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EVALUATION OF BACTERIAL CELLULOSE-SODIUM ALGINATE FORWARD OSMOSIS MEMBRANE FOR WATER RECOVERY

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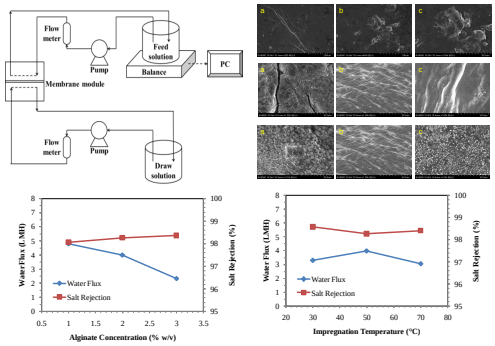
Graphical abstract



Abstract

Water resources are very important to sustain life. However, these resources have been subjected to stress due to population growth, economic and industrial growth, pollution and climate change. With these, the recovery of water from sources such as wastewater, dirty water, floodwater and seawater is a sustainable alternative. The potential of recovering water from these sources could be done by utilizing forward osmosis, a membrane process that exploits the natural osmotic pressure gradient between solutions which requires low energy operation. This study evaluated the potential of forward osmosis (FO) composite membranes fabricated from bacterial cellulose (BC) and modified with sodium alginate. The membranes were evaluated for water flux and salt rejection. The effect of alginate concentrations and impregnation temperatures were evaluated using 0.6 M sodium chloride solution as feed and 2 M glucose solution as the draw solution. The membranes were characterized by Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Contact Angle Meter (CAM). The use of sodium alginate in BC membrane showed a thicker membrane (38.3 μm to 67.6 μm), denser structure (shown in the SEM images), and more hydrophilic (contact angle ranges from 28.39° to 32.97°) compared to the pristine BC membrane (thickness = 12.8 μm and contact angle = 66.13°). Furthermore, the alginate modification lowered the water flux of the BC membrane from 9.283 L/m^2-h (LMH) to value ranging from 2.314 to 4.797 LMH but the improvement in salt rejection was prominent (up to 98.57%).

Keywords: Bacterial cellulose, sodium alginate, forward osmosis, water recovery, composite membrane

1.0 INTRODUCTION

Water is an important requirement to sustain life, the environment, and development; however, water resources are highly vulnerable to stress due to population growth, economic and industrial progress, pollution, and climate change. Four hundred fifty-eight million people from 31 countries since 1995 are currently experiencing water stress [1]. United Nation had predicted that by 2025, this number will increase

to over 2.8 billion people across 48 countries due to mostly half of the river and lakes across the globe are already polluted [2]. With these in mind, utilization of potential technologies that could recover water from untapped water resources is necessary. These potential sources of water include dirty water from agriculture, industry, flood and storm water [3]. Treatment of dirty water is considered one of the sustainable solutions for water scarcity. Conventional membrane processes that convert dirty water to clean water include microfiltration, ultrafiltration, nanofiltration, and reverse osmosis [4]. These technologies have been effective in removing contaminants in dirty water. However, some disadvantages limit its utilization such as high energy consumption and high tendency of fouling due to pressure-driven requirement resulting to high operation cost and product quality decline. This problem could be avoided by utilizing forward osmosis.

Forward osmosis (FO) is a membrane process that utilizes the natural osmotic pressure gradient which drives the transport of water molecules from the feed through a semipermeable membrane with the help of highly concentrated solution on the permeate side of the membrane (known as draw solution) [4]. FO has been applied on seawater desalination, water treatment, water purification, power generation, concentration of diluted solutions, food processing, removal of trace organic matter, and as membrane bioreactor [4-6]. It is considered as an alternative technology for water treatment due to the following advantages: (1) low energy consumption, (2) low fouling potential, and (3) high physical cleaning efficiency due to no applied hydraulic pressure [4, 7-8]. Several challenges of FO process include energy efficiency, membrane performance, optimal draw solutes, and its potential application [9].

