

## Evaluation of the operating parameters for the separation of xylitol from a mixed sugar solution by using a polyethersulfone nanofiltration membrane

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**Abstract**—Nanofiltration (NF) membranes may offer a good route for the recovery of xylitol due to the difference in the size of its particles compared to the other sugars. We evaluated the ability of an in-house polyethersulfone (PES) NF membrane to separate xylitol from a simulated broth solution containing xylose and arabinose. Initially, a Box-Behnken design was utilized to optimize the factors that were significantly involved in the recovery of xylitol, such as the concentration of the components, the composition of the solution, and the pressure. The results obtained from the analysis of the experimental response revealed that the fabricated PES membrane was able to retain 92% of the xylitol and remove 50% of the arabinose, with the purity of the xylitol being enhanced accordingly. The results of fouling showed a good membrane performance for long-term filtration. The concentration polarization was dominated by the membrane pores and the charge. It could be concluded that nanofiltration has a high potential to recover xylitol from its corresponding sugars.

Keywords: PES Membrane, Box-Behnken, Xylitol Solution, Fouling, Concentration Polarization

### INTRODUCTION

Xylitol is widely used in the food, pharmaceutical and dental fields as it has multiple properties, such as sweetness with low calorific content, is anticarcinogenic, has tooth hardening and remineralization properties, and also prevents otitis media [1]. Xylitol comes mainly from the fermentation of xylose by catalytic hydrogenation or through bioprocesses using microorganisms such as bacteria, fungi or yeast [2]. Xylose and arabinose are also common monosaccharides that are found in the fermentation process. The presence of these sugars in fermentation broths is believed to inhibit the production of high purity xylitol. The main challenge in purifying xylitol from these sugars is the similarity in the physical and chemical properties of xylitol and these sugars, especially at high concentrations, which limit the membrane selectivity [3]. It is known that when low concentrations of sugars are present in a broth, the membrane selectivity between xylitol and the sugars is increased, while at high concentrations of sugars, the membrane may lose this selectivity. However, at high sugar concentrations the membrane is still able to separate xylitol and its sugars from other impurities present in the broth such as yeast extract and proteins. The utilization of other separation techniques, such as adsorption,

could cause xylitol to leach out from the broth, while it would be difficult to perform the separation due to the presence of impurities such as proteins, that would reduce the purity of the xylitol and push up the separation cost further [4].

Previous studies have shown that separation using nanofiltration (NF) membranes is more effective at purifying xylitol in terms of saving energy and low maintenance cost [5,6]. Several variables affect the recovery of xylitol from its media, including (i) the composition of the solution, namely xylitol, xylose and arabinose; (ii) the concentration of xylitol and sugars in the solution, where the concentration varies based on the type of biomass used, the type of microorganism, the pH, and the temperature; (iii) the pressure, where a high pressure will force the xylitol to pass through the membrane, while a low pressure will push the sugars through the membrane, while enabling the xylitol to be retained; and (iv) the effect of temperature is always omitted since the process is usually conducted at room temperature [3]. On this basis, three variables were included in this study (composition, concentration and pressure), while the temperature effect was excluded since the experimental work was carried out at 25 °C. The screening process for the xylitol recovery through the use of single-factor experiments was conducted and reported in previous works [7,8] to confirm the range of compound concentrations. The concentration ranges were based on literature studies [9,10] to determine the composition and concentration of the solution. The interactions between the factors or the process parameters are a major concern to many engineers and

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managers, and should, therefore, be studied, analyzed and understood properly for troubleshooting and process optimization. It has been reported that the root cause of many process optimization problems in industries is sometimes due to the interaction between the different factors rather than the individual effect of each factor on gaining the output performance characteristic (or response) [11]. Thus, this work on the optimization of the operating parameters for the separation of xylitol from a mixed sugar solution was carried out to obtain high-purity xylitol from the mixed sugar solution by studying the effects of several parameters on the rejection of xylitol. Although the recovery of xylitol from sugars was more complex than expected, the process involved more than the controlling of the parameters, and included the nature of the xylitol, xylose and arabinose and their concentrations. Xylitol rejection can be maximized for a higher product purity, and both the xylose and arabinose rejections can be minimized by evaluating these separation factors using response surface methodology (RSM) [3,12]. RSM is a kind of mathematical and statistical technique for designing experiments, building models, evaluating the relative significance of several independent variables, and determining the optimum conditions for desirable responses. The most frequently used design matrices for RSM are the central composite design (CCD), Box-Behnken design and D-optimal design. An independent, quadratic Box-Behnken design has been used in various studies. The Box-Behnken design is a rotatable, second-order design which is based on incomplete factorial designs with three levels [13]. The other advantages of the Box-Behnken design compared to the CCD are that it mainly emphasizes on the middle points of a range of variables and it does not contain an embedded factorial design [14]. Box-Behnken design has been reportedly used for the determination of optimum conditions for xylose separation [3], chromatography methods [15], preparation of nanoparticles [16], and many other processes. The proper design of the statistical experiments showed that xylitol recovery can be investigated within a range of selected parameters at the same time. This design can generate the maximum information from limited experimental data in contrast to the one-variable-at-a-time (OVAT) approach, which keeps two variables constant, while the third one varies. The classical OVAT approach does not take into account the interaction effect of the process parameters [17]. Furthermore, the removal of insignificant terms from the samples makes them less predictive.

Membranes always encounter fouling after a long period of filtration. This may affect the quality of the product, and further cause the replacement of the membrane. Membrane fouling is commonly defined as an undesirable formation of deposits, resulting in the rapid decline of the permeation flux with time and causing poor membrane performance [18]. Fouling can be classified into reversible and irreversible, depending on the strength of the attachment of the foulants to the membrane surface. Reversible fouling is a type of temporary membrane fouling that can be easily removed with certain cleaning methods, such as water flushing or backwash [19]. Irreversible fouling is defined as permanent fouling, where the foulants still remain after the cleaning, and replacing the membrane is the only solution [20]. In general, the degree of fouling depends upon three main factors: operating parameters (pressure and stirring speed), feed characteristics (solute size and charge) and mem-

brane characteristics (porosity, pore size and surface properties) [21]. Membrane fouling may be due to (i) adsorption inside the membrane, (ii) blocking of the membrane pores, (iii) high concentration of foulants near the membrane (concentration polarization), (iv) deposition of foulants on the membrane surface forming a cake layer, and (v) compression of the cake layer.

The molar mass of a xylitol molecule is 152 g/mol, 2 g/mol higher than that of xylose and arabinose molecules of equal molar mass (150 g/mol). Xylitol also has a higher hydrophilicity ( $\log P$ ), followed by xylose and arabinose. When considering the properties of xylitol in comparison to xylose and arabinose, it is important to understand the interaction behavior of these compounds during membrane filtration.

This particular study was targeted at evaluating the factors affecting the separation of xylitol from xylose and arabinose using an in-house polyethersulfone (PES) NF membrane. PES was used because of its high mechanical strength, thermal stability, and chemical resistance [22]. First, the NF membrane was prepared via the phase inversion method, followed by its characterization using the contact angle for the hydrophilicity, and the porosity and AFM for the surface topography. Next, the filtration pressure and concentrations of xylitol, xylose and arabinose were varied in order to evaluate the separation performance of the membrane. Box-Behnken experimental design was selected to evaluate the effects of the process factors on the xylitol, xylose and arabinose rejections. The performance of the membrane was validated by running the filtration using the optimum results obtained for the concentration and pressure of xylitol, xylose, and arabinose. A membrane fouling test was used to evaluate the performance of the fabricated membrane for long-term filtration.

## EXPERIMENTAL METHOD

### 1. Materials

Polyethersulfone (PES) ( $M_w=37,000$ ), which was the only polymer used in the membrane synthesis, was supplied by Goodfellow USA. The PES granules were dried at 60 °C for 72 hours to remove the trapped moisture prior to use. *N*-Methyl-2-Pyrrolidone (NMP), purchased from Fluka, Germany and having an analytical purity of 99.7%, was used as the dope solvent. The xylitol, xylose and arabinose sugars were of commercial analytical grade and used without further purification in the preparation of the fermentation broth model.

### 2. Membrane Fabrication Procedure

The PES membrane was prepared by dissolving 18% of PES in 82% of NMP with stirring at 450 rpm for 8 hours at 60 °C. The dope solution was left for 24 hours for degassing purposes prior to casting. A suitable volume of the solution was poured onto a glass sheet, and was cast using a knife to a thickness of 200  $\mu\text{m}$ , followed by an immersion precipitation process in a water coagulation bath at 25 °C for 2 hours for solvent exchange. The deionized (DI) water in the coagulation bath was changed several times to ensure complete solvent exchange.

### 3. Membrane Characterization

The fabricated membrane was subjected to a series of analyses in order to investigate the performance and morphology of the mem-

brane. The water flux of the membrane was measured using a dead-end filtration unit (Sterlitech HP4750, Sterlitech Corporation, USA). The membrane was clamped inside the filtration unit and pressurized at 22 bars until a steady flow was accomplished. The water flux was calculated using the equation below:

$$J_w = \frac{V}{A \cdot t} \quad (1)$$

where  $J_w$  is the water flux ( $L/m^2 \cdot h$ ),  $V$  is the permeate volume (L),  $A$  is the effective area of the membrane ( $0.00146 \text{ m}^2$ ), and  $t$  is the operating time (h).

