FABRICATION OF HYBRID GRAPHENE-POLYETHERSULFONE SUPPORTED LIQUID MEMBRANE FOR ACETIC ACID REMOVAL FROM OIL PALM FROND BIOMASS HYDROLYSATE

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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 Doctor of Philosophy.

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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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ABSTRAK

Penukaran biojisim lignoselulosa kepada bahan bakar bio memberi sumbangan hebat kepada pembekalan sumber tenaga boleh diperbaharui. Untuk mencapai matlamat ini, penguraian asid telah digunakan untuk menguraikan bahan lignoselulosa kepada gula fermentasi. Penguraian asid adalah kaedah yang cekap, mudah dan pantas berbanding penguraian enzim. Walau bagaimanapun, masalah utama yang ditemui semasa proses penguraian adalah pembebasan kumpulan asetil seperti asid asetik (AA) sebagai hasil sampingan bersama komponen gula. AA boleh bertindak sebagai perencat kepada penukaran enzim gula ke dalam produk akhir etanol atau bahan kimia lain. Maka, AA perlu dikeluarkan daripada hidrolisat biojisim untuk memaksimumkan penghasilan produk akhir. Kajian ini menumpukan terhadap pembinaan sokongan membran hibrid yang digunakan di dalam cecair membran bersokong (SLM) proses untuk penyingkiran AA menggunakan teknik pemisahan fasa induksi wap (VIPS). Membran hibrid telah dicirikan dari segi morfologi dengan menggunakan pengimbas mikroskop elektron (SEM) dan pelepasan medan pengimbas mikroskop elektron (FESEM), hidrofobisiti membran dan kekuatan mekanikal. Cecair organik membran untuk pengestrakkan AA telah diformulasikan di bahagian pertama kajian ini. Cecair membran dan agen pelucutan terbaik adalah pada pembawa 0.5 M tri-n-otylamine (TOA) di dalam pelarut 2-ethyl-1-hexanol dan 0.5 M NaOH. Penggabungan 0.1 wt% graphene di dalam hibrid polyethersulfone (PES) lembaran rata sokongan membran didapati ketara meningkatkan tekanan tegangan membran hibrid dari 740 kPa kepada 1790 kPa, peningkatan sebanyak 140% kekuatan mekanikal berbanding membran PES yang asli. Sudut sesentuh membran juga meningkat dari 81.92º kepada 122.35º dan menjadi sokongan membran yang sangat tinggi hidrofobik yang dapat memperbaiki kestabilan SLM. PES-0.1 graphene (G) sokongan membran kekal stabil lebih daripada 116 jam (12 kitaran SLM) tanpa rendaman semula dalam cecair membran berbanding dengan membran asli yang hanya stabil untuk 16 jam (2 kitaran SLM). Keadaan terbaik untuk penghasilan membran hibrid lembaran rata melalui VIPS adalah menggunakan suhu rendaman 50 °C, 30 saat masa pendedahan dan 80% kelembapan udara. Ia menunjukkan 95% penyingkiran AA daripada larutan akueus 10 g/l. Semasa penghasilan sokongan membran gentian berongga, masa rendaman membran cecair dan mod operasi aliran suapan PES-0.1G gentian berongga telah dikaji. Masa rendaman terbaik untuk penyediaan modul gentian berongga adalah 4 jam. Fasa suapan yang mengalir di sisi lumen (Mod I) menunjukkan prestasi pemisahan yang lebih baik dibandingkan dengan sisi shell (Mod II). Peratusan penyingkiran AA menggunakan gentian berongga yang dikendalikan dengan Mod I dan Mod II masing-masing adalah 80.1% dan 42.4%. Kebolehan proses SLM dalam mengeluarkan AA dari hidrolisat biojisim pelepah kelapa sawit (OPF) telah diuji menggunakan membran PES-0.1G lembaran rata dan gentian berongga. Kepekatan AA dalam OPF hidrolisat dikurangkan dari 6.83 g/l kepada 1.33 g/l dan 2.01 g/l dengan menggunakan lembaran rata dan gentian berongga SLM. Kedua-dua sistem SLM memenuhi kepekatan minimum AA yang perlu wujud dalam hidrolisat biojisim untuk memastikan penghasilan etanol yang tinggi yang kurang daripada 5 g/l. Oleh itu, sistem SLM yang menggunakan sokongan membran hibrid G-PES yang dihasilkan dalam kajian ini terbukti berkesan menyingkirkan AA daripada larutan akueus dan hidrolisat biojisim OPF.

