

**EFFECT OF SURFACTANTS ON PORE STRUCTURE AND
PORE PROPERTIES OF PHASE INVERSION
ULTRAFILTRATION (UF) MEMBRANE**

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AUTHOR DECLARATION

بسم الله الرحمن الرحيم

I hereby declare that the work in this thesis entitled “Effect of Surfactants on Pore Structure and Pore Properties of Phase Inversion Ultrafiltration (UF) Membrane” is my own research except for quotations and summaries which have been duly acknowledged. I allow this work to be reviewed by other students and anyone else for study, teach and research purpose. I also agree to allow Universiti Sains Islam Malaysia to make copies of this work for the same purpose.

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ABSTRAK

Pembangunan proses membran untuk pemisahan protein oleh ultraturasan (UF) kini mendapat perhatian terutamanya bagi industri komersial. UF adalah proses membran yang didorong oleh tekanan telah dibangunkan untuk pemisahan cecair fasa. Membran polimer adalah satu kaedah yang mudah dan berkesan untuk mereka-bentuk bahan-bahan baru untuk meningkatkan prestasi membran. Polyvinylidene fluorida (PVDF) adalah polimer kelas penting yang telah digunakan secara meluas dalam pembuatan tidak-simetri membran UF polimer. Berdasarkan pada kering/basah teknik fasa penyongsangan, membran UF tidak simetri yang licin dan nipis telah disediakan. Membran yang disediakan telah dianalisis bercirikan Imbasan mikroskop elektron (SEM), Fourier spektroskopi inframerah (FTIR), dan teknik analisis gravimetrik termal (TGA). Prestasi semua membran yang disediakan telah dinilai dari segi penyerapan air tulen (PWP) pada tekanan operasi dari 50 hingga 300 kPa, penolakan fluks dan penyingkiran Bovine albumin serum (BSA) juga albumin telur (EA) protein pada 100 kPa. Berdasarkan kajian prestasi protein perpisahan, PVDF/NMP membran yang mengandungi 17% berat polimer membran menunjukkan prestasi yang terbaik kerana menghasilkan produktiviti air yang tinggi ($455.82 \text{ Lm}^{-2}\text{h}^{-1}$) dan prestasi pemisahan (84% BSA, 73% EA), juga kerana ia mempunyai struktur membran morfologi yang baik. Keputusan menunjukkan bahawa penambahan 3% berat Polyvinylpyrrolidone (PVP) dan Polyethylene glycol (PEG 200) membentuk membran dengan protein yang lebih tinggi penolakan dan dengan itu, liang membran telah bertambah baik. Kedua-dua PVP dan PEG 200 bahan tambahan telah dipilih sebagai bahan tambahan terbaik ke arah membran PVDF-UF. Keputusan eksperimen menunjukkan bahawa penambahan bahan surfaktan terhadap PVP meningkatkan PWP dalam lingkungan $577.37\text{-}811.92 \text{ Lm}^{-2}\text{h}^{-1}$ manakala, ke arah PEG 200 PWP meningkat dalam lingkungan $78.97\text{-}412.41 \text{ Lm}^{-2}\text{h}^{-1}$. Dari segi prestasi pemisahan BSA, PVDF/NMP/PVP membran dengan penambahan 2% berat Tween 80 menunjukkan penyingkiran protein tertinggi 90% pada kadar flux rendah ($105.51 \text{ Lm}^{-2}\text{h}^{-1}$). Penambahan surfaktan ke arah PVDF/NMP/PEG 200 membran, telah mengakibatkan peningkatan fluks meresap dan menurunkan penyingkiran protein. Dalam hubungan untuk mencapai prestasi pemisahan tinggi, gabungan daripada 1% berat surfaktan anionik dan surfaktan bukan ionik iaitu SDS, Tween 80 dan Triton X-100 terhadap prestasi dan struktur morfologi telah dikaji. Eksperimen dan pencarian data pengasingan protein menunjukkan bahawa SDS/Tween 80 dan Triton X-100/Tween 80 didapati memberi kesan kepada prestasi, morfologi dan liang membran AUF-PVDF. Oleh itu, dengan pakej prestasi pemisahan tinggi dan butiran struktur halus, membran AUF-PVDF yang disintesis juga disediakan potensi teknikal yang cemerlang ke arah pembangunan membran.