The membrane porosity ( $\varepsilon$ ) was calculated using Eq. (2). This value was essential for measuring the mean pore radius ( $r_m$ ) of the membrane. The membrane was first dried in a vacuum oven at  $60^\circ\text{C}$  for 6 hours and weighed. The porosity of the pure membrane was evaluated according to its capacity for water absorption, and was measured using a gravimetric method:

$$\varepsilon = \frac{W_1 - W_2}{(\rho_w)(A)(l)} \quad (2)$$

where  $W_1$  and  $W_2$  are the weights of the wet and dry membranes, respectively;  $\rho_w$  is the water density ( $998 \text{ kg/m}^3$ );  $A$  is the effective area of the membrane ( $0.00146 \text{ m}^2$ ); and  $l$  is the membrane thickness ( $0.0002 \text{ m}$ ).

The mean pore radius ( $r_m$ ) of the membrane was determined on the basis of the pure water flux and from the membrane poros-

ity ( $\varepsilon$ ) values. The mean pore radius ( $r_m$ ) was measured according to the Guerout-Elford-Ferry Eq. [23], as given in Eq. (3):

$$r_m = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta l Q}{\varepsilon \cdot A \cdot \Delta P}} \quad (3)$$

where  $\eta$  is the water viscosity ( $8.9 \times 10^{-4} \text{ Pa s}$ );  $l$  is the membrane thickness ( $0.0002 \text{ m}$ ); and  $\Delta P$  is the operating pressure ( $0.4 \text{ MPa}$ ).

The surface morphology of the fabricated membrane was investigated by:

(i) Atomic force microscopy (AFM). A digital instrument (Scanning Probe Microscope, NTEGRA Prima, NT-MDT, Russia) was used to obtain the surface roughness of the membrane by surface image analysis. The measurements were conducted in the semi-contact mode using an NSG01 (golden silicon) probe.

(ii) The contact angle was used to measure the hydrophilicity of the membrane. The membrane was dried for 48 hours prior to the measurements. A Rame-Hart Model 200 standard contact angle goniometer (Germany) was used with DROPimage Standard Software with an accuracy of  $60.10^\circ$ . The medium that was used to measure the contact angle was deionised water and air at ambient temperature.

#### 4. Nanofiltration Experimental Setup

The NF process was conducted using the filtration unit shown in Fig. 1. The dead-end filtration unit was filled with a xylitol mixed solution several times, with the xylitol concentrations varying from 19 to 88 g/L. The filtration process was started after a steady state

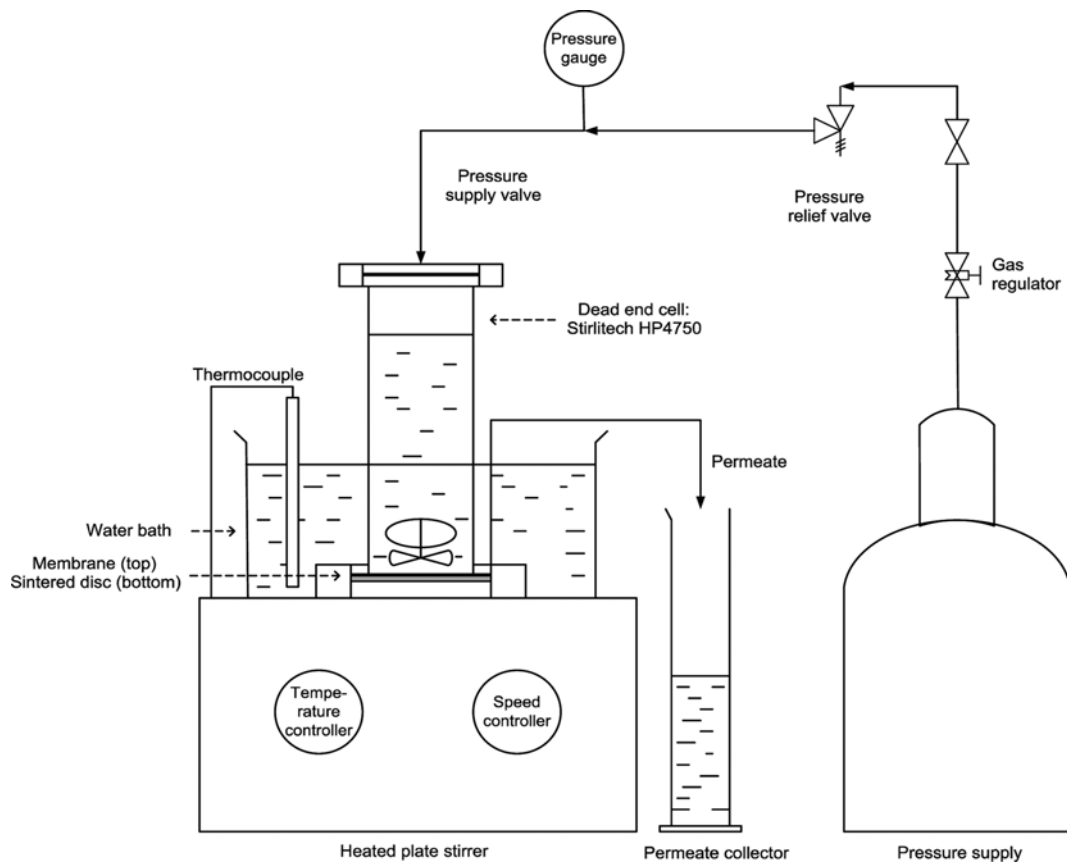


Fig. 1. Schematic diagram of the dead-end filtration unit [24].

**Table 1. Factors with their actual values for xylitol mixed solution filtration**

Factors	Symbols	Levels		
		-1	0	+1
Xylitol concentration (g/L)	X <sub>1</sub>	19	53.5	88
Xylose concentration (g/L)	X <sub>2</sub>	1	3	5
Arabinose concentration (g/L)	X <sub>3</sub>	2	13.5	25
Pressure (bar)	X <sub>4</sub>	4	7	10

flow had been obtained at different pressures ranging from 4 to 10 bars. 4 ml of the permeate was collected, and the xylitol and sugar concentrations were quantified using HPLC (Ultimate 3000, Thermo Scientific, USA) under the following conditions: the column was RPM (Rezex, dimensions: 300×7.8 mm, USA); the mobile phase was deionised (DI) water; the flow rate was 0.6 ml/min, using a refractive index (RI) detector (Refractomax 520, ERC, USA); and at a temperature of 60 °C.

### 5. Design of Experiment

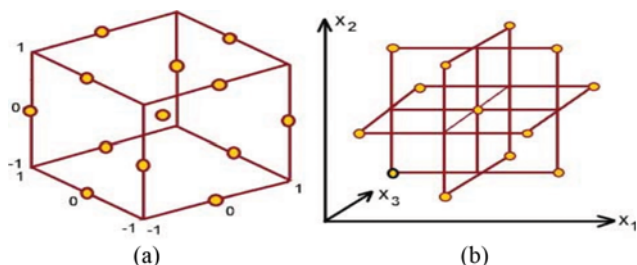
The experiments were designed by running the Design-Expert version 6.0.10 (stat-Ease, Inc.). Box-Behnken design was established to perform 29 runs with four input factors. The following factors were chosen due to their major effects on the filtration process: (i) xylitol concentration, (ii) xylose concentration, (iii) arabinose concentration, and (iv) operating pressure. These input factors, with their coded and actual values, are presented in Table 1. The concentration of each sugar was varied based on the values given in the literature: xylitol from 19 to 88 g/L, xylose from 1 to 5 g/L, and arabinose from 2 to 25 g/L. The operating pressure was also varied from 4 to 10 bars. The operating pressure range in the dead-end filtration unit was considered as limited and quite low so as to avoid a reverse effect on the rejection of xylitol if high pressure was applied [9]. The experiments were run randomly to determine the responses to xylitol rejection, xylose rejection and arabinose rejection. The rejection percentage was calculated using Eq. (4):

$$R = \frac{C_f - C_p}{C_f} \times 100 \quad (4)$$

where R is the rejection percentage (%), and C<sub>f</sub> and C<sub>p</sub> are the feed and permeate concentrations, respectively.

To determine the C<sub>p</sub> values of xylitol, xylose and arabinose, the same HPLC analytical method described in Section 4 above was used.

