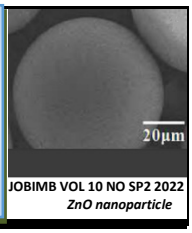
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Development of Polyethersulfone Nanofiltration Membrane with Layer-by-Layer Method for Xylitol Purification

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ABSTRACT

This work is aimed to develop polyethersulfone (PES) nanofiltration (NF) membrane with layer-by-layer (LBL) polyelectrolytes of chitosan (CHI) and poly(acrylic acid) (PAA) for xylitol purification from fermentation broth. Different number of bilayers and type of terminating layer were manipulated for producing more hydrophilicity, negatively charged with improved performance compared to pristine PES membrane. Successful deposition of polyelectrolyte layers onto PES membrane was able to be proven using various tests such as contact angle, Zetasizer and FT-IR. The results obtained have proven that LBL can develop PES membrane with higher resistance to fouling. From Zeta potential analysis, the value of pristine PES membrane's negativity confirmed the theory of negatively charged substrate for LDL. The negative value of PES membrane increased from -16.5 mV to -32.7 mV after being modified to PES $(CHI/PAA)_6$. From FT-IR spectra, the formation of CHI/PAA complexes on the membrane's surface is confirmed through the presence of stretching peaks of -COOH, $-NH^{3+}$ and $-NH^{2+}$ groups The pure water flux reduces from 47.40 ±6.30 L/m^2.h to 7.40 ±1.64 L/m^2.h after being modified to PES $(CHI/PAA)_2$. The rejection performance of xylitol for PES $(CHI/PAA)_2$ is higher (84.95%) than pure PES membrane (66.17%), while $(CHI/PAA)_4$ offered the lowest selectivity towards xylitol than arabinose and thus able to obtain higher purity of xylitol as retentate. LBL surface modification using CHI/PAA can develop PES membrane with higher hydrophilicity, negatively charge, and is able to give better xylitol rejection compared to pure PES.

INTRODUCTION

Xylitol is a rare sugar with molecular formula of $(CHOH)_3(CH_2OH)_2$. It is commonly known for its ability to substitute conventional sweetener by offering the same level of sweetness but with 33% less calories compared to sucrose (Martínez et al., 2015). Unlike other sweetener, this polyalcohol brings benefit to health by preventing carries on teeth, promote enamel's remineralization and does not affect insulin level of human when consumed. Therefore, xylitol is suitable for diabetic and hypertension patients. The surge of demand for xylitol in food and drugs industry have led to the need to improve xylitol production process.

Xylitol is produced in commercial scale via two route; chemical process and biotechnology process (Granström et al., 2007). In chemical process, D-xylose is reduced via hydrogenation to produce xylitol. This process is unattractive as it requires catalyst, high temperature, pressure, and the need to use pure xylose as feed. In the other hand, biotechnology process offers lower production cost and can produce high purity xylitol. This process is also sustainable as it uses agricultural wastes as

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feed to obtain xylose. Biotechnology process uses microbes (yeast, bacteria, and fungi) and enzymes as hydrogenating agent. In order to obtain high purity xylitol, studies have been conducted to improve the separation in downstream process. Past studies have shown that membrane technology is able to offer a better separation performance (82%) compared to crystallization (75%) and adsorption method (65%) (Faneer et al., 2017b) especially for the separation of xylitol.

Polymer membrane, particularly PES have been drawing attention in separation process as it has good mechanical strength, stable towards oxidizing agent and high heat compared to other polymer membrane (Ladewig & Al-Shaeli, 2017). From past study, PES was proven to be able to purify xylitol from fermentation broth (Faneer et al., 2016; 2017a, 2017b; 2017c). However, PES have relatively high hydrophobicity compared to other polymeric membrane. This property contributes to its tendency to have fouling during filtration process. There are several methods have been tested to improve PES membrane's hydrophilicity such as blending using copolymer and nano particles. According to Faneer et al. (2017b), separation of xylitol from xylose sugar is still a challenge as their molecular weight is almost similar. Further PES development is needed to improve the separation of xylitol from xylose in fermentation broth.