The membrane commonly used for FO application is cellulose triacetate membrane or commercial thin-film composite membrane, which is currently applied in water and wastewater treatment [3, 10]. However, in terms of cost and environmental issues, they are not considered as the best membrane for forward osmosis operation. One potential material that could be utilized as FO membrane is bacterial cellulose. Bacterial cellulose (BC) is a β-glucan biopolymer, which is composed of β-1, 4-glucopyranosyl units with polymerization degree up to several millions [11]. It can also be defined as an extracellular polymer produced by several species of microorganisms. This is popularly known as nata de coco in Asia. It has been considered as a potential material for FO membrane because of its properties such as high tensile strength, flexible, high porosity, high water holding capacity and permeable, low cost, easy control to the material characteristic by just modifying the bacterial culture condition and biodegradable [12]. *Acetobacter xylinum* is a non-pathogenic, rod shaped, aerobic gram negative bacterium, which is capable in producing bacterial cellulose with high yield in a short period of time [13]. Because of its properties, economical production cost, and ecofriendly nature, BC has been attempted to be applied in membrane technologies for many various purposes such as pervaporation, dialysis, electrodialysis, fuel cell membrane, and as wound-dressing [13-21].

In this study, forward osmosis membranes were fabricated from bacterial cellulose produced from *Acetobacter xylinum*. Then the membranes were modified with sodium alginate solutions at different concentrations and impregnation temperatures. The composite membranes were characterized using digital microcaliper, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Contact Angle Meter (CAM) for thickness, surface morphology, chemical properties, and hydrophilicity, respectively. The membrane performance was evaluated for water recovery using the laboratory-scale forward osmosis system.

2.0 METHODOLOGY

2.1 Materials

The *Acetobacter xylinum* stock culture was purchased from Department of Science and Technology's (DOST) Advanced Science and Technology Institute (Philippines). Sucrose was obtained from Techno Pharmchem Haryana (India). Ammonium sulfate and sodium alginate was purchased from HiMedia Laboratory Pvt. Ltd. (India). Glacial acetic acid (technical grade) was procured from Mars Laboratory Instruments Center. Sodium hydroxide and calcium chloride were obtained from Merck (Germany). Glucose was supplied by Crismon Enterprise (Philippines). Sodium chloride was obtained from Ajax FineChem (Autralia). Coconut water was obtained in a local market in Manila, Philippines.

2.2 Bacterial Membrane Preparation and Modification

The composition of the culture medium used in the experiment is: 5.0% (w/v) sucrose, 0.05% (w/v) ammonium sulfate, 1.0% (v/v) acetic acid and filtered coconut water. The medium was sterilized at 121°C for 20 min.

The pre-culture was aseptically prepared by transferring 10 mL of *Acetobacter xylinum* stock culture to 200 mL sterilized coconut-water-based medium [15]. The pre-culture was incubated at 30 ± 2°C for 7 days in static condition.

For the BC membrane fabrication, 4 mL pre-culture was transferred to 40 mL coconut-water-based medium in a sterilized petri dish and statically incubated at 30 ± 2°C for 7 days. The membranes were washed for 30 min under running water and treated with 1% (w/v) NaOH for 24 h. The treated

membrane was rinsed with deionized water until the pH of the rinsed water was about 7. Finally, the membrane was stored in deionized water at 4°C until further use.

The BC membranes were modified by immersing it into sodium alginate solution at specified concentration (1%, 2% and 3%) and temperature (30°C, 50°C, and 70°C) for 5 days. The membranes were rinsed with deionized water and cross-linked with 5% (w/v) calcium chloride solutions for 3 h. Then it was rinsed again with deionized water and dried at room temperature for 3 days.

2.3 Membrane Characterization

The membrane thickness was determined using a digital microcaliper. The measurement of thickness was carried out at fifteen different spots of the dried membrane and reported as average.

Surface morphologies of the membranes were viewed at 15 kV with magnifications of 200X, 500X, and 1000X using Scanning Electron Microscopy (SEM). Fourier-transform infrared (FTIR) spectroscopy was used to determine the chemical properties of the composite membrane with wavenumber range of 4000-400 cm^{-1}. Both SEM and FTIR analyses were conducted at the Environmental Chemistry and Catalysis Laboratory, Institute of Environmental Engineering, National Chiao Tung University, Taiwan.

A CAM-PLUS contact angle meter equipped with a charge-coupled device camera was utilized to measure the contact angle. The contact angle was determined by dropping droplets (~5 µL) of deionized water randomly on the surface of a dried membrane. The average contact angle value of five different droplets was reported.

