisation by Central Composite Design (CCD)  | 52            |
| 3.8   | Gas Permeation Test   | 53            |
| 3.9   | Membrane Characterisation   | 55            |
| 3.9.1                                       | Field Emission Scanning Electron Microscope (FESEM)   | 55            |
| 3.9.2                                       | Fourier Transform Infrared Spectroscopy (FTIR)  | 55            |
| 3.9.3                                       | Atomic Force Microscopy (AFM)   | 56            |
| 3.9.4                                       | X-Ray Diffraction instrument (XRD)  | 56            |
| <br><b>CHAPTER 4 RESULTS AND DISCUSSION</b> |   | <br><b>57</b> |
| 4.1   | Introduction  | 57            |
| 4.2   | Determination of Nanoadsorbent Concentration Range in the Preparation of Nanocomposite Membrane | 57            |
| 4.2.1                                       | Characterisation of Raw Materials and Nanoadsorbents  | 57            |
| 4.2.2                                       | Characterisation of Nanocomposite Membranes   | 62            |
| 4.2.3                                       | Performance Study on the Effect of Nanoadsorbent Concentration                                  | 68            |
| 4.3   | Screening on Factors by 2 Level Factorial Design  | 69            |
| 4.3.1                                       | Analysis of Variance (ANOVA)  | 71            |
| 4.3.2                                       | Pareto chart of the Main and Interaction Effects of Factors                                     | 76            |
| 4.3.3                                       | Effects of Independent Processing Factors on Gases permeability and Selectivity                 | 78            |
| 4.3.4                                       | Interaction Effects Between Factors on Gases Permeability and Selectivity                       | 84            |
| 4.3.5                                       | Main Findings from Screening of Factors towards Membrane Performances and CCD data Prediction   | 88            |

|       |   |            |
|-------|---|------------|
| 4.4   | Optimisation of Membrane Separation Performances by Central Composite Design            | 89         |
| 4.4.1 | Analysis of Variance (ANOVA)  | 90         |
| 4.4.2 | Analysis of The Response Surface on Membrane Performances                               | 95         |
| 4.5   | Validation test on Membrane Performances  | 102        |
| 4.6   | Characterisation of Nanocomposite Membranes   | 103        |
| 4.6.1 | Field Emission Scanning Electron Microscope (FESEM) analysis on Nanocomposite Membranes | 103        |
| 4.6.2 | Atomic Force Microscopy (AFM) study of Nanocomposite Membranes                          | 107        |
| 4.6.3 | Fourier Transform Infrared spectroscopy (FTIR) Analysis on Nanocomposite Membranes      | 110        |
| 4.6.4 | X-ray Diffraction (XRD) Study on Nanoadsorbent and Optimum Nanocomposite Membranes      | 113        |
| 4.7   | Summary of Key Experimental Findings  | 114        |
|       | <b>CHAPTER 5 CONCLUSION</b>   | <b>117</b> |
| 5.1   | Conclusion  | 117        |
| 5.2   | Recommendations   | 118        |
| 5.3   | Contributions   | 119        |
| 5.3.1 | Journals  | 119        |
| 5.3.2 | Conferences   | 120        |
|       | <b>REFERENCES</b>   | <b>121</b> |
|       | <b>Appendix A: Calculation of Membrane Composition</b>                                  | <b>140</b> |
|       | <b>Appendix B: Gas Permeation Results</b>   | <b>142</b> |

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