One interesting approach is to modify membrane by coating its surface using polyelectrolyte. This modification can be done using layer-by-layer (LBL) dynamic method and the use of weak polyelectrolytes such as poly(acrylic acid)(PAA) and chitosan (CHI). Weak poly- electrolytes are proposed as it is possible to tailors the thickness of bilayers formed by manipulating its degree of ionizations. This can be done by selecting a suitable local pH values for the polyelectrolytes (Choi, 2004). This charged layers will improve membrane's hydrophilicity by attracting water molecules and also ensuring that the modified membrane is in NF range by entering the membrane's pore (Zhao et al., 2011).

In this study, polyelectrolyte pair such as CHI/PAA is of interest. Chitosan is a linear polymer consisted of glucosamine. The presence of amino group with pKa 6 in chitosan enables the polyelectrolyte to be protonated using acidic to neutral liquid medium. Due to its ability to dissolve in water and bio-adhesive, chitosan is attracted by negatively charged surface (Sun, 2015). On the other hand, PAA is a soluble polymer with high charge density. This is due to its nature as weak polyelectrolyte. As other weak polyelectrolytes, PAA is very sensitive to pH. Both are non-toxic chemicals, which are suitable for LBL application in food industry. According to Zhu et al. (2015), CHI/PAA forms supramolecular co-polymer as –COOH group on PAA interacts with amino and hydroxyl group from CHI. The intramolecular interactions present are due to hydrogen bond.

In this paper, the fabrication of PES membrane, LBL surface modification using dynamic LBL, and the effect of LBL parameters such as number of bilayers deposited and type of terminating layer on the physical, chemical properties and the performance on purification of xylitol of the modified membrane are discussed. PES 18% was fabricated and its performance was studied for xylitol purification by previous researches (Faneer et al., 2016; 2017a, 2017b; 2017c).

MATERIALS AND METHODS

Materials

The materials used in this study were obtained in analytical grade. Polyethersulfone in granule form (Goodfellow,USA) was used as membrane's polymer. N-methyl-2-pyrrolidone (NMP) of high purity (99.7%) (Fluka, Germany) was used as solvent and distilled water were used as the non-solvent component. Two types of polyelectrolytes; poly(acrylic acid) (PAA) and chitosan (CHI) (Sigma Aldrich, USA) were used to modify the membrane. Acetic acid (Sigma Aldrich, USA) was used to prepare CHI solution. Sodium hydroxide, NaOH and hydrochloric acid (HCl) was obtained from Friendemann Schmidt. Xylitol, arabinose (Sigma Aldrich, USA), xylose (Sigma Aldrich, China) and sodium bisulphate, $NaHSO_3$ (Fluka, Germany) were used for fermentation broth modelling.

Membrane preparation

Membrane fabrication using phase inversion method

PES NF membrane was fabricated via phase inversion by using immersion precipitation technique. First, obtained PES granules were dried at temperature of 60 °C for 72 hours to remove trapped moisture prior to use. The polymer dope solution was prepared by adding 18%wt of PES granules into 82%wt of NMP. The solution was stirred at 450 rpm for 8 hours, at temperature of 60 °C to ensure the granules were completely dissolved in the solvent. The obtained dope solution was left for degassing at room temperature for 24 hours. In order to obtain flat sheet membrane, casting machine (Elcometer 4340, Germany) was used. A suitable amount of dope solution was poured carefully on a clean glass sheet, and casted to 150 µm of thickness using the casting knife. The covered glass sheet was then immersed in a water coagulation bath for precipitation process to occur, the membrane was left for 2 hours at room condition to allow solvent exchange and then the non-solvent, DI water was replaced for a few times for a complete solvent exchange.

Membrane modification using dynamic layer-by-layer (LBL) method

The fabricated PES membrane was modified by depositing polyelectrolyte layers onto its active surface using dynamic LDL. Both polyelectrolytes were prepared at the same concentration, 4mg/mL. Distilled water was used to dissolve PAA while 2% acetic acid was used for chitosan. To tune the pH of both solutions into desired value (pH 3 for PAA, pH 2.8 for PAA), 1 M of HCl and NaOH were used (Yang et al., 2017). In dynamic LDL, a membrane holder was used. The fabricated PES membrane was cut into a circle with 2.45 cm radius before it was modified. Since PES membrane was a negatively charged membrane, the first layer deposited was polycation, CHI. 10 ml of CHI solution was poured into the holder containing the membrane and left for 15 minutes. After that, the solution was removed, the holder and the membrane were rinsed using distilled water for 1 minute before 10 ml of polyanion, PAA was poured to form the first CHI/PAA bilayer. The process was repeated until *n* number of bilayers formed. The modified membrane was stored in distilled water until further use. In this report, the PES membrane modified with n number of CHI/PAA bilayer is referred to $(CHI/PAA)_n$. Membrane terminated with CHI layer is referred as $CHI/(PAA)_{n.5}$ where n is the number of bilayer deposited.