Box-Behnken design is considered as a second-order technique



**Fig. 2. Box-Behnken design representation (a) derived from a cube; (b) interlocking 22 factorial designs [27].**

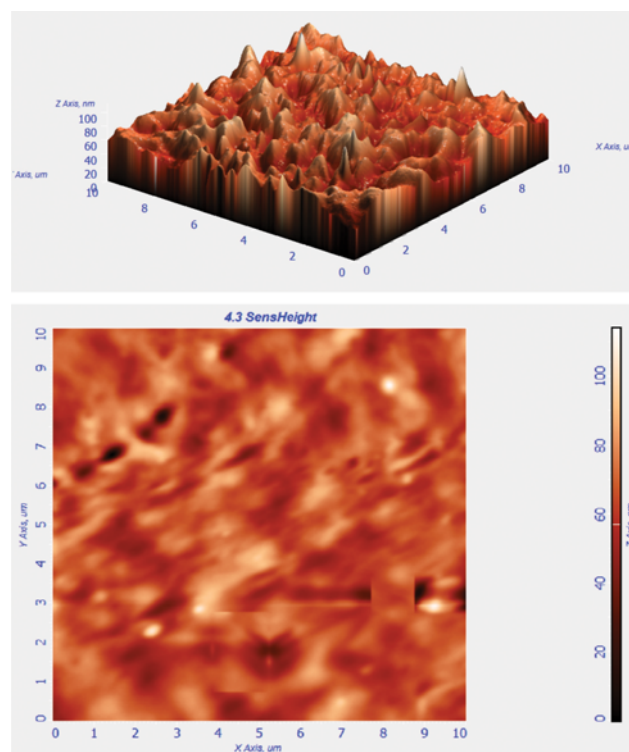
based on a three-level factorial design for three factors or more with selected points from a system arrangement [25]. This design has two different forms in which to present the three-factor graphs: (i) a cube, which contains the central and the middle points of the edges, as in Fig. 2(a), and (ii) three interlocking 22 factorial designs with a central point, as shown in Fig. 2(b). The number of experiments required (N) was calculated using the equation,  $N=2k(k-1)+C$ , where k is the number of factors, and C is the centre point. The main advantage of this design is that it can reduce the number of runs, and can be used for a large number of factors in one process. To improve the performance of the Box-Behnken design, the three levels of the factors were adjusted to -1 (lower), 0 (medial) and +1 (higher) [26]. The advantage of applying the Box-Behnken design compared to other surface designs is its high efficiency, where the efficiency of an experimental design is defined as the number of coefficients in the estimated model divided by the number of experiments [26]. However, the Box-Behnken design does not contain combinations at the highest or lowest levels of all the selected process factors [23].

To optimize the rejection of xylitol and other sugars, the following quadratic polynomial equation was followed:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=i+1}^k \beta_{ij} X_i X_j + \varepsilon \quad (5)$$

**Table 2. Properties of PES 18% membrane**

Membrane type	Contact angle (°)	Porosity (%)	Pore size (nm)	Water flux (L/m <sup>2</sup> ·h)
PES 18%	80±4.95	48	7	13



**Fig. 3. AFM results of pure PES membrane.**

where Y is the predicted response (target of study),  $\beta_i$  is the coefficient of the linear term,  $\beta_{ii}$  is the coefficient of the quadratic term,  $\beta_{ij}$  is the coefficient of the interaction factor,  $X_i$  and  $X_j$  indicate the independent variables, and  $\varepsilon$  is the random error. The mathematical relationship between the four factors:  $X_1$ ,  $X_2$ ,  $X_3$  and  $X_4$ , with their coefficients is represented by the second-order equation below:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{44} X_4^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{14} X_1 X_4 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4 \quad (6)$$

In this work, 29 experiments were run for the filtration of the xylitol mixture solution using a Box-Behnken design with four factors and three responses, as summarized in Table 2. The analysis of variance (ANOVA) was applied to derive the interaction between these factors to improve the performance of the membrane in order to be selective for xylitol, and to allow the sugars to permeate through the membrane.

## RESULTS AND DISCUSSION

### 1. Membrane Characterization

The PES membrane was initially subjected to several analyses

and characterizations following the synthesis. The surface morphology of the membrane was analyzed using AFM to determine the contact angle, porosity and pore size. The results of the analyses showed that the NF membrane had promising properties. A pure water flux (PWF) was carried out to estimate the effective pore radius ( $r_m$ ) using a mathematical model based on Eq. (3). The prediction of  $r_m$  for the fabricated membrane was to make sure the developed membrane was within the nanofiltration range. The pore size of the membrane was 7 nm, which confirmed the NF level of the membrane, where the membrane porosity was only 48%, while the water flux value showed a good filtration performance of 13 L/m<sup>2</sup>·h at 4 bars. The morphological results showed that the PES membrane lacked hydrophilicity [7] because the contact angle was only at 80±4.95° (results presented in Table 2).

Fig. 3 shows the 3-D and 2-D AFM images of the PES membrane. AFM analyses were performed to investigate the surface morphology at a nanoscopic scale and to quantify the surface roughness of the membrane, which was obtained from the root mean square roughness (RMS). Referring to a previous work [8], the RMS value of the PES 18% membrane was 18.57 nm. The brightest areas in the figures represent the highest points of the membrane surface,

**Table 3. All the experimental runs with their interactions of selected factors and the actual and predicted values of the responses**

Std.	Run	$X_1$	$X_2$	$X_3$	$X_4$	$Y_{1actual}$ %	$Y_{1predicted}$ %	$Y_{2actual}$ %	$Y_{2predicted}$ %	$Y_{3actual}$ %	$Y_{3predicted}$ %
1	18	19	1	13.5	7	50	51.08	48	48.25	46	46.46
2	16	88	1	13.5	7	92	88.42	88	85.92	91	90.63
3	14	19	5	13.5	7	39	43.08	42	43.58	44	44.13
4	4	88	5	13.5	7	95	94.42	94	93.25	92	91.29
5	6	53.5	3	2	4	90	90.42	86	86.92	70	71.63
6	7	53.5	3	25	4	94	93.58	93	92.08	92	93.46
7	27	53.5	3	2	10	91	91.92	89	89.42	80	78.29
8	2	53.5	3	25	10	93	93.08	92	90.58	91	89.13
9	20	19	3	13.5	4	51	49.46	50	49.54	47	45.71
10	3	88	3	13.5	4	93	93.79	92	92.71	91	89.88
11	23	19	3	13.5	10	52	49.96	50	49.54	44	45.38
12	26	88	3	13.5	10	94	94.29	93	93.71	91	92.54
13	10	53.5	1	2	7	88	89.79	86	87.38	76	76.38
14	29	53.5	5	2	7	86	87.29	80	81.71	71	73.04
15	21	53.5	1	25	7	93	90.46	85	83.54	92	90.21
16	12	53.5	5	25	7	94	90.96	93	91.88	92	91.88
17	19	19	3	2	7	55	51.63	52	49.21	33	31.5
18	8	88	3	2	7	85	83.96	83	81.38	75	74.17
19	15	19	3	25	7	40	41.79	39	40.88	44	44.83
20	11	88	3	25	7	94	98.13	93	96.04	92	93.5
21	5	53.5	1	13.5	4	92	93.63	90	90.71	90	90.33
22	1	53.5	5	13.5	4	92	91.13	91	90.04	90	89
23	17	53.5	1	13.5	10	91	92.63	88	89.21	90	91
24	28	53.5	5	13.5	10	94	93.13	93	92.54	91	90.67
25	9	53.5	3	13.5	7	94	93.8	93	92.6	91	91.2
26	22	53.5	3	13.5	7	95	93.8	93	92.6	92	91.2
27	13	53.5	3	13.5	7	93	93.8	92	92.6	90	91.2
28	24	53.5	3	13.5	7	95	93.8	94	92.6	92	91.2
29	25	53.5	3	13.5	7	92	93.8	91	92.6	91	91.2

while the dark regions illustrate the valleys or pores in the membrane. It can be seen that the PES membrane showed a flat surface.

## 2. Model Fitting and ANOVA Analysis

To achieve a high membrane performance in the retention of xylitol and the permeation of the sugars, the main factors affecting the filtration process were investigated. Several experimental runs were carried out to determine the effect of each factor on the performance of the membrane. 29 experiments were triplicated to verify the optimum conditions for the high rejection of xylitol and the permeation of xylose and arabinose, and also to validate the adequacy of the final prediction. The validity of the predicted results was ensured by comparing them with the actual runs; this evaluation of the model fit is mandatory. A summary of the final model prediction of the filtration of the xylitol solution with the affecting factors based on the Box-Behnken design is shown in the following equations:

$$Y_1 = 22.03001 + 2.32235 X_1 - 1.42899 X_2 - 0.24679 X_3 - 1.22379 X_4 - 0.019030 X_1^2 - 0.47500 X_2^2 - 0.017202 X_3^2 + 0.080556 X_4^2 + 0.050725 X_1 X_2 + 0.015123 X_1 X_3 + 2.14536E-017 X_1 X_4 + 0.032609 X_2 X_3 + 0.12500 X_2 X_4 - 0.014493 X_3 X_4 \quad (7)$$

$$Y_2 = 20.67722 + 2.27209 X_1 - 1.01377 X_2 - 0.14102 X_3 - 1.43792 X_4 - 0.01853 X_1^2 - 0.7 X_2^2 - 0.02779 X_3^2 + 0.091667 X_4^2 + 0.043478 X_1 X_2 + 0.014493 X_1 X_3 + 2.42E-03 X_1 X_4 + 0.15217 X_2 X_3 + 0.16667 X_2 X_4 - 0.02899 X_3 X_4 \quad (8)$$

$$Y_3 = -19.7467 + 2.5479 X_1 - 0.91522 X_2 + 2.47993 X_3 + 1.30229 X_4 \quad (9)$$

$$-0.01888 X_1^2 - 0.15 X_2^2 - 0.05841 X_3^2 - 0.03889 X_4^2 + 0.01087 X_1 X_2 + 3.78E-03 X_1 X_3 + 7.25E-03 X_1 X_4 + 0.054348 X_2 X_3 + 0.041667 X_2 X_4 - 0.07971 X_3 X_4 \quad (9)$$

where  $Y_1$ ,  $Y_2$  and  $Y_3$  are the rejections of xylitol, xylose and arabinose, respectively, and  $X_1$ ,  $X_2$ , and  $X_3$  are the concentrations of xylitol, xylose and arabinose, respectively, while the pressure is represented by  $X_4$ .