ABSTRACT

The development of the membrane process for proteins separation by ultrafiltration (UF) is now get attention especially for commercial industry. Polymeric membrane is a simple and efficient method for designing new materials to improve the performance of the membranes. As continuous developed of new membrane materials, Polyvinylidene fluoride (PVDF) is an important class of polymer that has been widely used in the manufacture of asymmetric polymeric UF membrane. Based on the dry/wet phase inversion technique, the smooth, even and thin asymmetric UF membranes were prepared. The prepared membranes were characterized by Scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and Thermal gravimetric analysis (TGA) techniques. The performance of all the membranes prepared have been evaluated in terms of pure water permeation (PWP) at operating pressure from 50 to 300 kPa, permeate flux and Bovine serum albumin (BSA) also Egg albumin (EA) proteins rejection at 100 kPa. Based on the proteins separation performance study, the PVDF/NMP membrane which contains 17 wt% of polymer show the best performance membrane due to its high water productivity ($455.82 \text{ Lm}^{-2}\text{h}^{-1}$) and separation performance (84% BSA, 73% EA) as well as it has good membrane morphological structure. The results revealed that the addition of 3 wt% Polyvinylpyrrolidone (PVP) and Polyethylene glycol (PEG) 200 were formed membrane with higher proteins rejection and thus, the membrane pores were improved. Both of PVP and PEG 200 additives were chosen as the best additive towards the PVDF-UF membranes. Besides that, the incorporating of surfactant materials which are Sodium dodecyl sulfate (SDS), Triton X-100 and Tween 80 towards PVP increased the PWP in the range of $577.37\text{--}841.92 \text{ Lm}^{-2}\text{h}^{-1}$ meanwhile towards PEG 200, the PWP increased in the range of $78.97\text{--}412.41 \text{ Lm}^{-2}\text{h}^{-1}$. In term of BSA separation performance, the PVDF/NMP/PVP membrane fabricated with addition of 2 wt% Tween 80 showed highest rejection of 90% at low permeate flux ($105.51 \text{ Lm}^{-2}\text{h}^{-1}$). The addition of surfactant towards the PVDF/NMP/PEG 200 membrane were resulted in increase the permeate flux and decreased the proteins rejection. In relations to attain high separation performance, the effect combination of 1 wt% anionic and non-ionic surfactants that are SDS, Tween 80 and Triton X-100 formulations were investigated. Experimental and characterization data on protein separation demonstrated that the SDS/Tween 80 and Triton X-100/ Tween 80 were found to affect the performance, morphologies and membrane pores of AUF-PVDF membrane. Therefore, with the packages of high separation performance and fine structural details, the synthesized AUF-PVDF membrane also provided the excellent technical potentials towards membranes development.