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Membrane Characterization

The membranes hydrophilicity was measured via contact angle (Model Kruss GmbH, Germany with Drop Shape Analysis software with a 60.10° accuracy). Prior investigating, the membranes have been dried for 72 h.

The membrane surface and cross-sectional morphology was carried out by using FESEM (Zeiss 55VP SUPRA, USA), in which the membranes samples were initially prepared by soaking in liquid nitrogen (N2) to crack membranes' top and cross section.

Zeta potential of membrane surface was measured in water at pH 7 using polystyrene latex particles as the tracer particles, with Malvern Surface Zeta Potential Cell (Malvern Instruments, UK) by applying field strength of 25 V/cm.

Preparation of fermentation broth model

Fermentation broth model was used to study the application of developed membrane on xylitol purification. The following **Table 1** summarizes the composition of broth model used in this work. The composition is adapted from Mussatto et al. (2007) and Affleck (2000).

Table 1. Composition of fermentation broth model.

Components	Concentration (g/L)
Xylitol	19.10
Xylose	1.44
Arabinose	2.70
Sulfate salt	0.11

PES membrane filtration performance

Prior to testing, the modified membrane was compacted with DI water at 12 bars until steady water flux was achieved in 2 hours. This step was carried out by using a dead-end filtration cell (Rohani et al., 2011). Next, pure water flux (PWF) was measured at 4 bars. Average measurement was obtained to minimize inaccuracy. Equation (1) was applied to determine the PWF, where J_w was value of water flux in L/m^2h, V was the permeate volume in L, A was the area of membrane (1.46×10^{-3} m^2) and t was the time taken for filtration in h.

$$J_w = V/A.t. \quad \text{(Eqn. 1)}$$

To evaluate xylitol rejection by the membrane, the model broth solution was filtered in batch of 300 ml by using a dead-end cell. The test was carried out at same pressure of 4 bars. Equation (2) was used to evaluate the rejection of xylitol, where R was the rejection in %, C_p was the xylitol concentration in permeate and C_f was the initial concentration of xylitol in feed.

$$R\% = [1-(Cp/Cf)]*100 \quad \text{(Eqn. 2)}$$

The rejection value obtained was then used to analyse the membrane's xylitol/sugar separation performance in terms of separation factor, S as shown by **Eqn. 3**.

$$S = [100-R_{xylitol}/100-R_{sugar}]*100 \quad \text{(Eqn. 3)}$$

RESULTS AND DISCUSSION

Fabrication of polyethersulfone membrane using phase inversion method

18% PES membrane was successfully fabricated in flat sheet form using phase inversion and immersion coagulation method. From **Fig. 1** shown below, the membrane is seen to be porous, asymmetric; thin, dense upper layer and having finger-like micro-voids on the sublayer. This particular structure enable the membrane to have good permeate flow and mechanically strong at the same time (Buonomenna et al., 2011).

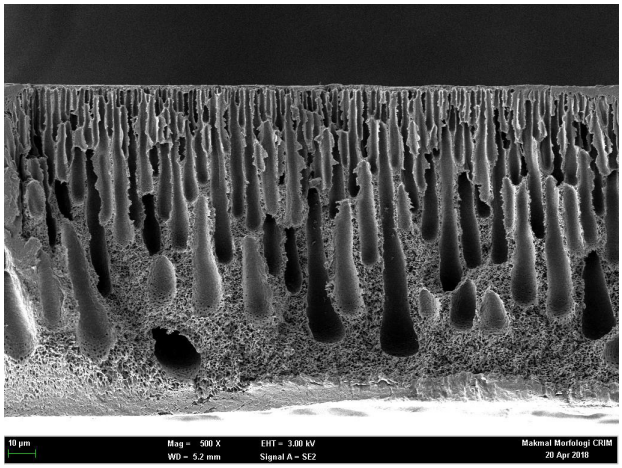


Fig. 1. Cross section of PES membrane using SEM imaging.

