SILVER NANOPARTICLES EMBEDDED POLYAMIDE NANOFILTRATION MEMBRANE FOR ANTIMICROBIAL PROPERTIES

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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 degree of Master of Engineering (Chemical).

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I hereby declare that the work in this thesis is based on my original work except for quotations and citation 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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In the name of Allah, the Most Gracious the Most Merciful,

Special dedication to:

My parents,

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My husband,

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For all your care, supports and best wishes.

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ABSTRACT

In nanofiltration and other membrane processes, membrane fouling become a common problem that contributes to a major problem in water filtration. Fouling normally occurs due to natural organic matters like humic acid and various bacteria that already exist in the water. Some common methods like coagulation, flocculation, ion exchange, electrodeposition and extraction were used to remove pollutants from the water and wastewater. However, they encounter disadvantages which required series steps of heterogeneous reactions or substances distribution between different phases that normally require a lengthy operating period. Thin film composite membrane serves a physical barrier that can removes solids, bacteria, viruses and other unwanted molecules in such a better way. Previously, there were several attempts to produce polyamide membrane using different monomers. The aim of this present study was to produce antibacterial polyamide-silver nanofiltration thin film composite membrane using M-phenyldiamine and trimesoyl chloride as monomers on polyethersulfone membrane via interfacial polymerization for humic acid removal. The silver nanoparticles were synthesized from silver nitrate and polyphenols in tea extract was used as reducing agent. Success of silver formation were characterized by UV- Vis spectrophotometer, Transmission Electron Microscopy, X-ray Diffractometer and Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy. Antimicrobial properties of silver nanoparticles were tested against *Escherichia coli* bacteria. On the membrane preparation part, the variable change such as reaction time can produce good nanofiltration membrane. The effect of reaction time (dipping time) and the addition of silver nanoparticles onto the membrane were investigated. The success of formation of polyamide layer on membrane were determined by analysis of Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy and Field Emission Scanning Electron Miscroscopy. All membranes were characterized in term of chemical structure parameters, pure water permeability, rejection of different charged solutes (sodium chloride and sodium sulphate) and uncharged solute (Vitamin B12) and rejection of humic acid solution. These properties include hydrophilicity (θ), pore size (r_p) and effective thickness over membrane porosity ($\Delta x/A_k$). From the characterization, all polyamide membrane was considered as tight nanofiltration membranes. From the flux-loss analysis, polyamide-silver nanoparticles membrane with 60seconds reaction time exhibited the lowest total flux-loss which is 1.69% in the filtration of humic acid. The addition of silver nanoparticles with concentration of 60×10^{-3} g/mL onto the membrane slightly gave an effect to the membrane and reduced the fouling. This study will provide a valuable guideline to produce nanofiltration thin film composite membrane that have antibacterial properties for removal of humic acid in the water and wastewater filtration.

ABSTRAK

Dalam membran nanoturasan dan lain-lain proses membran, pengotoran merupakan masalah biasa yang menyumbang kepada masalah utama dalam proses penapisan air. Biasanya, pengotoran berlaku disebabkan oleh bahan organik semula jadi seperti asid humik dan dan pelbagai bakteria yang wujud di dalam air. Kaedah umum seperti pembekuan, pemberbukuan, pertukaran ion, elektrodeposisi dan pengekstrakan digunakan untuk menghilangkan bahan pencemar daripada air dan air buangan. Walau bagaimanapun, kaedah-kaedah ini terdapat kelemahan yang memerlukan beberapa siri tindak balas heterogen atau pengedaran bahan-bahan di antara fasa yang berbeza yang biasanya memerlukan tempoh operasi yang panjang. Membran komposit filem nipis berfungsi sebagai penghalang fizikal yang boleh menapis pepejal, bakteria, virus dan molekul yang tidak dikehendaki dengan cara yang lebih baik. Sebelum ini, terdapat beberapa percubaan untuk menghasilkan membran poliamida menggunakan monomer yang berbeza. Tujuan kajian ini adalah untuk menghasilkan membran komposit filem nipis poliamida-perak nanofiltration dengan menggunakan M-phenyldiamina dan trimesoyl klorida sebagai monomer pada membran polyethersulfone melalui pempolimeran antara muka untuk penyingkiran asid humik. Nanopartikel perak yang disintesis dari perak nitrat dan polifenol dalam ekstrak teh digunakan sebagai agen penurunan. Kejayaan pembentukan perak dicirikan oleh spektrofotometer UV-Vis, Mikroskopi Elektron Transmisi, Diffractometer sinar-X dan Total Reflectance-Fourier Transform Spektroskopi Inframerah. Ciri-ciri antimikrobial nanopartikel perak telah diuji terhadap bakteria *Escherichia coli*. Dalam bahagian penyediaan membran, pemboleh ubah seperti masa tindakbalas dapat menghasilkan membran nanoturasan yang baik. Kesan tindakbalas (masa pencelupan) dan penambahan nanopartikel perak ke dalam membran disiasat. Kejayaan pembentukan lapisan poliamida pada membran ditentukan dengan analisis Reflectance Total Reflectance-Fourier Transform Spektroskopi Inframerah dan mikroskop elektron pengimbas pancaran medan. Semua membran telah dicirikan dari segi parameter struktur kimia, kebolehtelapan air tulen, penolakan larutan yang bercaj (natrium klorida dan natrium sulfat) dan larutan tidak bercaj (Vitamin B12) dan penolakan larutan asid humik. Sifat-sifat membran ini termasuklah sifat hidrofilik (θ), saiz liang (r_p) dan ketebalan berkesan membran terhadap keporosan ($\Delta x/A_k$). Daripada pencirian membran, semua membran poliamida yang dihasilkan dipertimbangkan sebagai membran nanoturasan padat. Daripada analisis kehilangan fluks, poliamida-nanoperak membran dengan masa tindakbalas 60saat menunjukkan jumlah kehilangan fluks terendah iaitu 1.69% dalam penapisan asid humik. Penambahan nanopartikel perak dengan kepekatan $60x10^{-3}$ g/mL ke atas membran sedikit memberi kesan kepada membran dan mengurangkan pengotoran. Kajian ini akan memberi garis panduan yang berharga untuk menghasilkan membran komposit filem nipis nanoturasan yang mempunyai sifat antibakteria untuk penolakan asid humik dalam penapisan air dan air buangan.

