

OPTIMIZATION OF INTERFACIAL POLYMERIZATION THIN FILM
COMPOSITE MEMBRANE FOR SEPARATION OF XYLOSE FROM GLUCOSE

MAH KAH HONG

MASTER OF SCIENCE
UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

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LIST OF SYMBOLS

A	Effective membrane area (m^2)
C_b	Concentration of respective solute in bulk (g/100 g of solution)
C_{b_glu}	Concentration of glucose in the bulk (g/100 g of solution)
C_{b_xyl}	Concentration of xylose in the bulk (g/100 g of solution)
C_f	Concentration of respective solute in feed (g/100 g of solution)
C_p	Concentration of respective solute in permeate (g/100 g of solution)
C_{p_glu}	Concentration of glucose in permeate (g/100 g of solution)
C_{p_xyl}	Concentration of xylose in permeate (g/100 g of solution)
C_r	Concentration of respective solute in retentate (g/100 g of solution)
J_0	Pure water flux before filtration ($\text{Lm}^{-2} \text{h}^{-1}$)
J_f	pure water flux after filtration ($\text{Lm}^{-2} \text{h}^{-1}$)
J_v	Separation flux ($\text{L m}^{-2} \text{h}^{-1}$)
J_w	Water flux ($\text{L m}^{-2} \text{h}^{-1}$)
P_m	Pure water permeability ($\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$)
R^2	Coefficient of determination
R_{obs}	Observed retention of respective solute
r_p	effective pore radius (nm)
Δt	measured period of time for collection of ultrapure water (h)
ΔV	volume of ultrapure water collected (L)
X_{xyl}	xylose separation factor
$\Delta x/A_k$	ratio of effective membrane thickness over porosity (μm)

LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
ANOVA	Analysis of variance
ATR	Attenuated total reflectance
CCD	Central composite design
DOE	Design of experiments
DSPM	Donnan-steric-pore model
EFBF	Empty fruit bunch fiber
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infrared
HPLC	High performance liquid chromatography
IP	Interfacial polymerization
LBL	Layer-by-layer
LC	Liquid chromatography
MPD	M-phenylene-diamine
MWCO	Molecular weight cut-off
NF	Nanofiltration
NRF	Normalized relative flux
OFAT	One-factor-at-a-time
PES	Polyethersulfone
PP	Polyetherimide, polypropylene
PSf	Polysulfone
RFR	Relative flux reduction
RI	Refractive index

RO	Reverse osmosis
RSM	Response surface methodology
TEOA	Triethanolamine
TFC	Thin-film composite
TMC	Trimesoyl chloride

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ABSTRACT

Most hydrolysis studies on biomass in Malaysia produce high amount of xylose and glucose compared to other monosaccharides. These monosaccharides are important ingredients often needed in pure fraction in food and pharmaceutical industries. Chromatography and commercial nanofiltration membrane were able to separate xylose from glucose. However, few treatment steps on biomass hydrolysate were needed because most biomass hydrolysate are acidic. Acidity reduces the performance of these separation technology by inhibiting chromatography resins and fouling of membrane. Thin film composite membrane developed via interfacial polymerization using triethanolamine and trimesoyl chloride as monomers allows separation at low pH to occur without damaging its performance. Currently, almost none has attempted to separate xylose from glucose using self-made thin-film composite membrane that is specially tailored for biomass hydrolysate. The aim of this present study was to produce optimized thin-film composite nanofiltration membrane for separation of xylose from glucose using triethanolamine and trimesoyl chloride as monomers on polyethersulfone membrane via interfacial polymerization using a series of experimental design. Success of thin layer formation was probed by attenuated total reflectance-Fourier transform infrared spectroscopy, and prepared membranes were characterized by field emission scanning electron microscope, contact angle and pure water permeability. Separation performance of thin-film composite membranes are affected by several factors during formation of thin upper layer. Series of experimental designs were applied to screen and optimize the different interfacial polymerization factors studied. In screening, 2^{5-1} fractional factorial design were used to find significant factors affecting xylose separation factor, which are reaction time and curing process. Also, the responses in screening were fitted with a multiple linear regression equation and obtained a high correlation ($R^2 = 0.9998$) between the experimental data and model data. Then central composite design was used to identify the optimum interfacial polymerization conditions for the highest xylose separation factor. The response was fitted with the second-order polynomial equation with R^2 of 0.92, implying a high correlation between the observed and predicted values. The optimum interfacial polymerization conditions were determined to be reaction time of 45.25 minutes, curing time of 15.53 minutes, and curing temperature of 58.4 °C. At optimum conditions, the xylose separation factor was found to be 1.334 ± 0.007 . The developed model in this study is adequate for predicting xylose separation factor under different interfacial polymerization conditions within the range used. This study will provide valuable guideline to develop membrane that specially tailored for xylose separation from glucose as alternative to the cost intensive chromatographic processes in use.