During the fabrication of membrane, "old lady" symptom was formed on the membrane. This symptom refers to the wrinkling phenomena occurred after the membrane was immersed in the coagulation basin. Membrane with this symptom is not acceptable to be used as it has a non-smooth surface. **Fig. 2** shows the difference between acceptable and rejected membrane due to "old lady" formation.

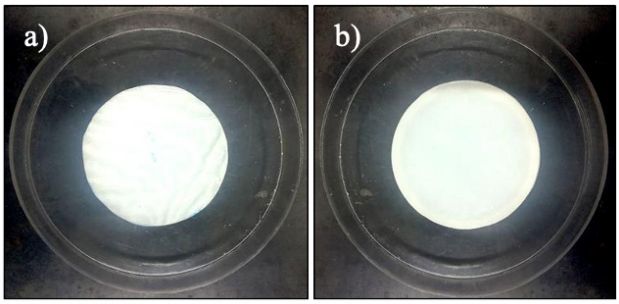


Fig. 2. Pure PES membrane (a) with "old lady" formation throughout the membrane, (b) without "old lady" formation.

Membrane characterization

Contact angle

The value of contact angle quantifies the property of hydrophilicity or wetness of the membrane. When the contact angle of a membrane is smaller, it has higher hydrophilicity and thus, has lower tendency to fouling.

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The results and comparison of contact angle between pure PES and modified membrane are shown in **Fig. 3**. From **Fig. 3**, the modified membranes have lower contact angle than pure PES. Membrane $(CHI/PAA)_4$ can reduce the contact angle from 76.5±0.19° to 34.4 ± 0.31°. It is also noticed that membrane with 2 bilayers terminated with different polyelectrolytes exhibit different contact angle. When terminated with PAA, $(CHI/PAA)_2$ has lower contact angle than $(CHI/PAA)_{2.5}$ which is terminated using CHI. This results shows that terminating polyelectrolyte multilayers membrane (PEM) using polyanion produces a more hydrophilic membrane (Ng et al., 2013). By comparing the modified membrane, the membrane with 4 bilayers, $(CHI/PAA)_4$ poses a lower contact angle compared to 2 and 6 bilayers probably due to optimum electronegativity charge presence in the membrane (to be confirmed in the following characterization). From the previous study of PES 18% modification for xylitol purification, LBL modification produced the most hydrophilic membrane as shown in **Table 2**.

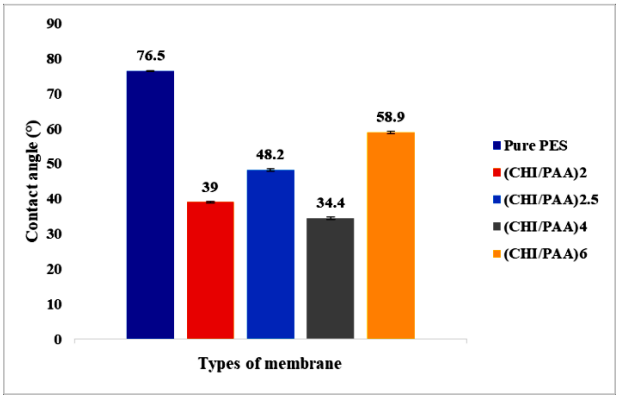


Fig. 3. Comparison of contact angle for pure and modified PES membrane.

Table 2. Comparison of contact angle obtained from different modification method of PES 18%.