The results of the responses, as presented in Table 3, show that there was good agreement between the actual values from the experiments and the predicted values from the obtained model. These results may be attributed to the high accuracy of the experimental results for all the responses involved as a result of using a well-fabricated PES membrane. The ANOVA results of the specific responses of the rejection of xylitol, xylose and arabinose are given in Tables 4 to 6. It was found that the second-order regression model for the rejections of xylitol, xylose and arabinose was at a significantly high confidence level (95%). All the predicted coefficients estimated by the ANOVA analysis were at a high significant p-value ( $p < 0.05$ ). Meanwhile, the coefficients of  $R^2$  for all the responses were higher than 0.97, which indicated a good validity of the predicted rejections. Considerably high values of the adjusted  $R^2_{adj}$  coefficients of 0.9782, 0.9888 and 0.9930 for xylitol, xylose and arabinose, respectively, were obtained. The  $R^2$  and  $R^2_{adj}$  coefficients were close to 1, indicating that the final predicted results were in good agreement with the results of the experiments performed in this study. The F-values of the xylitol, xylose and arabi-

**Table 4. Summary of ANOVA results of xylitol rejection using a Box-Behnken design**

Source	Sum of squares	DF	Mean square	F value	p>F
Model	9633.62	14	688.12	90.56	<0.0001*
A	5896.33	1	5896.33	775.95	<0.0001*
B	3	1	3	0.39	0.5399
C	14.08	1	14.08	1.85	0.1949
D	0.75	1	0.75	0.099	0.758
A <sup>2</sup>	3327.71	1	3327.71	437.93	<0.0001*
B <sup>2</sup>	23.42	1	23.42	3.08	0.101
C <sup>2</sup>	33.57	1	33.57	4.42	0.0541
D <sup>2</sup>	3.41	1	3.41	0.45	0.5139
AB	49	1	49	6.45	0.0236*
AC	144	1	144	18.95	0.0007*
AD	0	1	0	0	1
BC	2.25	1	2.25	0.3	0.5949
BD	2.25	1	2.25	0.3	0.5949
CD	1	1	1	0.13	0.7222
Residual	106.38	14	7.6		
Lack-of-fit	99.58	10	9.96	5.86	0.0516**
Pure error	6.8	4	1.7		
Total	9740	28			

\*Significant at <0.05% level; \*\*Not significant;  $R^2=0.9891$ ; Adj R-Squared=0.9782; Standard deviation=2.76; Mean=83.00; C.V=3.32; Adequate precision=28.415

**Table 5. Summary of ANOVA results of xylose rejection using a Box-Behnken design**

Source	Sum of squares	DF	Mean square	F value	p>F
Model	9307	14	664.79	177.45	<0.0001*
A	5720.33	1	5720.33	1526.88	<0.0001*
B	5.33	1	5.33	1.42	0.2526
C	30.08	1	30.08	8.03	0.0133*
D	0.75	1	0.75	0.2	0.6614
A <sup>2</sup>	3153.75	1	3153.75	841.8	<0.0001*
B <sup>2</sup>	50.85	1	50.85	13.57	0.0025*
C <sup>2</sup>	87.6	1	87.6	23.38	0.0003*
D <sup>2</sup>	4.41	1	4.41	1.18	0.296
AB	36	1	36	9.61	0.0078*
AC	132.25	1	132.25	35.3	<0.0001*
AD	0.25	1	0.25	0.067	0.7999
BC	49	1	49	13.08	0.0028*
BD	4	1	4	1.07	0.319
CD	4	1	4	1.07	0.319
Residual	52.45	14	3.75		
Lack-of-Fit	47.25	10	4.72	3.63	0.1126**
Pure error	5.2	4	1.3		
Total	9359.45	28			

\*Significant at <0.05% level; \*\*Not significant;  $R^2=0.9944$ ; Adj R-Squared=0.9888; Standard deviation=1.94; Mean=81.14; C.V=2.39; Adequate precision=39.630

**Table 6. Summary of ANOVA results of arabinose rejection using a Box-Behnken design**

Source	Sum of squares	DF	Mean square	F value	p>F
Model	10698.49	14	764.18	283.65	<0.0001*
A	6256.33	1	6256.33	2322.28	<0.0001*
B	2.08	1	2.08	0.77	0.394
C	800.33	1	800.33	297.07	<0.0001*
D	4.08	1	4.08	1.52	0.2386
A <sup>2</sup>	3276.49	1	3276.49	1216.2	<0.0001*
B <sup>2</sup>	2.34	1	2.34	0.87	0.3676
C <sup>2</sup>	387.09	1	387.09	143.68	<0.0001*
D <sup>2</sup>	0.79	1	0.79	0.29	0.5956
AB	2.25	1	2.25	0.84	0.3763
AC	9	1	9	3.34	0.089
AD	2.25	1	2.25	0.84	0.3763
BC	6.25	1	6.25	2.32	0.15
BD	0.25	1	0.25	0.093	0.7651
CD	30.25	1	30.25	11.23	0.0048*
Residual	37.72	14	2.69		
Lack-of-Fit	34.92	10	3.49	4.99	0.0676*
Pure error	2.8	4	0.7		
Total	10736.21	28			

\*Significant at <0.05% level; \*\* Not significant; R<sup>2</sup>=0.9965; Adj R-Squared=0.9930; Standard deviation=1.64; Mean=78.31; C.V=2.10; Adequate precision=52.522

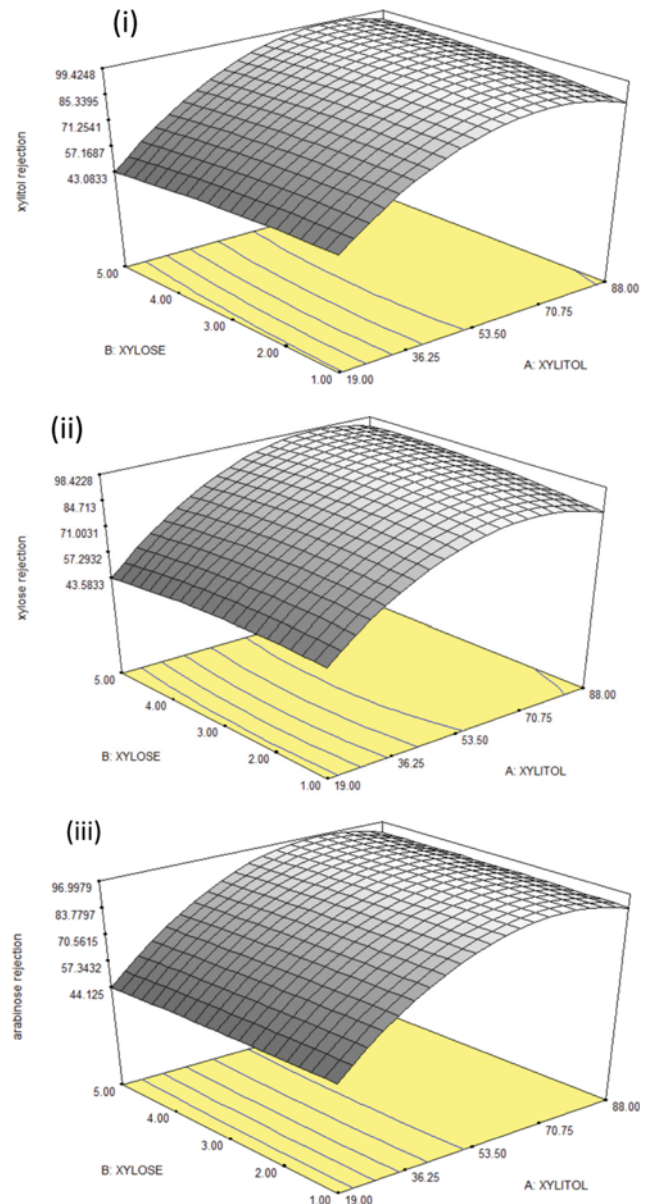
nose filtration processes were 90.56, 177.45, and 283.65, respectively, which showed the significance of the developed model. The term 'Adequate Precision' was used to evaluate the prediction range of the rejections of xylitol, xylose and arabinose, which were 28.415, 39.630, and 52.522, respectively. However, these coefficient values, which measured the contrast in the predicted response relative to its associated error, were found to be greater than 4.0. These values are usually required to support the statistical prediction fitness [26].

