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SEPARATION OF XYLOSE FROM GLUCOSE USING PILOT SCALE SPIRAL WOUND COMMERCIAL MEMBRANE

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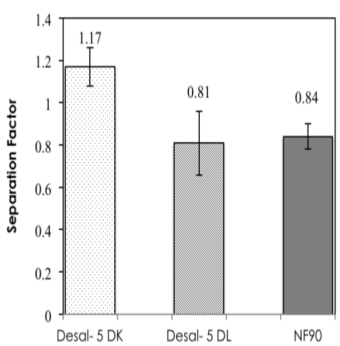
Graphical abstract



Abstract

Xylose is an intermediate product in xylitol production and glucose interferes in the process of separation. Thus the aim of this study is to investigate the performance of pilot scale commercial spiral wound NF membrane namely Desal-5 DK, Desal-5 DL and NF90 for separation of xylose from glucose. Separation of xylose and glucose model solutions was done in a pilot scale cross-flow system, using a commercial nanofiltration (NF) membrane with molecular weight cut off (MWCO) ranging from 150 to 1000 g/mol. The model solution consists of 1:1 ratio of xylose to glucose at 10 g/L each diluted in ultrapure water. The filtration was operated in total recycled mode at 10 bar. The sugar concentration was analyzed using high performance liquid chromatography (HPLC). From this study, the pure water permeability (PWPs) of the Desal-5 DK membrane was considerably higher at 6.78 ± 0.06 than PWPs of the Desal-5 DL and NF90 membranes at 1.28 ± 0.24 and 1.33 ± 0.05, respectively. The Desal-5 DK also gave the higher xylose separation factor at 1.17 as compare to Desal-5 DL (0.81) and NF90 membranes (0.84). This indicates that membrane Desal-5 DK was the most selective membrane to separate xylose form glucose. Overall, it can be concluded that the spiral wound nanofiltration membrane offers cost-effective and easy-maintenance, which has a potential in xylose-glucose separation.

Keywords: Nanofiltration, spiral wound, separation, xylose, glucose

Abstrak

Xilosa merupakan produk perantaraan dalam pengeluaran xilitol, dan glukosa telah mengganggu dalam proses pemisahannya. Oleh itu kajian ini telah dilakukan bertujuan untuk mengkaji prestasi skala perintis membran komersil nanoturasan gulungan lingkar pilin iaitu Desal-5 DK, Desal-5 DL dan NF90 untuk pemisahan xilosa dari glukosa. Pemisahan model larutan xilosa dan glukosa telah dilakukan dengan sistem skala perintis aliran silang, menggunakan membran nanoturasan (NF) komersil dengan potongan berat molekul (MWCO) di antara 150-1000 g/mol. Larutan model terdiri daripada 1:1 di antara xilosa dan glukosa pada 10 g/L yang dilarutkan ke dalam air tulen. Operasi penapisan dilakukan mod kitar semula pada 10 bar. Kepekatan gula telah dianalisa menggunakan kromatografi cecair prestasi tinggi

(HPLC). Daripada kajian ini, kebolehtelapan air tulen (PWPs) daripada Desal-5 DK membran adalah jauh lebih tinggi iaitu pada 6.78 ± 0.06 daripada PWPs oleh membran Desal-5 DL (1.28 ± 0.24) dan NF90 (1.33 ± 0.05). Membran Desal-5 DK juga memberi faktor pemisahan xilosa yang lebih tinggi iaitu pada 1.17 berbanding dengan Desal-5 DL (0.81) dan membran NF90 (0.84). Ini menunjukkan bahawa membran Desal-5 DK berkebolehan untuk memisahkan xilosa daripada glukosa. Secara keseluruhan, dapat disimpulkan bahawa membran nanoturasan gulungan lingkar pilin boleh dijadikan sebagai pemisah alternatif kos rendah serta mudah diselenggarakan yang mana ianya mempunyai potensi besar dalam pemisahan xilosa-glukosa.

Kata kunci: Nanopenurasan, gulungan lingkar pilin, pemisahan, xilosa, glukosa

1.0 INTRODUCTION

Separation of xylose and glucose is extremely challenging since they have similar charges, structures, and sizes. The molar mass of xylose and glucose are 150 g/mol and 180 g/mol respectively. The equivalent molar diameter is almost similar at 0.68 nm for xylose and 0.72 nm for glucose as the glucose is only slightly bigger than xylose molecules [1].