Modification	Contact angle
Blending with 5% SiO_2	59.1°±0.15°
Blending with 5% Pluronic	61.7° ± 2.24°
Blending with 1% TiO	68.6° ± 2.16°
LBL $(CHI/PAA)_4$	34.4° ± 0.31°-

Zeta potential

The zeta potential of a membrane is used to define the magnitude of electrostatic repulsion and attraction force (charge) of the surface. The higher the value of negativity of membrane, the greater the Donnan effect. Donnan effect is an important mechanism in reducing membrane's fouling and also aids in separation performance (Childress et al., 2012). Negatively charged surface will repel cationic foulant (Choudhury et al., 2018). From **Fig. 4**, the negativity value (zeta potential) of PES membrane increased after modification using LBL, terminated using PAA layer. The greater the number of bilayers deposited, the greater the value of negativity of the membrane. The value of negativity of membrane increased from -16.5 mV to -32.7 mV when 6 bilayers of CHI/PAA was deposited. According to Avram (2017), at higher value of negativity, the dominance of electrostatic effect increases, but at a very high value, the separation of PEM is limited to sieving effect as too much resistance is added by the multilayer.

It is observed that terminating PEM with polycation CHI reduces the negativity of the membrane. This proves that PAA is more anionic than CHI. Theoretically, like-charged molecules is rejected, while oppositely-charged molecules is attracted by the negatively charged surface (Tieke & Krasemann, 2000). In this study, it is believed that Donnan effect aids in the xylitol purification by rejecting negatively charged bisulphate salt. When the PEM is terminated with PAA, the rejection of the salt is due to the presence of negatively charged $–COO^-$ at the PAA structure (Park et al., 2010).

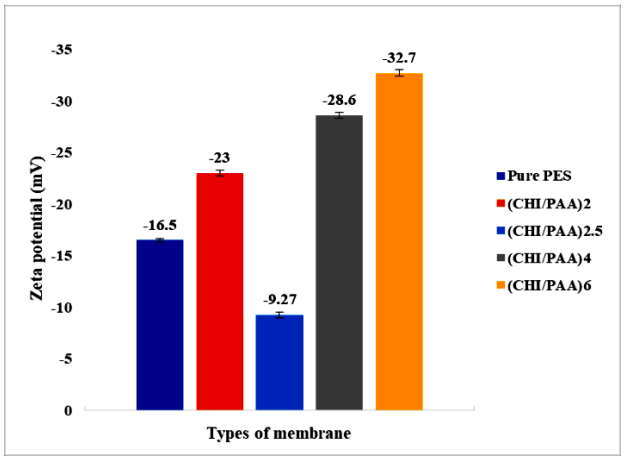


Fig. 4. Comparison of zeta potential for pure and modified PES membrane.

Fourier transformation infrared spectroscopy (FT-IR)

FT-IR analysis is used to study the presence and the abundance of chemical components such as CHI/PAA complex in modified membrane. This analysis is to confirm the formation of CHI/PAA complexes and the success of multilayer deposition on the surface of PES. **Fig. 5** shows the full FT-IR spectra of different types of PES membrane in function of transmittance (%).

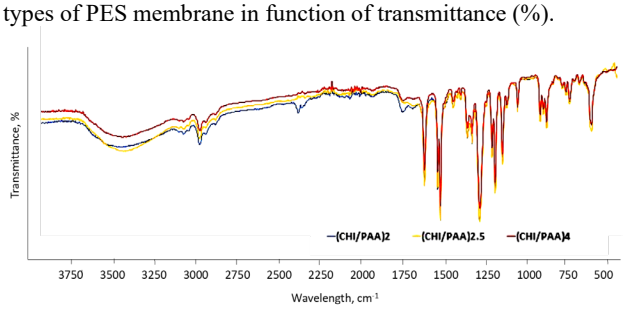


Fig. 5. FT-IR spectra of modified PES membrane.

Fig. 6 shows the layered spectra of the same data to illustrate the difference of absorbance at frequency of -C=O bond stretch (from COOH group of PAA) at range area of 1620-1780 cm^{-1}. The presence of CHI can be confirmed by observing the peaks for $–NH^{3+}$ and $–NH^{2+}$ group at the wavelength number 1538 cm^{-1} and 1623 cm^{-1} (Zhu et al., 2015) as shown in **Fig. 7**. From the FT-IR results, it is observed that the intensity of absorbance peaks increases with increase in bilayer deposited. This indicates that there is an accumulation and growth of polyelectrolytes multilayer on the surface The same trend is also observed by (Baburaj et al., 2012) using CHI/PAA on Nylon. The presence of –COOH, $–NH^{3+}$ and $–NH^{2+}$ groups in the spectra proved that CHI/PAA complex was formed during LBL.