2.3 Forward Osmosis Experiments

The performance of the fabricated composite membranes was evaluated using a laboratory-scale forward osmosis system illustrated in Figure 1. An osmotic dilution mode was operated with a recirculation loop on each side of the membrane. The feed solution and draw solution containers were linked to the custom-made membrane module via gear pumps. On both sides of the membrane, two symmetric flow channels were set up.

Membrane performance was determined using 0.6 M NaCl solution as feed and 2 M glucose solution as draw solution with 0.5 L volume each [5]. The recirculation of the feed solution and draw solution were set at a flow rate of 200 mL/min. The mass change of the feed solution was monitored for two hours to determine the water flux across the membrane.

The water flux was calculated using (1):

$$J_w = \frac{V_{Draw,i} - V_{Draw,f}}{\Delta t * A_{mem}} \quad (1)$$

where J_w is the water flux reported as LMH (L/m^2-h), $V_{Draw,i}$ and $V_{Draw,f}$ are the initial and final volume of draw solution, respectively, Δt is the time of forward osmosis operation and A_{mem} is the membrane effective area [5].

The salt rejection was determined using (2):

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

where C_p and C_f are the concentration of target solute in the permeate and the feed, respectively.

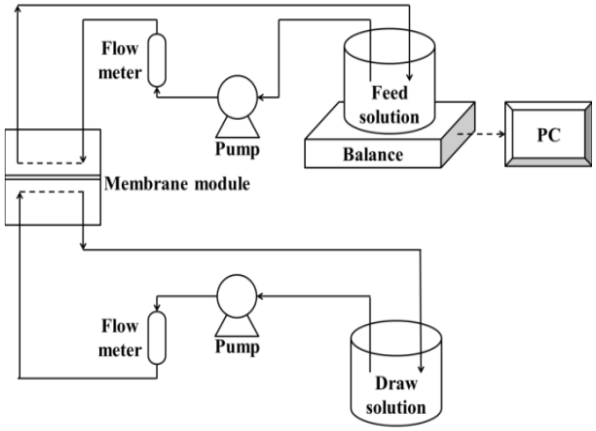


Figure 1 Set-up of Laboratory-scale Forward Osmosis System

3.0 RESULTS AND DISCUSSION

3.1 Membrane Thickness

The pristine BC membrane has a thickness of 12.8 µm. The thickness of the BC membrane was observed to increase after the modification with alginate. The thickness of the modified BC membrane ranges from 38.3 µm to 67.6 µm. The effects of alginate concentration and impregnation temperature on the membrane thickness are shown in Figure 2 and Figure 3, respectively.

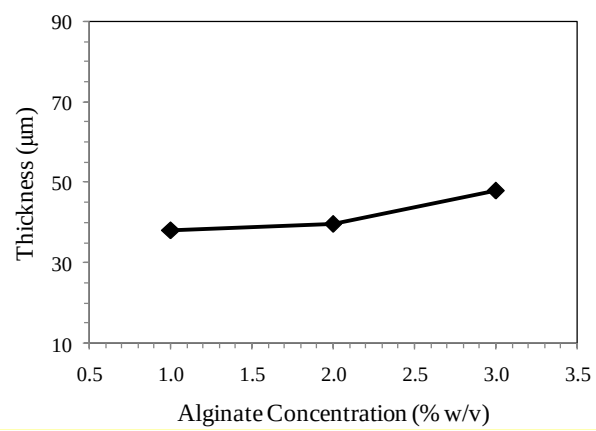


Figure 2 Effect of alginate concentration on membrane thickness at 50°C impregnation temperature and 3-h cross-linking

Figure 2 shows that the membrane thickness increases as the alginate concentration increases. The increase was caused by the incorporation of more alginate molecules in the BC membrane. The alginate molecules penetrate the pores of the BC membrane and were cross-linked thru the cation exchange of Na^+ with Ca^+ that forms the water-insoluble calcium-alginate complex within the BC matrix [15].