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LIST OF SYMBOLS

Ag^0	Argentum colloid (silver colloid)
Ag^+	Argentum ion (silver ion)
A	Stokes radius, nm
$A_k/\Delta x$	Ratio of porosity to thickness of a membrane, m^{-1}
C_i	Concentration of ion in membrane, mol m^{-3}
C_b	Concentration of bulk solution, mol m^{-3}
$C_{i,b}$	Concentration of i component in bulk solution, mol m^{-3}
$C_{i,p}$	Concentration of permeate, mol m^{-3}
C_w	Concentration of solution at the membrane wall, mol m^{-3}
$D_{i,p}$	Permeate diffusivity, $m^2\ s^{-1}$
$D_{i,\infty}$	Bulk diffusivity, $m^2\ s^{-1}$
F	Faraday constant, C mol^{-1}
G	Hydronamic resistance coefficient
J_i	Flux of ion, mol m^2s^{-1}
J_v	Volume flux, m s^{-1}
J_w	Flux of pure water, m s^{-1}
k	Mass transfer coefficient, m s^{-1}
K^{-1}	Drag coefficient
$K_{i,c}$	Hindrance factor for convection
$K_{i,d}$	Hindrance factor for diffusion
M_w	Molecular weight, g mol^{-1}
P_{em}	Peclet number
P_m	Permeability, m s^{-1} Pa^{-1}
ΔP	Transmembrane pressure, kN m^{-2}
r	Radius of filtration cell, m
r_p	Pore radius, nm
r_s	Stokes radius, nm

R	Gas constant, J mol^{-1} K^{-1}
R_{obs}	Rejection from the observation
R_{real}	Real rejection
V	Solute velocity, m s^{-1}
X_d	Effective membrane charge density
z_i	Ion valency
λ	Ratio of solute radius to pore radius
μ	Solvent viscosity, Pa. s
ξ	Ratio of effective volume charge density of membrane to the electrolyte concentration of feed solution
Ψ	Electrostatic potential of the system, V
Ψ_D	Donnan potential difference, V
ω	Stirring speed of filtration cell, rad s^{-1}
ν	Kinematic viscosity

LIST OF ABBREVIATIONS

Abs	Absorbance
Ag	Silver
$AgNO_3$	Silver Nitrate
AgNPs	Silver Nanoparticles
ATR	Attenuated Total Reflectance
BPA	Bisphenol A
CA	Cellulose Acetate
E.coli	*Escherichia coli*
FA	Fulvic Acid
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
HA	Humic Acid
HCL	Hydrochloric Acid
HS	Humic Substances
IP	Interfacial Polymerization
MD	Membrane Distillation
MF	Microfiltration
MPD	M-phenyl Diamine
MWCO	Molecular Weight Cut Off
NaCl	Sodium Chloride
NaOH	Sodium Hydroxide
Na_2SO_4	Sodium Sulphate
NF	Nanofiltration
NOM	Natural Organic Matter
PA	Polyamide
PAN	Polyacrylonitrile
PE	Polyethylene

PEEK	Polyetheretherketone
PES	Polyethersulfone
PP	Polypropylene
PPESK	Poly(phthazine ether sulfone ketone)
PTFE	Poly(tetrafluoroethylene)
PVDF	Polyvinylidene Fluoride
PS	Polysulfone
RO	Reverse Osmosis
SHP	Steric Hindrance Pore
TEOA	Triethanolamine
TEM	Transmission Electron Microscopy
TFC	Thin Film Composite
TMC	Trimesoyl Chloride
UF	Ultrafiltration
UV-Vis	UV-Vis Spectrophotometer
XRD	X-ray Diffractometer

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