### 3. Effect of Total Solution Concentrations

It has been reported that the sugar concentration has both positive and negative effects on the separation performance of nanofiltration. On the positive side, the larger molecules that are retained form a kind of second or dynamic membrane, thereby inducing high selectivity of small molecules [28]. On the negative side, the higher total sugar concentration may increase the concentration of the larger molecules at the membrane surface, creating resistance against the passage of the smaller molecules. This is also known as concentration polarization [29]. The effects of these factors with their interactions were explained individually and statistically with more evidence to show the effects that were present within the variations in the factor ranges.

#### 3-1. Effects of Interaction between Xylitol and Xylose Concentrations

Fig. 4 shows the interaction between the concentrations of xylitol and xylose, and their effect on the response to the rejection of xylitol, xylose and arabinose. The xylitol concentration varied from



**Fig. 4. Effect of interaction between the xylitol and xylose concentrations on (i) xylitol rejection, (ii) xylose rejection, and (iii) arabinose rejection as 3D response surfaces.**

19.0 to 88.0 g/L, and the xylose concentration from 1.0 to 5.0 g/L. All the rejections increased as the concentration of xylitol and xylose was increased to 70.75 and 3.0 g/L, respectively. As the concentration of xylitol and xylose exceeded 70.75 and 3.0 g/L, respectively, the rejections fell gradually. The presence of low amounts of xylitol and xylose in the real xylitol fermentation broth would benefit the membrane filtration by minimizing the concentration polarization. Concentration polarization is a major factor that may lead to membrane fouling, and further reduce membrane selectivity. Meanwhile, at higher solution concentration, a possible impact of membrane compression and high concentrations at the membrane surface could be reduced rejection. Therefore, low concentrations of sugars in the xylitol mixture would minimize or eliminate con-

centration polarization and control membrane fouling [24,30]. The separation of xylitol from xylose is based on their difference in hydrophilicity,  $\log P$ . However, knowledge of the molecular interactions near the pore inlet and inside the pores with concentrated solutions is limited. A more sophisticated explanation would be to consider the equilibrium between the hydrophilicity and solubility of the sugars.

### 3-2. Effects of Interaction between Xylitol and Arabinose Concentrations

The optimum concentrations for xylitol and arabinose were investigated, and the results are presented in Fig. 5. The best rejections were observed at a concentration of 70.75 g/L and 19 g/L of xylitol and arabinose, respectively. However, higher concentrations of xylitol and arabinose in the mixture had a negative influence on their rejection, especially when the concentration of xylitol and

arabinose exceeded 70.75 g/L and 19 g/L, respectively. In general, the presence of high concentrations of sugars and xylitol resulted in a clear drop in the membrane selectivity due to the concentration polarization effect. The difference in the solubility of xylitol and arabinose also had a clear impact on the membrane selectivity at high concentrations of arabinose. The higher the concentration of arabinose molecules, the greater the possibility of the formation of a cake layer close to the surface of the membrane, thereby resulting in the retention of xylitol molecules, enhanced rejection of xylitol, but reduced membrane selectivity between xylitol and arabinose. Moreover, the effects of the concentration of the sugars and the filtration pressure on the retention of the sugars were vague. However, the hydrophilicity and solubility played a big role in the retention, as the studied sugars presumably had the same proper-

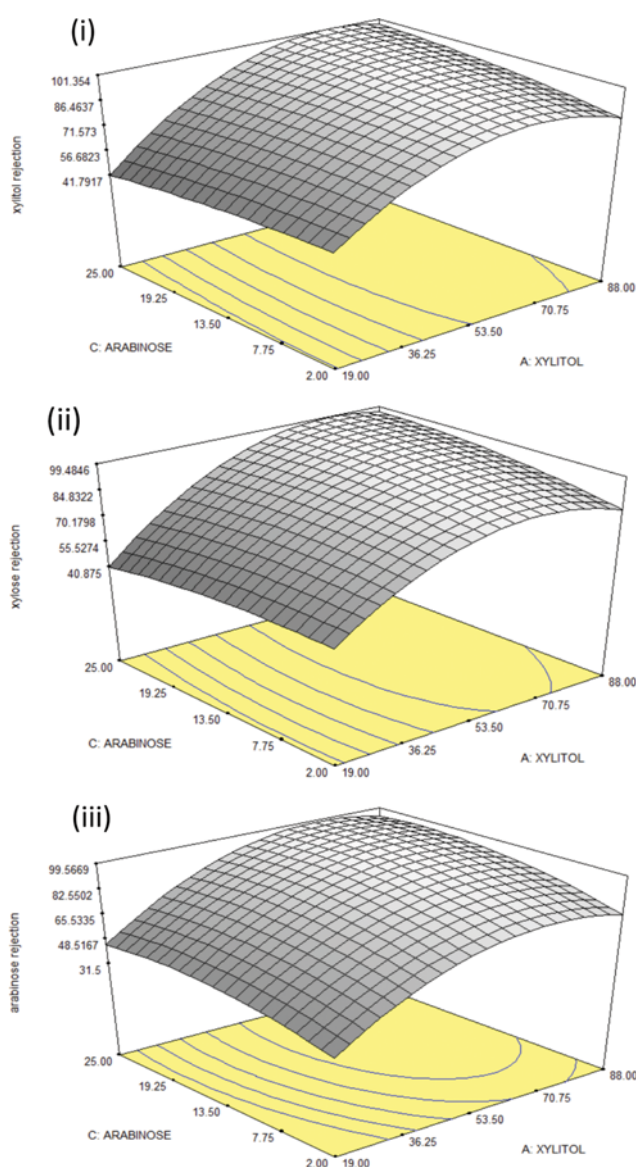


Fig. 5. Effect of interaction between the xylitol and arabinose concentrations on (i) xylitol rejection, (ii) xylose rejection, and (iii) arabinose rejection as 3D response surfaces.

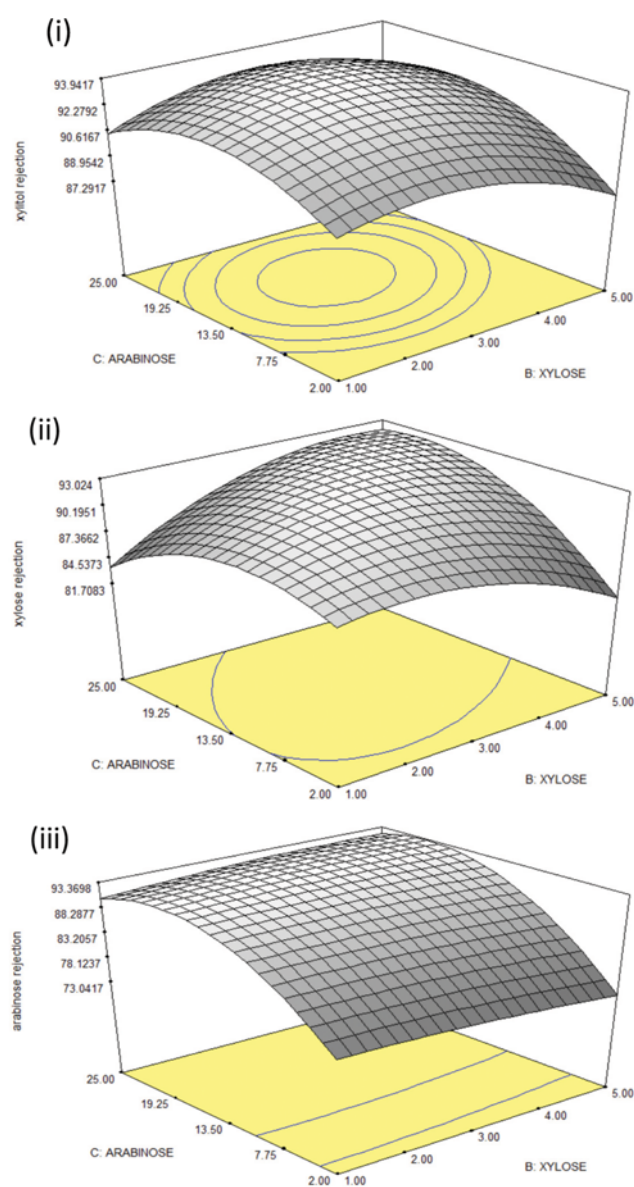


Fig. 6. Effect of the interaction between the xylose and arabinose concentrations on the rejection of (i) xylitol, (ii) xylose and (iii) arabinose as 3D response surfaces.

ties in an aqueous solution.