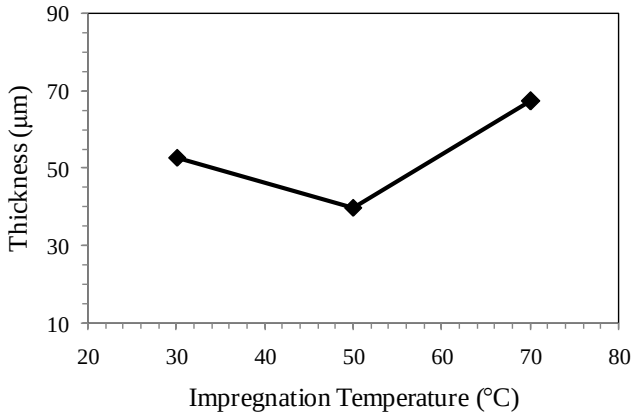


Figure 3 Effect of impregnation temperature on membrane thickness at 2% alginate concentration and 3-h cross-linking

On the other hand, Figure 3 shows the effect of impregnation temperature in the membrane thickness. It was observed that the thickness was reduced as the impregnation temperature was increased from 30°C to 50°C. However, further increase in impregnation temperature to 70°C increased the membrane thickness, even beyond the 30°C thickness.

3.2 Membrane Surface Morphology

SEM images of pristine BC membrane surface are shown in Figure 4. The bare BC membrane shows the presence of pores on the surface at 200X magnification (Figure 4a) while at higher magnification (Figure 4b and 4c) lumps of cellulose are present causing roughness on the membrane surface.

Figure 5 shows the SEM images of the modified BC membranes of varying alginate concentrations (1000X magnification) at 50°C impregnation temperature and 3-h cross-linking. The surface of the modified membrane shows smoother surface, more compact and denser structure compared to the pristine BC membrane. This is an indication that alginate altered the structure of the pristine BC membrane by filling its pores and coated the membrane surface of the cross-linked calcium-alginate, thus increasing the thickness of the membrane.

Figure 5a shows that modification of BC membrane with 1% (w/v) sodium alginate solution produced a membrane with rough surface and cracks. This is attributed to the low alginate concentration. On the other hand, the surface of the modified membrane with 2% (w/v) sodium alginate solution (Figure 5b) was smoother and completely covered. Furthermore, the surface of the modified membrane with 3% (w/v) sodium alginate solution (Figure 5c) was completely covered with lumps of calcium-alginate which provide roughness to the membrane surface due to high alginate concentration.

The effect of impregnation temperature in the modified BC membrane surface (using 2% (w/v) sodium alginate solution and cross-linked for 3 hours) is shown in Figure 6. At 30°C impregnation temperature (Figure 6a), the surface of the modified BC membrane was rough due to the presence of globular structures attributed to calcium-alginate. While at 50°C, the membrane surface has a smoother surface. Increasing the impregnation temperature to 70°C (Figure 6c) create a finer globular structure of calcium-alginate on the surface of the membrane.

3.3 Chemical Properties

Figure 7 shows the FTIR analysis of the pristine and modified BC membranes. The existence of bands at 1600 and 3100-3500 cm^{-1} for the unmodified BC membrane is a strong indication of the presence of glucose carbonyl and hydroxyl functionality, respectively [15].

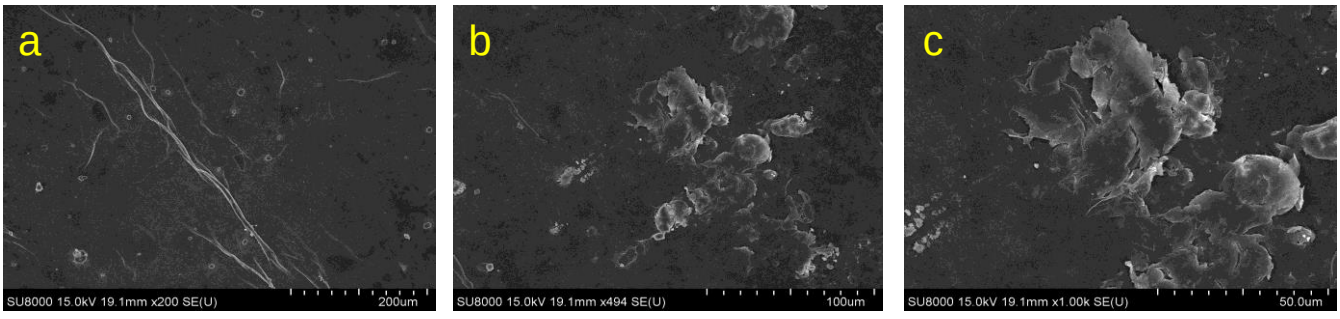


Figure 4 SEM images of the pristine BC membrane at (a) 200X, (b) 500X, and (c) 1000X magnification