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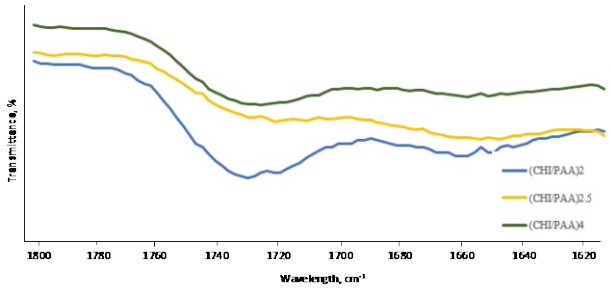


Fig. 6. FT-IR spectra at stretch area of -COOH group.

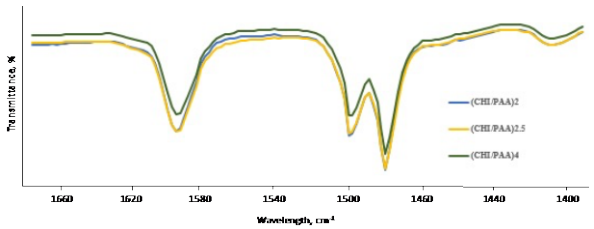


Fig. 7. FT-IR spectra at stretch area of $-NH^{3+}$ and $-NH^{2+}$ group.

Filtration and separation properties

Water and permeate flux

The performance of NF membrane used in liquid separation is characterized by water and permeate flux. **Fig. 8** shows the water flux and sugar flux for pure and modified PES membrane. For pure PES, 10% difference between water and permeate flux indicate that there is more resistance occurred when the sugar solution tried to permeate through the membrane. After modification using LBL, the water flux of PES dropped drastically at 93.94% slower when 6 bilayers of CHI/PAA are deposited. This effect might be due to the deposition of polyelectrolytes, not only on the surface, but also in the pores of the membrane. By comparing the flux among the modified membrane, the number of bilayers has a small effect on the water flux. Meanwhile, the pattern of reduction in the permeate flux is more pronounced with the increase of bilayer number. This is probably due the additional resistance presence by Donnan effect on ions that are present in the sugar broth model.

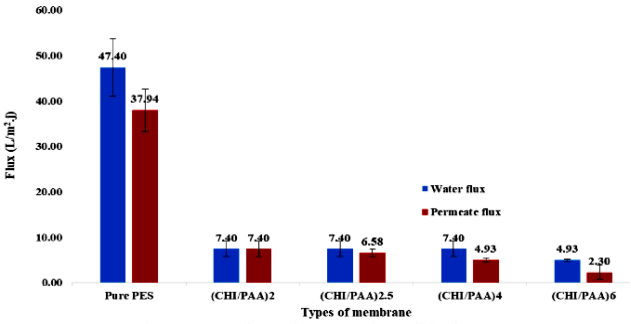


Fig. 8. Water and permeate flux of pure and modified PES.

Xylitol rejection test

The concentration of each component in permeate are obtained using HPLC U3000-RefractoMax 520 and determined by using equation (2). The comparison of the amount of each broth component are tabulated in **Table 3**. From **Table 3**, there is no bisulphate salt presence in the permeate after filtration. This indicates that the salt remains in the retentate side and unable to pass through the membrane. The rejection of salt is due to both the steric and Donnan effects as the bisulfate ion is negatively charged.

Table 3. Amount of each component before and after filtration.

Component	Concentration in feed (g/L)	Concentration in permeate (g/L)				
		Pure PES	$(CHI/PAA)_2$	$(CHI/PAA)_{2.5}$	$(CHI/PAA)_4$	$(CHI/PAA)_6$
Xylitol	19.1	6.46	2.47	6.41	2.87	2.47
Xylose	1.44	0.31	0.44	0.33	0.15	0.44
Arabinose	2.7	0.77	0.20	0.54	0.57	0.19
Bisulfate salt	0.11	0.00	0.00	0.00	0.00	0.00

Table 4 shows the rejection performance of the pure and modified PES membranes by using **Eqn. 3**. Overall, the performance of sugar rejection for the modified membrane, terminated using PAA, is found to be better than the pure membrane. By modification using LBL, the deposition of the polyelectrolyte on the surface and inside the pores of the membrane have increased the sieving effect. Large neutral sugar molecules have to overcome a greater resistance to pass through the membrane. However, xylitol rejection decreases with the increase in the number of bilayer while there are no obvious and common patterns for other sugar components.