Xylose can be found inside hemicellulose in woody biomass. It is the raw material in production of xylitol, a sugar alcohol that used as sweetener by confectionary industry since it have equal sweetness with sucrose but has none of negative side effect of sugar [1]. Xylitol is hard to harvest abundantly since it has high percentage of glucose in woody materials. According to [2], high concentration of glucose in lignocellulosic biomass lead to saturated the transport system and cause inhibiting the transport of xylose. This phenomenon has implications for the conversion of the xylose in lignocellulose to xylitol as well as for the production of fuel ethanol in bioethanol industry.

In most cases, the method used to separate glucose and xylose is using liquid column chromatography due to its high selectivity. However, low product yield and complexity of purification procedures will push the cost of xylose purification to high level [4]. Hence, membrane system has been introduced for separation of xylose from glucose. There are several type of membrane elements such as flat-sheet ,tubular, hollow fiber and spiral wound. Spiral wound membrane elements currently have become most popular and economical form of packaging nanofiltration (NF) membrane. It is due to relatively large amount of membrane area per element, the efficient water flow and mass transfer in the element, and the low cost of the materials that used to construct the element [3,4]. Among membrane, NF is found to be extremely effective in the fractionation as well as concentration of solutes from complex process stream and advanced method for the concentration of organic and inorganic aqueous solution [5]. The advantages of NF is low operating pressure, high flux, high retention of multivalent anion salts and an organic molecular above 300, and low operation cost [6].

Many commercial NF membranes have an active layer formed from aromatic cross-linked polyamide and thin film composite membrane [7]. Example of commercial NF membranes are Desal-5 DK,Desal-5 DL, NF270, NF90 and many more. These membrane cover more 60% of food-industry applications,mainly for dairy and purification of saccharide [4]. From previus study by Goulas *et al.* [5], commerical NF-CA-50 membrane and Desal-5 DL membrane were applied to purify between model sugar solution and commercial galacto-olygosaccharide mixture. Meanwhile, Feng *et al*. [6] present the sugar separation performance testing using four different commercial spiral wound NF membrane which is NF-2,NF-3, NF-1812-50 and HBRO-1812-2.

The aim of this study is to evaluate the performance of pilot scale spiral wound nanofiltration membrane by using three different commercial nanofiltration (NF) spiral wound membrane, which are Desal-5 DK, Desal-5 DL and NF90 for separation of xylose from glucose. The performance of NF membrane was measured base on the separation factor of xylose.

2.0 METHODOLOGY

D-(+)-Xylose (>99%) and D-(+)-Glucose (>99.5%) were purchased from Sigma-Aldrich, Inc. The main characteristic of these components is shown in the Table 1. The xylose to glucose concentrations was kept at the ratio 1:1. The feed concentration was 10 g/L. The transmembrane pressure was tested at 10 bar.

Table1 The main characteristic of xylose and glucose

Chemicals	Xylose	Glucose
Abbreviation	Xyl	Glc
Molecular Formula	$C_5H_{10}O_5$	$C_6H_{10}O_6$
Molecular weight (g/mol)	150	180

The nanofiltration membranes used were Desal-5 DK, Desal-5 DL and NF90 with spiral wound elements. The properties of this membrane were summarized in Table 2 below.

Table 2 The commercial membrane used in this study

	NF90	Desal-5 DL	Desal-5 DK
Manufacturer	Dow/Filmtec	GE Osmonics	GE Osmonics
Support material	Polysulfone	Polysulfone	Polysulfone
Surface material	Polyamide	Polyamide	Polyamide
Average pore diameter (nm)	0.68 [7]	0.45 [8]	0.42[8],~1 [9]
Recommended pH range	3-10 [7], [10]	1-11[10], 2-11 [11]	2-11 [11], 3-9 [10]
Maximum Pressure (bar)	41[7]	2-30 [8]	4.6-26.6 [9]
MWCO (g/mol)	90,100 [7],200-400 [10]	150-300	150-300 [11],200 [10]

A pilot scale filtering unit in Figure 1 and Figure 2 with total reflux of both permeate and retentate was used. A circular cell made of stainless steel with radial flux held the NF membrane. The feed tank had a capacity of 20 L. The membranes were flushed with distilled water at atmospheric pressure. The pressure was increased to 10 bar and the membranes were treated at high pressure for 1 hour. Next, in order to calculate the membrane permeability, the pure water fluxes were measured at different operating pressures between 5 to 10 bar. Water permeability was always measured at room temperature.