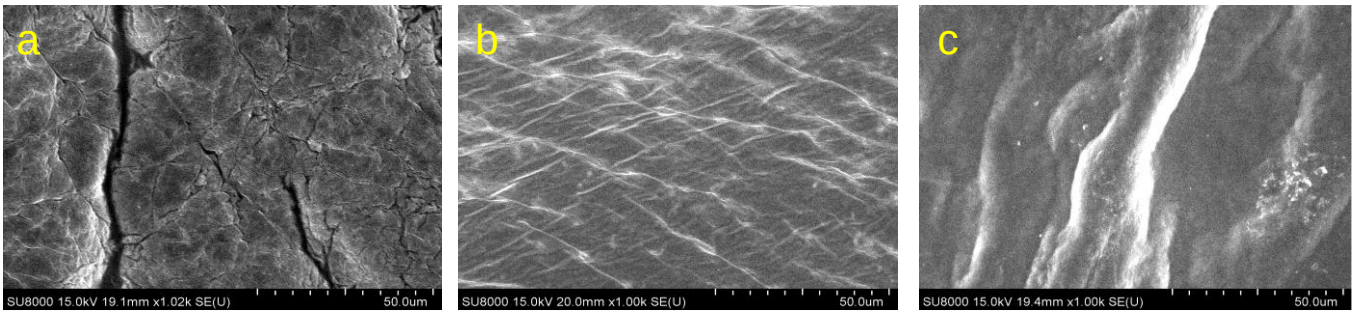


Figure 5 SEM images of the active layer of the modified BC membrane with (a) 1% (w/v), (b) 2% (w/v), and (c) 3% (w/v) sodium alginate at 50°C impregnation temperature and 3-hrs cross-linking (1000X magnification)

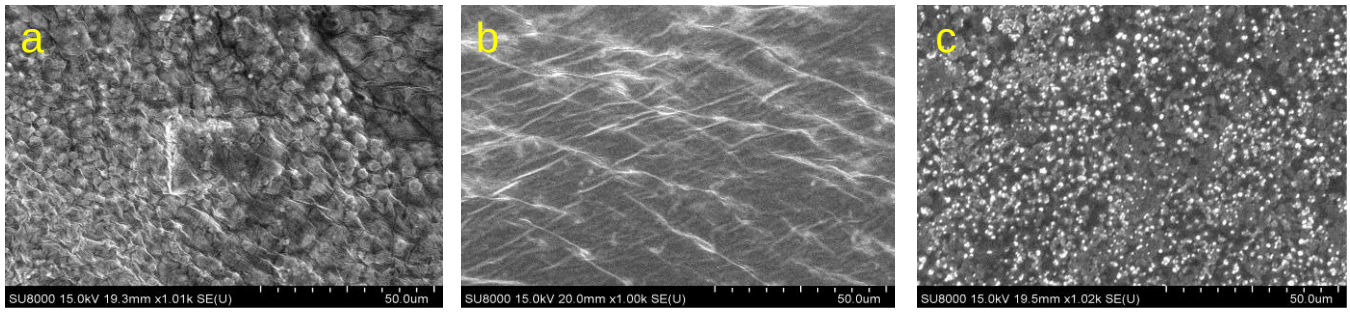


Figure 6 SEM images of the active layer of the modified BC membrane with 2% (w/v) sodium alginate at varying impregnation temperatures of (a) 30°C, (b) 50°C, and (c) 70°C, crosslinked for 3 hours (1000X magnification)

The appearance of the broad signal at 2900-3600 cm^{-1} and disappearance of the peak signals from 900-1200 cm^{-1} are strong indications of the presence of carboxyl group associated to alginate on the surface of the BC membrane. These changes in surface functionality strongly suggest that the BC membrane was modified.