ABSTRACT

Conversion of lignocellulosic biomass to biofuel gives a great contribution to the supplement of renewable energy source. To achieve this purpose, acid hydrolysis was used to hydrolyze the lignocellulosic materials to fermentable sugars. Acid hydrolysis is efficient, simple and fast method compared to enzymatic hydrolysis. However, the major problem encountered during the hydrolysis process is the releasing of acetyl group such as acetic acid (AA) as byproducts with the hydrolyzed sugar component. AA can acts as inhibitors to the enzymatic conversion of sugar into the final product of ethanol or other chemicals. Therefore, AA needs to be removed from the biomass hydrolysate to maximize the yield of products. This study focused on development of the hybrid membrane support for used in the supported liquid membrane (SLM) process for AA removal using vapor induced phase separation (VIPS) technique. The hybrid membrane were characterised in term of morphology by scanning electron microspcope (SEM) and field emission scanning electron microscope (FESEM), porosity, membrane hydrophobicity and mechanical strength. The organic liquid membrane phase for extraction of AA was formulated in the first part of the study. The best liquid membrane phase and stripping agent were 0.5 M tri-n-octyl-amine (TOA) carrier in 2-ethyl-1-hexanol diluent and 0.5 M NaOH, respectively. Incorporation of 0.1 wt% graphene in the hybrid polyethersulfone (PES) flat sheet membrane support was found significantly improved the tensile stress of the hybrid membrane from 740 kPa to 1790 kPa, an improvement about 140% in mechanical strength compared to pristine PES membrane. The contact angle of the hybrid membrane also increased from 81.92º to 122.35º and becoming highly hydrophobic membrane support that improved the SLM stability. PES-0.1graphene (G) membrane support remains stable for more than 116 hours (12 SLM cycles) without requires reimpregnation in the liquid membrane phase compared to the pristine membrane that only stable for 16 hours (2 SLM cycles). The best condition to prepare the flat sheet hybrid membrane through VIPS are using 50 °C coagulation bath temperature, 30 second air exposure time and 80% air humidity. It showed 95% removal of the AA from 10 g/l aqueous solution. During production of hollow fiber membrane support, impregnation time of liquid membrane and feed flow operating modes of PES-0.1G hollow fiber membrane was studied. The best impregnation time for preparing hollow fiber module was 4 hours. The feed phase flowed in lumen side (Mode I) showed better separation performance compared to the shell side (Mode II). The removal percentage of AA using hollow fiber operated with Mode I and Mode II were 80.1% and 42.4%, respectively. The capability of SLM process in removing of AA from oil palm frond (OPF) biomass hydrolysate was tested using PES-0.1 G flat sheet and hollow fiber membrane. The concentration of AA in the OPF hydrolysate was reduced from 6.83 g/l to 1.33 g/l and 2.01 g/l using flat sheet and hollow fiber SLM, respectively. Both SLM systems meet the minimum concentration of AA that should present in the biomass hydrolysate for ensuring highest ethanol production which is less than 5 g/l. Thus, the SLM system using hybrid G-PES membrane support developed in this study is proven effective for removing AA from aqueous solution and OPF biomass hydrolysate

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LIST OF SYMBOLS

A	Area
°C	Degree Celcius
ρ	Density
g/g	Gram per Gram
g/l	Gram per Liter
g/mol	Gram per Mole
h	Hour
K	Kelvin
kPa	Kilo Pascal
L	Liter
µm	Micrometer
ml	Mililiter
ml/min	Mililiter per Minutes
mm	Milimeter
min	Minute
M	Molar Concentration
N	Newton
%	Percentage
ϕ	Porosity
min^{-1}	Reciprocal Minutes
sec	Second
W_1	Weight of the Dry Membrane
W_2	Weight of the Wet Membrane

LIST OF ABBREVIATIONS

AA	Acetic Acid
BLM	Bulk Liquid Membrane
CBT	Coagulation Bath Temperature
DMAc	Dimethylacetamide
ELM	Emulsion Liquid Membrane
FESEM	Field Emission Scanning Electron Microscope
FSSLM	Flat Sheet Supported Liquid Membrane
HPLC	High Performance Liquid Chromatography
HFSLM	Hollow Fiber Supported Liquid Membrane
HCl	Hydrochloric Acid
HMF	Hydroxylmethylfurfural
LSR	Liquid Solid Ratio
PES	Polyethersulfone
SEM	Scanning Electron Microscope
NaOH	Sodium Hydroxide
H_2SO_4	Sulfuric Acid
SLM	Supported Liquid Membrane
TOA	Tri-n-octylamine
VIPS	Vapour Induced Phase Separation
ID	Internal Diameter
OD	Outer Diameter
G	Graphene
GO	Graphene Oxide

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