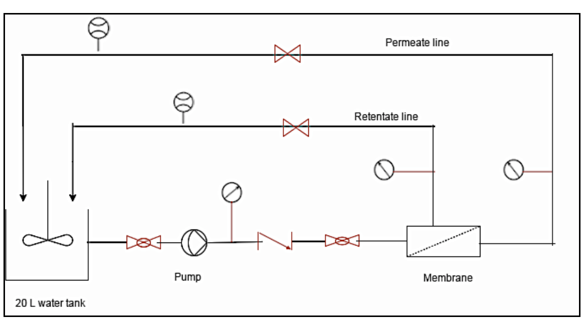


Figure 1 Schematic diagram of the experimental set-up for nanofiltration

Figure 2 The pilot scale cross flow spiral wound nanofiltration set-up

Single solution of xylose and glucose were prepared at 10g/L. The filtration was performed at 10 bar and at room temperature. The filtration was in total reflux for 1 hour before take the permeate. After each filtration, the membrane module was cleaned by recirculating distilled water for 1 hour through the membrane module and a pressure of 10 bar, in order to remove the reversible polarized layer. In the second step, the membrane module was rinsed with distilled water for 20 min, at room temperature and pressure. After filtration using the three different commercial membrane, the concentration of xylose and glucose were analyzed by High Performance Liquid Chromatography (HPLC) equipped with refractive index (RI) detector and Rezex RHM-Monosaccharide H+ (8%) column (300 mm x 7.8 mm, 8 μm). Water was used as the mobile phase at flow rate of 1.0 mL/min and the column temperature maintain at ambient temperature.

The observed retention (R_i^{ob}) was calculated from Eq. (1), where C_f and C_p are the concentration of feed sugar in the tank and permeate, respectively [14]. During the experiments, samples from the tank and permeate streams were taken and their concentrations were determined.

$$R_i^{ob} = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \qquad (1)$$

Alternatively, the separation factor, X_{xyl} can be used to evaluate the performance of the membrane. In Eq. (2), C_p (xyl) and C_p (glu) are the concentrations of xylose and glucose in the permeate, while C_f (xyl) and C_f (glu) are the concentrations of xylose and glucose in the feed. A separation factor greater than one agrees to xylose enrichment in the permeate as compared to the feed solution [15].

$$X_{xyl} = \frac{\left(C_p(xyl) / C_p(glu)\right)}{\left(C_f(xyl) / C_f(glu)\right)} = \frac{1-R_{xyl}}{1-R_{glu}} \qquad (2)$$

3.0 RESULTS AND DISCUSSION

The water flux stability for each membrane at 10 bar is shown in Figure 3 below. At high pressure, the permeate flux increases most probably because when the pressure increase, the water molecules has been pushed through the membrane and cause the membrane structure open. In comparison, Figure 3 shown Desal-5 DK has high water flux stability as compare to NF90 and Desal-5 DL.

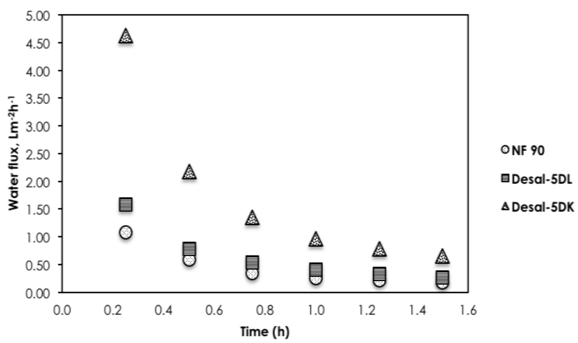


Figure 3 Water flux stability at 10 bar for membrane NF90, Desal-5 DL and Desal-5 DK

Pure water permeability (PWP) was used to measure the flux recovery of the membrane. The PWP was measured at 10, 8 and 4 bars and 35 ^{0}C with ultrapure water as shown in Table 3. The PWP of the Desal-5 DK membrane was considerably higher compared to Desal-5 DL and NF90. However, the PWP of a used membrane was lower than virgin membrane for Desal-5 DL. This might be because of inefficient water flushing and cleaning, as the duration of the cleaning was not sufficient enough to remove fouling on the membrane. According to [11] the decline of PWP might be due to the membranes swelling during measurements or membrane compaction. The other possible reason is internal blocking by model solution at the membrane structured that will cause decrease in water flux.