ملخص البحث

تطوير عملية الفصل للغشاء البروتيني بواسطة الترشيح الفائق ' Ultrafiltration (UF) والتي تحضى الآن باهتمام كبير خاصة بالنسبة للقطاع التجاري. الأغشية البوليمرية هي طريقة بسيطة وفعالة لتصميم مواد جديدة لتحسين أداء الأغشية. والاستمرار في تطوير الأغشية عن طريق مواد جديدة، فلوريد البولي فينيل (PVDF) هو البوليمر الأكثر أهمية للطبقة التي استخدمت على نطاق واسع في صناعة أغشية UF البوليمرية غير المتماثلة. واستنادا على المرحلة التقنية العكسية (الجافة / الرطبة)، كما ويتميز الأغشية التي تم تحضيرها بواسطة تقنيات SEM، FTIR، TGA باللمس الناعم والسمك الرقيق من أغشية UF اللامتماثلة. وقد تم تقييم أداء جميع الأغشية التي أعدت من حيث نفاذ الماء النقي (PWP) تحت ضغط 50-300 كيلو باسكال، حيث تم التدفق والنفاذ ورفضت البروتينات عند 100 كيلو باسكال. والهدف من هذه الدراسة هو تحديد تأثير التراكيز المختلفة للبوليمر (13_19٪ بالوزن) في المحلول من حيث الأداء والخصائص المميزة على تصنيع أغشية الفصل البروتينية. واستنادا على دراسة أداء فصل البروتينات، أغشية كل من PVDF و NMP التي تحتوي على 17٪ بالوزن من البوليمر تظهر أفضل أداء للأغشية بسبب إنتاجيتها عالية للمياه ($455.82 \text{ Lm}^{-2}\text{h}^{-1}$) وأداء الفصل (84% BSA, 73% EA) فضلا عن أن لديها هيكل جيد لغشاء الصرف. تمت دراسة تأثير المواد المضافة المختلفة والتي هي بولي فنانيل بايروليدون (PVP)، ليثيوم كلوريد (LiCl) والبولي إيثيلين جلايكول (PEG 200) على أفضل غشاء ل (PVDF / NMP). وكشفت النتائج أن إضافة 3٪ بالوزن PVP و PEG 200 تقلل حجم المسام كما في المسام السابق وبالتالي تكوين غشاء لديه أعلى نسبة رفض للبروتينات (85% نسبة الرفض لأغشية PVP و PEG 200) وبالتالي تم تحسين المسامات للغشاء. وقد تم اختبار كل من PVP و PEG 200 كأفضل مواد مضافة إلى أغشية PVDF-UF. وعلاوة على ذلك، هناك ثلاثة أنواع المختلفة من المواد السطحية والتي هي الصوديوم دوديسيل كبريتات (SDS)، تريتون X-100، وتوين 80 أدخلت إلى PVP و PEG 200. وظهرت نتائج التجارب أن إضافة عنصر من المواد السطحية إلى PVP زاد نسبة نفاذ المياه النقية (PWP) إلى ($577.37-811.92 \text{ Lm}^{-2}\text{h}^{-1}$) وكذلك عن اضافته إلى PEG 200، زاد نسبة نفاذية المياه النقية (PWP) إلى ($78.97-412.41 \text{ Lm}^{-2}\text{h}^{-1}$). أثناء أداء الفصل BSA وعند تصنيع غشاء PVDF/NMP/PVP وإضافة 2٪ بالوزن من توين 80 أظهرت النتائج أعلى نسبة رفض 90 وكذلك انخفاض تغلغل أو نفاذية التدفق (بما عند غشاء PVDF / NMP / PEG 200) وعند إضافة المواد السطحية زاد تغلغل أو نفاذية التدفق وانخفضت نسبة رفض البروتينات. وأخيرا، تمت دراسة دور المواد السطحية بنسبة 2٪ بالوزن في تشكيل نشاط الترشيح الفائق PVDF لأغشية (AUF). في العلاقات لتحقيق أعلى أداء للفصل تم التحقيق من تأثير مزيج من المواد السطحية الأنيونية وغير الأيونية والتي هي صيغ من SDS، توين 80 و تريتون X-100. وعلاوة على ذلك، فإن تأثير المواد السطحية يمزج على مسام الغشاء تم فحصه أيضا. كما و برهنت التجارب السابقة والبيانات والمعطيات في مجال فصل البروتين على أن SDS / توين 80 و تريتون X-100 / توين 80 وجدت لتؤثر على الأداء، والأشكال الهيكلية لمسام الغشاء -AUF PVDF من تم حزم انفصال عالية الأداء والتفاصيل الهيكلية الدقيقة، وايضا توليف أغشية AUF-PVDF زودت من الإمكانيات الفنية ممتازة نحو تطوير الأغشية.