### 3-3. Effects of Interaction between Xylose and Arabinose Concentrations

The xylose and arabinose concentrations had a remarkably clear effect on the solution rejection. Fig. 6 shows that the best solution rejection was obtained when the xylose and arabinose concentrations were 3.0 g/L and 13.50 g/L, respectively. At low concentrations of xylose and arabinose, the membrane was able to reject both sugars due to the difference between the hydrophilicity of the sugars. However, at high concentrations, the existence of concentration polarization gave rise to membrane selectivity between both sugars. There was an appreciable interaction between the total concentration of the sugars and the xylitol : xylose : arabinose ratio, as observed by the significant changes to the performance of the membrane. An increase in the concentrations of xylose and arabinose in the solution caused a drop in the membrane selectivity. These results contradicted the findings of a study by Sjöman et al. [31], where it was concluded that a higher xylose concentration in the feed results in higher total permeate flux and xylose rejection than when the glucose concentration is high. A high glucose concentration in the feed does enhance the xylose permeation and reduce the total permeate flux. In this study, a drop in the membrane selectivity was observed at a high concentration of arabinose in the feed. This was mostly caused by the occurrence of concentration polarization, which hindered the permeation of xylitol and xylose. The increase in the concentration polarization may have been due to the difference in solubility between the xylitol, xylose and arabinose, which caused the build-up of both xylose and arabinose on the surface of the membrane, thereby creating a kind of second membrane that blocked the xylitol molecules from passing through.

## 4. Effect of Operating Pressure

### 4-1. Effects of Interaction between Xylitol Concentration and Pressure

The operating pressure may also affect the filtration process since the molecular weights of the components to be separated are very near to one another. A low operating pressure is believed to consume lower energy, thus enabling smaller molecules of xylose and arabinose to permeate through the membrane, while the xylitol is retained. Affleck [9] proposed a different range of membranes from UF to RO to recover xylitol from biomass fermentation broth. The effect of operating the filtration unit at high pressure was apparent, where xylitol was forced to permeate through the membranes together with other sugars. In this work, a low operating pressure was preferred for the removal of xylitol from the sugar solution. The effect of pressure ranging from 4 to 10 bars on various concentrations of xylitol from 19.0 to 88.0 g/L was studied. As illustrated in Fig. 7, the rejection of the components of the solution was found to increase as the xylitol concentration increased up to 70.75 g/L, before it started to decrease due to concentration polarization.

Based on the results presented in Table 3, the membrane selectivity may drop at any operating pressure when higher sugar (xylose or arabinose) concentrations are used, while at a lower sugar concentration, even at a high xylitol concentration, the membrane selectivity is enhanced. Therefore, a low operating pressure and low

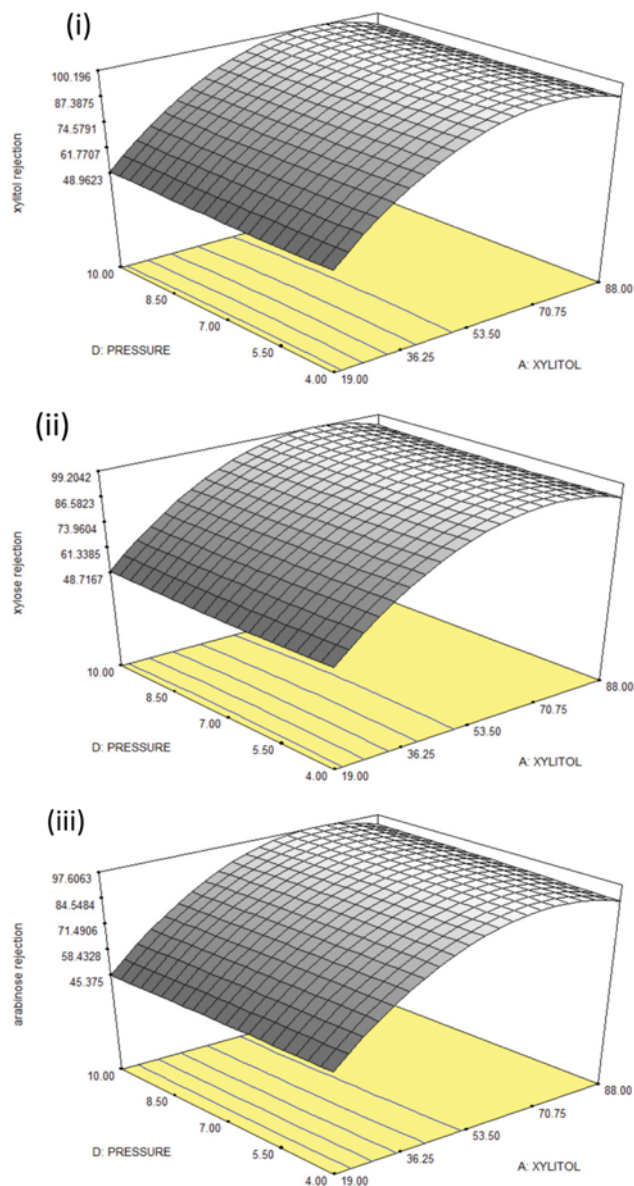


Fig. 7. Effect of interaction between the xylitol concentration and pressure on the rejection of (i) xylitol, (ii) xylose, and (iii) arabinose as 3D response surfaces.

sugar(s) concentration are preferred for the best separation. Membrane fouling or concentration polarization may exist because of the high concentration of sugars, and thus, the membrane performance would be limited to some extent.

### 4-2. Effects of Interaction between Xylose Concentration and Pressure

Based on the analysis using Design-Expert software, the increase in the operating pressure from 4 to 10 bars increased the rejections of xylitol and xylose as the xylose concentration reached 3 g/L, as shown in Fig. 8(i) and (ii). The rejections then decreased as the concentration reached 5 g/L, while the arabinose rejection increased as the pressure increased from 4 to 10 bars, as shown in Fig. 8(iii). The rejection values for the three compounds were directly dependent on the total sugar concentration of the feed solution, and in-

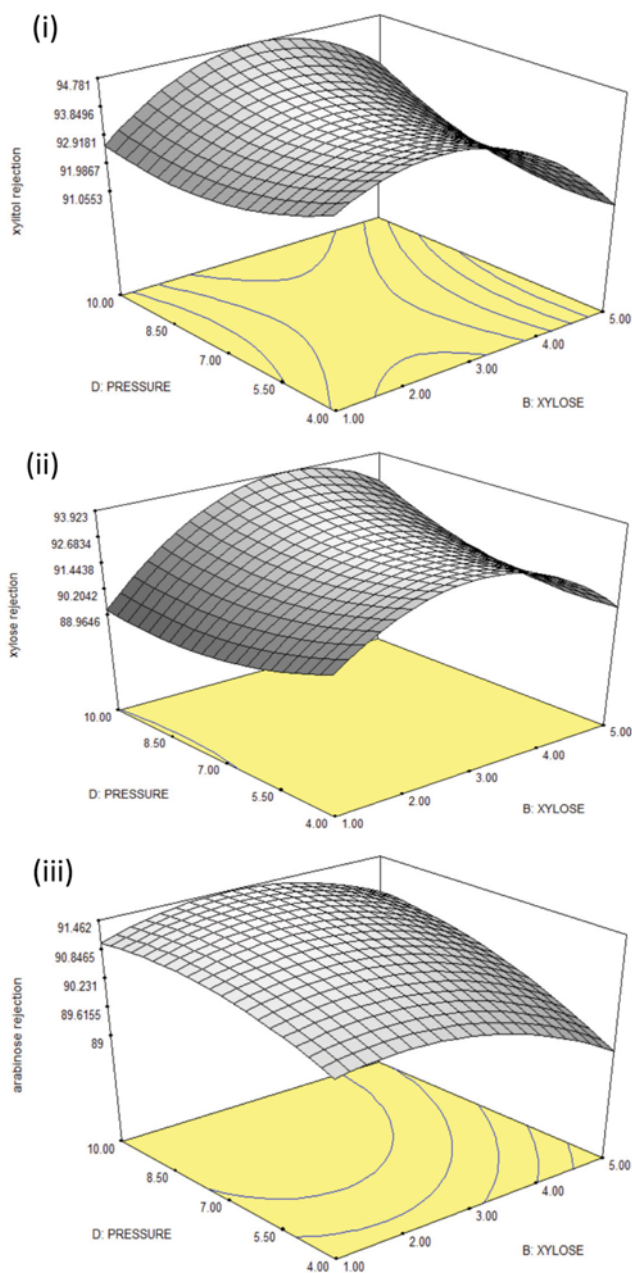


Fig. 8. Effect of interaction between the xylose concentration and pressure on the rejection of (i) xylitol, (ii) xylose, and (iii) arabinose as 3D response surfaces.

creased with pressure due to membrane compaction. The thickness of the membrane was reduced by compaction, and that would normally lead to a reduction in the pore size, which was the predominant characteristic on which depended the rejection of neutral solutes (sieving effect), hence causing an overall increase in the observed rejections. Increased pressure also caused the differences between the rejections of the three sugars to decrease, indicating a less effective separation. This was because the effect of pressure on the rejections was less marked as the molecular weight of the sugars increased with respect to the pore size of the membrane used. As the pore size decreased, the increasing pressure caused greater solute rejection [29]. It was preferable to use a low pressure of 4