Table 3 Pure water permeability ($Lm^{-2}h^{-1}bar^{-1}$) of the virgin membranes and pure water permeability of the used membranes. Permeability was measured at 35 ^{0}C and at 10,6 and 4 bars with ultrapure water [values shown as minimum-maximum (average ± standard deviation)]

Membrane	PWP of a virgin membrane ($Lm^{-2}h^{-1}bar^{-1}$)	PWP of a used membrane ($Lm^{-2}h^{-1}bar^{-1}$)
NF 90	1.30-1.37 (1.33 ± 0.05)	0.99-1.92 (1.46 ± 0.65)
Desal-5 DL	1.11-1.44 (1.28 ± 0.24)	0.21-0.46 (0.34 ± 0.17)
Desal-5 DK	6.74-6.82 (6.78 ± 0.06)	6.16-6.68 (6.42 ± 0.37)

The retention of organic compounds by NF depends not only on their physical but also their chemical properties [12]. Table 1 shows that xylose has a molecular weight 150 g/mol, while glucose has 180 g/mol. Meanwhile, the molecular weight cut-off for the three membranes was in the range of 150-300 g/mol. In addition, Desal-5 DK, which was considered as the membrane with smallest pore size compare to Desal-5 DL and NF90 (see in Table 2). Based on Desal-5 DK properties, it suggests that glucose was more retained on the membrane surface compare to xylose, because of its tightest pore size. It can be seen in Figure 4 where glucose rejection was higher than xylose for membrane Desal-5 DK.

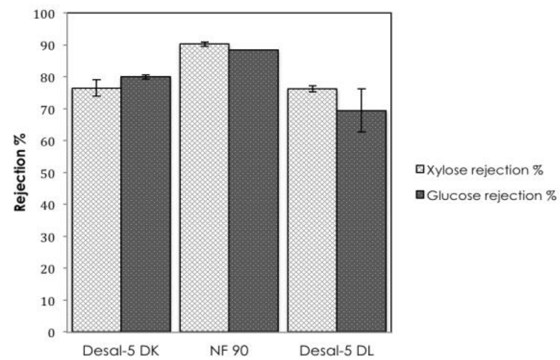


Figure 4 Rejection of xylose and glucose for NF 90, Desal-5 DK and Desal-5 DL membrane

The difference separation performances by the three membranes are shown in Figure 5. Desal-5 DK obtained higher separation factor of xylose which is average at 1.17 but the permeate flux was slightly lower than NF90. However, for NF90 and Desal-5 DL, their separation factor is less than 1, which is at 0.84 and 0.81 respectively. This indicates that these two membranes were not able to separates the xylose from glucose. This might be possible because the membrane structure was not open or concentration polarization occurred. The phenomena of concentration polarization was explained that additional fouling layer on the membrane surface or a higher solute concentration is present at the membrane surface due to the gradual increase of the rejected solute near the membrane surface hinders xylose passage through the membranes which may gave the highest permeate concentration of xylose [13]. This result was comparable to previous study by [1] for membrane Desal-5 DK, where xylose separation factor was obtained at 3.25 when increase the pressure up to 40 bar. This indicates that high xylose separation factor can be gained at high pressure. However, it may depend on the system capability as the system used in this present study can only be operated at 10 bar.

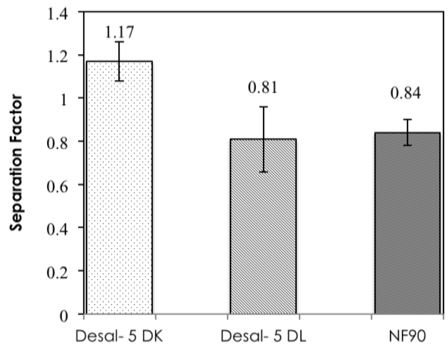


Figure 5 Xylose separation factor for NF 90, Desal-5 DK and Desal-5 DL membrane

4.0 CONCLUSION

The performances of pilot scale commercial spiral wound nanofiltration membrane were evaluated using three commercial membranes namely Desal-5 DK, Desal-5 DL and NF90 for separation xylose from glucose. From this study, it shows that Desal-5 DK membrane has a great potential for xylose separation in the food industry with separation factor up to 1.17 compared to Desal-5 DL and NF90. Both Desal-5 DL and NF90 were unable to separate the xylose from glucose. Thus, it can be concluded that the spiral wound nanofiltration Desal-5 DK membrane has a great potential in separation of xylose from glucose. More works on improving the separation factor is still in progress especially in working at pressure higher than 10 bar.

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