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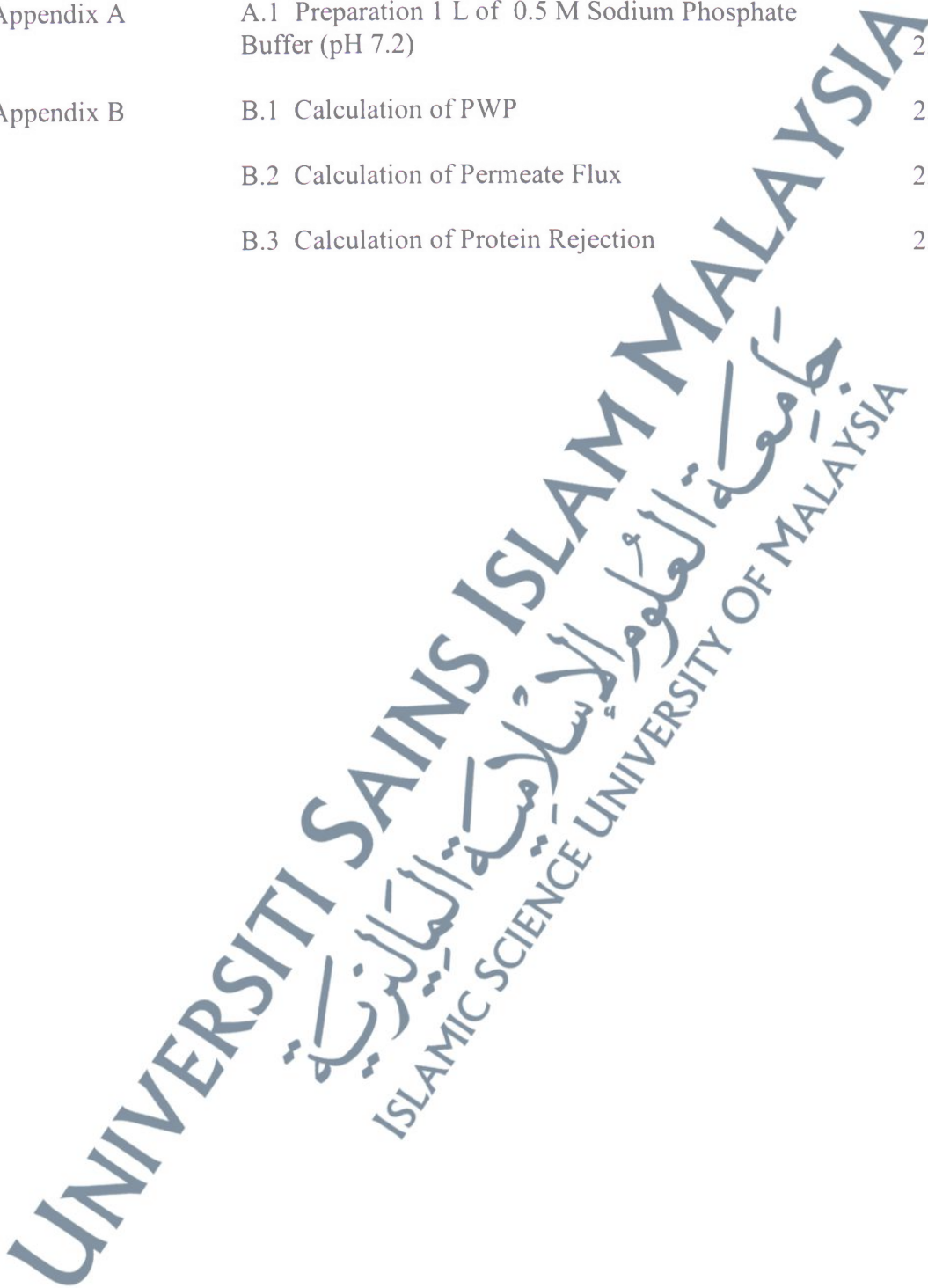
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ABBREVIATIONS

°C	Degrees Celsius
µm	Micrometer
Å	Angstrom
kPa	Kilopascals
Da	Dalton
MW	Molecular Weight
MWCO	Molecular Weight Cut-Off
mg	Milligram
COD	Chemical Oxygen Demand
BOD	Biochemical Oxygen Demand
SS	Suspended Solids
CMC	Critical Micelle Concentration
PVDF	Polyvinylidene difluoride
PSF	Polysulfone
PES	Polyethersulfone
CA	Cellulose acetate
APES	Aminated polyethersulfone
SPEI	Sulfonated poly(ether imide)
PAN	Polyacrylonitrile
PI	Polyimide
PEI	Polyethylenimine
NMP	N-methyl-2-pyrrolidone
DMAc	Dimethylacetamide
DMF	Dimethylformamide
DMSO	Dimethylsulphoxide
UF	Ultrafiltration
MEUF	Micellar-Enhanced Ultrafiltration
MF	Microfiltration
UF	Ultrafiltration
UFS	Ultrafiltration-Surfactant
NP-UFS	Narrow-Pores Ultrafiltration-Surfactant

NF	Nanofiltration
RO	Reverse Osmosis
PVP	Polyvinylpyrrolidone
LiCl	Lithium Chloride
PEG	Poly(ethylene Glycol)
SDS	Sodium Dodecyl Sulphate
CTAB	Cetyl Trimethyl Ammonium Bromide
SEM	Scanning Electron Microscope
FTIR	Fourier-Transform Infrared Spectrophotometer
IR	Infrared
TGA	Thermogravimetry Analysis
NMR	Nuclear Magnetic Resonance
AFM	Atomic Force Microscopy
PWP	Pure Water Permeation
BSA	Bovine Serum Albumin
EA	Egg Albumin