ABSTRAK

Kebanyakan kajian hidrolisis ke atas biojisim di Malaysia menghasilkan xilosa dan glukosa dalam jumlah yang tinggi berbanding monosakarida lain. Monosakarida-monosakarida ini adalah bahan penting yang selalunya diperlukan dalam pecahan tulen dalam industri makanan dan farmaseutikal. Kromatografi dan membran nanoturasan komersil mempunyai keupayaan dalam memisahkan xilosa dari glukosa. Nanum, beberapa langkah-langkah perawatan ke atas hidrolisat biojisim perlu dilakukan kerana kebanyakan hidrolisat biojisim berasid. Keasidan mengurangkan prestasi teknologi pemisahan tersebut melalui perencatan resin-resin kromatografi dan pengotoran ke atas membran. Membran komposit filem nipis yang dihasilkan melalui pempolimeran antara muka menggunakan trietanolamina dan trimesoyl klorida sebagai monomer membolehkan pemisahan berlaku pada pH rendah tanpa merosakkan prestasinya. Pada masa ini, hampir tiada yang mencuba memisahkan xilosa dari glukosa menggunakan membran komposit filem nipis buatan sendiri khusus untuk hidrolisa biojisim. Tujuan kajian ini adalah untuk menghasilkan membran komposit filem nipis optimum bagi pemisahan xilosa dari glukosa menggunakan trietanolamina dan trimesoyl klorida sebagai monomer ke atas membran polietersulfon melalui pempolimeran antara muka. Kejayaan pembentukan lapisan nipis ini disahkan oleh spektroskopi pantulan keseluruhan dikesilkan - infrared transformasian Fourier dan pencirian membran yang terhasil ditentukan menggunakan mikroskop elektron pengimbas pancaran medan, sudut sesentuh dan keterlapan air tulen. Prestasi pemisahan oleh membran komposit filem nipis dipengaruhi oleh beberapa faktor semasa pembentukan lapisan atas nipis melalui kaedah pempolimeran antara muka. Rekabentuk eksperimen secara bersiri telah diaplikasikan bagi menyaring dan mengoptimumkan faktor-faktor pempolimeran antara muka berlainan yang dikaji. Dalam penyaringan, reka bentuk eksperimen berfaktor pecahan 2^{5-1} telah digunakan untuk mencari faktor-faktor yang paling bererti dalam mempengaruhi faktor pemisahan xilosa, iaitu masa tindakbalas dan proses pematangan. Juga, respon dalam penyaringan telah dipadankan dengan persamaan regresi linear berganda dan memperolehi korelasi yang tinggi ($R^2 = 0.9998$) di antara data eksperimen dan data modal. Kemudian, reka bentuk eksperimen gabungan berpusat telah digunakan untuk mencari keadaan optimum bagi faktor-faktor pempolimeran antara muka untuk mendapat faktor pemisahan xilosa yang tertinggi. Respon telah dipadankan dengan persamaan polinomial tertib kedua dengan R^2 of 0.92, mengimplikasikan korelasi yang tinggi di antara nilai cerapan dan nilai ramalan. Keadaan optimum adalah pada tindakbalas, 45.25 minit; masa pematangan, 15.53 minit dan suhu pematangan, 58.4 °C. Dalam keadaan optimum ini, nilai tertinggi faktor pemisahan xilosa ialah 1.334 ± 0.007 . Model yang diterbitkan dalam kajian memadai bagi menganggarkan faktor pemisahan xilosa dibawah keadaan-keadaan pempolimeran berbeza dalam julat yang dikaji. Kajian ini memberi garis panduan yang bernilai bagi menghasilkan membran khusus untuk pemisahan xilosa dari glukosa sebagai alternatif kepada proses kromatografi yang mahal dalam penggunaan.

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