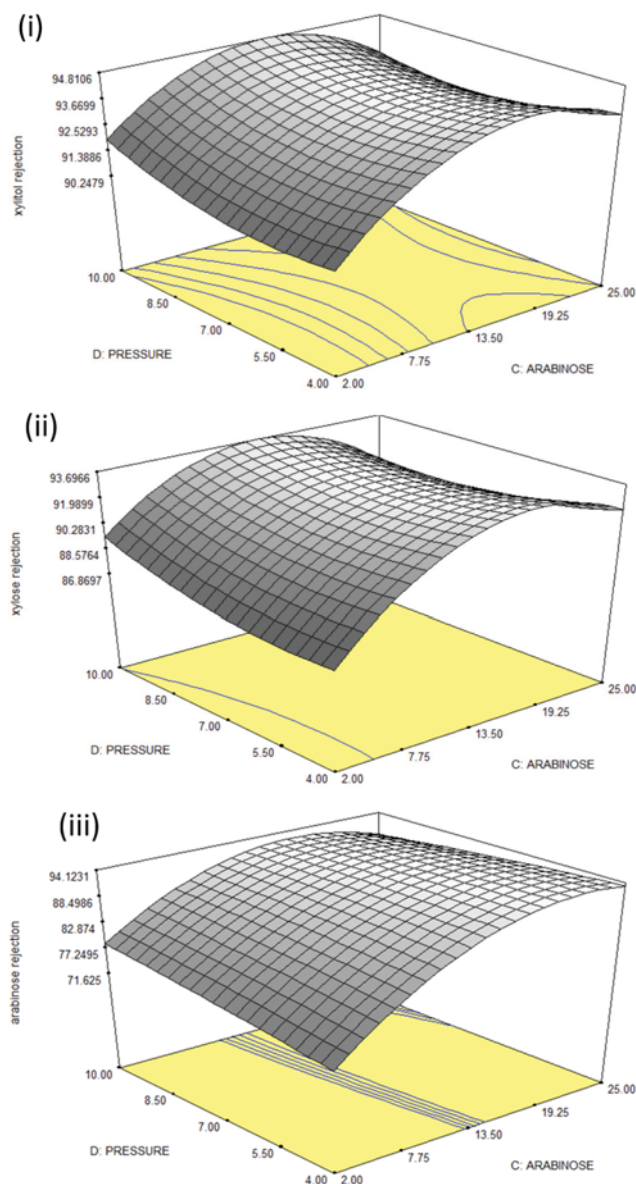


Fig. 9. Effect of interaction between the arabinose concentration and pressure on the rejection of (i) xylitol, (ii) xylose, and (iii) arabinose as 3D response surfaces.

bars to allow the arabinose to permeate through the membrane, thus retaining xylitol and xylose in the retentate in order to increase the purity of the xylitol.

#### 4-3. Effects of Interaction between Arabinose Concentration and Pressure

The analysis performed by Design-Expert software showed that when the pressure was increased from 4 to 10 bars, the solution rejection increased, and then dropped as the concentrations increased, as illustrated in Fig. 9(i) and (ii). However, Fig. 9(iii) shows that the arabinose rejection dropped at high pressure. With the NF membrane and neutral solutes, as higher pressure was applied, the molecular size of the sugar decreased in the feed, leading to greater changes in rejection. The increase in the rejection (at low concentrations) when a higher pressure was applied led to higher solute

**Table 7. The binary solution results using pure PES 18% membrane**

Solution compositions	Xylitol rejection %	Xylose rejection %	Arabinose rejection %
Xylitol+Xylose	94	87	-
Xylitol+Arabinose	94	-	93
Xylose+Arabinose	-	86	92

flux. Thus, the transport of the solutes through the membrane remained the same at a higher pressure, thereby reducing the solute concentration in the permeate stream. To retain the xylitol and to save energy during the filtration, a low operating pressure of 4 bars was chosen to purify the xylitol from its mixture.

### 5. Effect of Xylitol/Sugars Ratio

The effect of sugars (xylose and arabinose) on the rejection of xylitol in a binary solution was investigated and carried out according to Affleck (xylitol 88.0 g/L, arabinose 25.0 g/L, and xylose 4.5 g/L) [9]. The final rejection results are summarized in Table 7.

From Table 7, arabinose was clearly the dominant factor during the separation of xylitol using the fabricated PES 18% membrane. This membrane was able to be used to separate xylitol from xylose as well as xylose from arabinose, but the selectivity of the membrane was very much lowered when a solution of xylitol and arabinose together was filtered. This phenomenon may have been due to two main reasons:

1. The hydrophilicity of the xylitol and sugars: The hydrophilicity is expressed by the logarithm of the octanol-water partition coefficient ( $\log P$ ). The hydrophilicity of xylitol at  $-2.946$  is higher than that of xylose and arabinose at  $-2.74$ , as reported in the Crippen Method [32], which might cause a high rejection of xylitol, followed by both sugars. This effect of hydrophilicity on the rejection was also referred to by Braeken et al. [33], where the influence of hydrophobicity was investigated for organic molecules, where their molecular weight was below the MWCO of the membrane. A good correlation was found between hydrophobicity and retention, where the molecules with a high  $\log P$  (hydrophobic) generally showed low retention, while molecules with a negative  $\log P$  (hydrophilic) showed a high retention.

2. The solubility of xylitol and sugars: Sugar alcohol has a higher solubility than its corresponding sugars, which makes the purification of xylitol in a mixture of sugars easy [34,35]. The solubility of xylitol and sugars in water at room temperature follows the order of xylitol>xylose>arabinose [36].

This explains the high rejection of xylitol and the low rejection of xylose followed by arabinose in the mixture solution when low concentrations of xylose and arabinose were used. Since arabinose is the least soluble, it may crystallize on the membrane surface and in the membrane pores. As a result, it may cause the pores to be blocked, and further result in a higher rejection of xylitol based on its hydrophilicity. This would further justify the results obtained by the DOE, where arabinose agglomeration possibly occurred at high concentrations of xylose and arabinose, thereby causing low membrane selectivity. This result corresponded with the optimum solution rejection, where all the membranes showed a high rejection of xylitol, followed by xylose and arabinose. The relationship be-

tween the effect of the membrane pores and the total solution concentration might be helpful for understanding the effect of the types of sugars and their concentrations on the solution rejection. As explained by Sjöman et al. [31], the xylose-to-glucose ratio affects both the total sugar flux and the solvent, i.e., the water flux. Interactions of monosaccharides with water molecules are believed to influence changes in the composition of the solution. Thus, friction inside the pores will grow as the ratio of glucose increases in the solution, and the sugar and water fluxes are reduced.

### 6. Optimization and Model Validation

The optimum conditions for high xylitol rejection were predicted by the Box-Behnken design, and the results are shown in Figs. 4 to 9. The best membrane separation performance was estimated to be at a relatively high xylitol concentration and at low xylose and arabinose concentrations, as well as at low operating pressures. Based on the experimental results, any change in the set factors, either more or less than the optimal values, may lead to undesirable membrane selectivity. A validation step is very important to check the prediction stability of the model for the factors selected in the separation of xylitol. To investigate the validity of the prediction, a higher desirability function of the final prediction value of the factors for optimization was required. A numerical optimization of the DOE software was selected to obtain the maximum value of the desirability function ( $D=1.000$ ) according to the location of the specific points of the controlling factor [37]. The optimization criteria were specified at five options--none, maximum, minimum, target and within range, which were applied to determine the optimum conditions of the separation performance. In this study, all the input factors were set at the range options for all the identified factors. The maximum option was fixed for the rejections of xylitol and xylose, whereas the rejection of arabinose was chosen at the minimum option. The rejection of xylose was selected at the maximum option in order to obtain a high xylitol rejection. If the rejection of xylose had been set at the minimum option, the xylitol rejection would have dropped sharply, and thus, the purity of the xylitol would have been lost.