Table 4. Rejection performance of pure and modified PES.

Type of membrane	Rejection (%)			
	Xylitol	Xylose	Arabinose	Bisulfate salt
Pure PES	66.17	78.22	71.44	100.00
$(CHI/PAA)_2$	87.06	69.33	92.90	100.00
$(CHI/PAA)_{2.5}$	66.45	77.26	79.87	100.00
$(CHI/PAA)_4$	84.95	89.83	78.80	100.00
$(CHI/PAA)_6$	80.34	87.98	89.56	100.00

By comparing the rejection performance of $(CHI/PAA)_2$ and $(CHI/PAA)_{2.5}$, the modified membrane terminated with CHI layer has a lower rejection towards xylitol even though both type of membrane has 2 bilayers. This result indicates that the outer surface charge contributes to the separation performance of neutral sugar molecules. Theoretically, Donnan effect (charge of surface) would not affect the rejection of sugar molecules as they are neutrals. However, the presence of salt in the broth may influence the charge of the sugar molecule by forming a salt-sugar complexed. According to the past study by Kim et al. (1985), the researcher found out that the presence of salt such as NaCl, Na_2CO_3 and $NaHSO_3$ could increase the separation selectivity of fructose and glucose. The addition of salt component to sugar mixture may form a complex exclusively to affect the sugar components compared to others. These phenomena introduced the selectivity between sugars. Therefore, the sugar separation in the broth is not only dependent on sieving effect, but also dependent to Donnan repulsion and attraction.

Measurement of separation factor between xylitol and sugars

Table 5 shows the selectivity of PES membranes towards xylitol. The separation factor of xylitol/xylose for the pure PES membrane was 1.553. The value dropped to 0.422 when the membrane was modified using $(CHI/PAA)_2$. With separation factor of lower than 1, the result implies that the membrane was able to retain xylitol as a retentate, while xylose is separated as permeate. According to Faneer et al., (2017b), among significant challenge in purifying membrane is to separate xylitol from arabinose as both sugar are almost similar in size. From the result, PES $(CHI/PAA)_6$ has the best performance in separating xylitol/arabinose with separation factor as low as 0.710. Although the membrane has a high selectivity toward xylose than xylitol, in overall, it is identified as the best membrane to purify xylitol because the xylitol/arabinose separation performance is found more significant in this study. Xylitol is preferred to be at

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the retentate based on its molecular weight compared to other sugars presence. Low separation factors are desired between xylitol and other sugars.

Table 5. Xylitol/sugar separation factor of PES membranes.

Type of membrane	Separation factor, S	
	Xylitol/Xylose	Xylitol/Arabinose
Pure PES	1.553	1.185
$(CHI/PAA)_2$	0.422	1.822
$(CHI/PAA)_{2.5}$	1.476	1.666
$(CHI/PAA)_4$	1.479	0.710
$(CHI/PAA)_6$	1.635	1.884

CONCLUSION

As a conclusion, fabrication of PES 18% in a flat sheet form with desired characteristics has been successfully carried out. The membrane is found to be porous and asymmetric. The membrane is further developed by modifying the surface using LBL method. The polyelectrolytes used are PAA and CHI. The deposition of polyelectrolytes multilayers by dynamic LBL was carried out. The modification was to improve PES quality by making it has smooth, hydrophilic and negatively charge surface, suitable for xylitol purification. From the results and discussion, LBL was able to reduce its contact angle to 34.4° (more hydrophilic) and increase the surface electrostatic charge with zeta potential as high as -32.70 mV (6 bilayers) However, deposition of polyelectrolytes reduced the membrane's flux significantly. The rejection of xylitol for the modified membrane is better than the pure membrane. At 2 bilayers, the modified membrane is able to reject up to 84.95% of xylitol. At 4 bilayers, the membrane is able to separate xylitol and arabinose with separation factor as low as 0.71 compared to 1.185 by pure membrane.

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