The optimum conditions for all the factors to obtain the best separation performance were obtained by the Box-Behnken design, and the results are presented in Fig. 10. The predicted input factors were 43.4 g/L xylitol, 1.8 g/L xylose, 2 g/L arabinose, and the operating pressure was 4 bars at a good level of desirability ( $D=0.67$ ). On the other hand, the responses were 85% xylitol rejection, 82% xylose rejection and 65% arabinose rejection. To validate the predictions, experimental runs were carried out using the predicted optimal conditions, duplicating them and recording the average values. Table 8 shows the predicted and experimental results of the rejections for comparison and validation purposes. The software provided the predicted values, and a confirmation run was required to verify the predictions. The differences between the predicted and real values were calculated to determine the error percentages. The error percentages were calculated based on Eq. (10):

$$\text{ERROR (\%)} = \frac{(\text{Experimental value} - \text{Predicted value})}{\text{Experimental value}} \quad (10)$$

The experimental values of the rejection of xylitol, xylose and arabinose obtained by HPLC were found to be mathematically in good

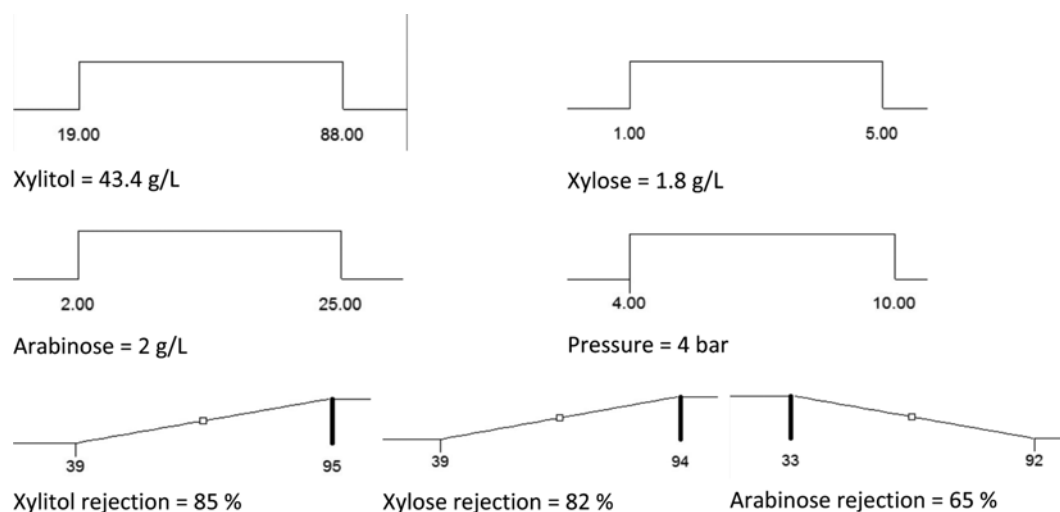


Fig. 10. Predicted conditions to obtain best separation performance using a Box-Behnken design.

Table 8. Predicted and experimental results using pure PES 18% membrane

Component	Concentration	Predicted rejection	Experimental rejection	Relative error
Xylitol	43.4 g/L	85%	94%	9.6%
Xylose	1.8 g/L	82%	76%	-7.9%
Arabinose	2 g/L	65%	72%	9.7%

agreement with the predicted results. This confirmed the adequacy of the selected model and the evaluated properties. This relative error corresponded with the results obtained by other researchers [38,39]. The deviation in the values between the experimental and the predicted results could be attributed to a combination of both the measurement error and experimental error. This could also probably be due to the inconsistent performance of the fabricated membrane compared to commercial membranes [40]. These results demonstrated that the selection of a Box-Behnken design to study the separation performance of xylitol solution was a good choice. In addition, the appropriate choice of factor levels played an important role in the optimization of the xylitol solution filtration, as reported earlier [41].

### 7. Membrane Fouling of Pure PES Membrane

Under a constant pressure of 4 bars with rigorous stirring at 500 rpm near the surface of the membrane, the optimum solution obtained from the predicted RSM inputs was filtered through the membrane in order to monitor the decline in the solute flux along with the time variation, which may have been caused by membrane fouling. The filtration process was conducted in three stages, with 60 minutes for each stage, as shown in Fig. 11. The first stage was the pure water flux, the second stage was the xylitol nanofiltration, and the third stage was the water flux again. These results were used to evaluate the membrane performance over the filtration time. As seen in Fig. 11, the initial water flux was at 13 L/m<sup>2</sup>·h (first stage), then the solute flux started at 9 L/m<sup>2</sup>·h (second stage), while the final water flux was recovered at 12 L/m<sup>2</sup>·h (third stage). There was a decline in the first few minutes of filtration before the flux became

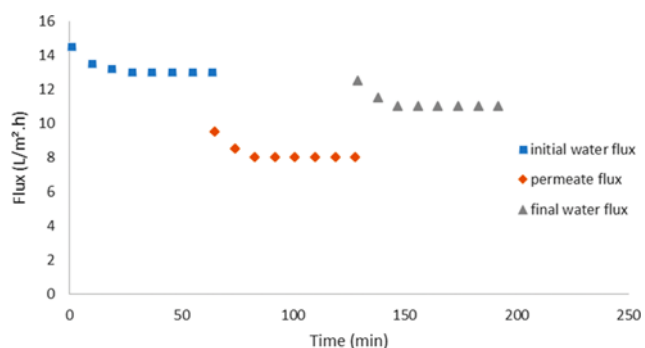


Fig. 11. Flux variation of pure water and xylitol solution as a function of time for pure PES membrane during nanofiltration. The NF process included three steps: initial water flux, nanofiltration of xylitol solution, and final water flux.

steady, which was due to the deposition of xylitol molecules. Later, the membrane recovered almost the same water flux, which indicated the presence of reversible fouling. According to the literature [42], the occurrence of reversible fouling in this study was caused by the concentration polarization of xylitol, which was used at high concentrations. In long-term filtration (as observed in this study), the membrane filtration process is not expected to experience irreversible fouling since the membrane is able to recover the water flux easily. In some cases, a reversible fouling layer on the membrane might be transformed into an irreversible fouling layer due to the formation of a strong matrix composed of a fouling layer with dissolved materials and the compaction of the fouling layer during continuous filtration. However, in this particular work, neither a strong attachment of foulants nor a cake formation was observed. The use of a highly concentrated solution only led to concentration polarization, as represented by the reversible fouling. This was probably due to the use of the broth solution model, which consisted of only three main components. A high concentration solution and the neutrality of xylitol and sugars were the dominant variables (effects) during the filtration. The effects of the membrane pores and the membrane surface charge, however, were

subservient. It could be concluded from the results that the fabricated membrane was able to operate in a long-term process with swift water flux recovery.

### 8. Validation of the Filtration Process Optimization Compared to Other Studies

The effects of the concentration of sugars and xylitol, the composition of the solution and the operating pressure on the membrane separation performance have been reported in the literature [34,43]. In this study, the predicted results of the separation performance were compared to those in other studies. A good separation between xylitol and sugars was achieved using the fabricated PES NF membrane. Further, the effects of the hydrophilicity of the sugars and their solubility in water were investigated thoroughly. The effect of a low operating pressure was apparent, where at 4 bars, the membrane showed good separation selectivity. However, Mah et al. [34] found that the sugar ratio has a high impact on the separation performance, while the operating pressure has no effect. Meanwhile, Sjöman et al. [31] found that the filtration pressure has a significant effect on sugar retention, while the total solution concentration has an insignificant effect, which is similar to our findings. Goulas et al. [29] also investigated the effect of pressure, solution concentration and filtration temperature on the purification of a sugar solution model, and their results on the operating pressure and solution concentration were in agreement with those of this study when an NF membrane was used for the purification. Vegas et al. [44] found that NF membranes are the most suitable candidates for the concentration of sugars as the membranes are able to retain 84 to 93% of oligosaccharides as the transmembrane pressure increases. Finally, Almazán et al. [43] analyzed the effects of concentration polarization and osmotic pressure on the nanofiltration of glucose. The results showed that increasing the transmembrane pressure will increase the rejection of glucose, while a high glucose concentration will cause a low rejection of glucose due to concentration polarization. This concentration polarization effect was also observed in this study, where the membrane selectivity dropped when high concentrations were used.

Furthermore, the goal of this optimization was to obtain high purity xylitol. The target was achieved by accomplishing the high rejection of xylitol. Based on this goal, this study offered low energy requirements by applying an operating pressure of 4 bars, while other reported studies [9] used pressures ranging from 14 to 50 bars. The application of a low pressure in the system enabled the xylitol to be in the retentate rather than in the permeate.

### CONCLUSION

A PES NF membrane was successfully synthesized via a phase inversion immersion precipitation technique. The membrane was characterized using the contact angle and AFM, and was tested for the filtration of a xylitol-sugars mixture at different concentrations, compositions and operating pressures. The water flux test gave the average pore membrane size at around 7 nm, based on the calculated pore size value using the Guerout-Elford-Ferry Equation. The relationship between the xylitol/sugars ratio and the solution rejection demonstrated that an increase in the arabinose concentration in the solution led to a decrease in the separation perfor-

mance. This study achieved its goals by obtaining a high purity of xylitol, good membrane performance for long filtration periods, and energy savings by using a low pressure of 4 bars. The membrane selectivity was observed to be quite low as a high xylitol/sugars ratio was used, and thus the solubility of the sugars was the dominant factor. This was mostly caused by the occurrence of concentration polarization, which hindered the permeation of xylitol. The increase in the concentration polarization may have been due to the small difference in size between xylitol and the sugars. This caused the build-up of both xylitol and sugars on the surface of the membrane, creating a kind of second membrane blocking the smaller molecules of xylitol from passing through. The coefficient of determination,  $R^2$  from the ANOVA study was 0.9891, thus proving that the statistical model was significant for the rejection of xylitol. The study on fouling showed the ability of the fabricated membrane to achieve good performance by recovering water flux easily. Overall, the PES NF membrane used in the pressure filtration study has a high potential for the separation of xylitol and is similar to the widely-used chromatographic method.

### ACKNOWLEDGEMENTS

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