3.4 Contact Angle

Membrane contact angle is an indication of the surface energy properties of a material which include interfacial interaction, wettability and favorable hydrophilicity/hydrophobicity of a surface [22]. The contact angle of the fabricated membranes is shown in Figure 8. The contact angle of the pristine BC membrane was 66.13°. On the other hand, the contact angle of the BC membrane was reduced to 28.39 - 32.97° upon modification. This shows that the decrease in the contact angle of the BC membrane was due to the surface modification with alginate that resulted to the improvement in the hydrophilicity of the membrane. While among the modified membranes, there was no significant variation observed among contact angles. This signifies that the hydrophilicity of modified membranes was similar at different modification conditions.

3.5 Membrane Performance

The performance of the different BC membranes was evaluated by measuring the water flux and percent salt rejection using the lab-scale forward osmosis system. Water flux and salt rejection of pristine BC membrane were recorded at 9.283 LMH and 93.99%, respectively. It was observed that the water flux of the BC membrane is comparable to the performance of the HTI cellulose triacetate [5]. However, the percent salt rejection was found to be lower than the commercial membrane.

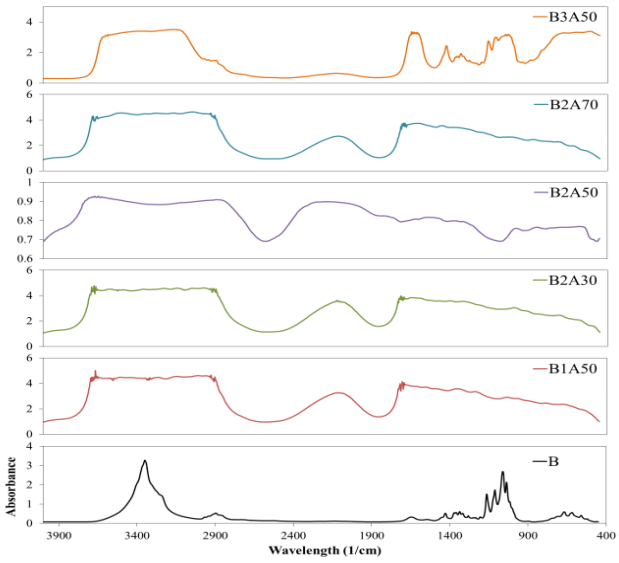


Figure 7 FTIR spectra of pristine BC membrane (B), modified BC membrane with 1% sodium alginate at 50°C (B1A50), 2% sodium alginate at 30°C (B2A30), 2% sodium alginate at 50°C (B2A50), 2% sodium alginate at 70°C (B2A70), and 3% sodium alginate at 50°C (B3A50)

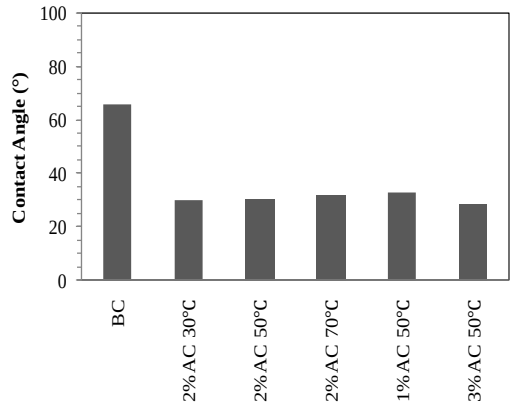


Figure 8 Contact angles of various fabricated BC membranes

The performance of the modified BC membrane is presented in Figure 9 and Figure 10. Compared to the pristine BC membrane performance, the water fluxes of modified BC membranes were lower whereas the salt rejections were higher, which is a strong indication that the capability of a BC membrane in FO process to recover water was improved by alginate modification. The presence of cross-linked alginate in the BC membrane resulted to a denser membrane which lessen water and salt molecule transport.

Effect of alginate concentration in the performance of BC membrane is shown in Figure 9. The data shows that as the alginate concentration in the membrane modification increases, the water flux decreases whereas the salt rejection increases. This could be attributed to the formation of denser and thicker membrane structure which increased the resistance for both the water and salt permeation across the membrane. This behavior was also observed in the pervaporation performance of an alginate modified BC membrane for ethanol-water separation [15]. Highest salt rejection (98.36%) was observed for modified BC membrane with 3% sodium alginate solution and water flux of 2.31 LMH. At 1% sodium alginate solution where the highest water flux was recorded at 4.80 LMH, 98.07% salt rejection was observed.

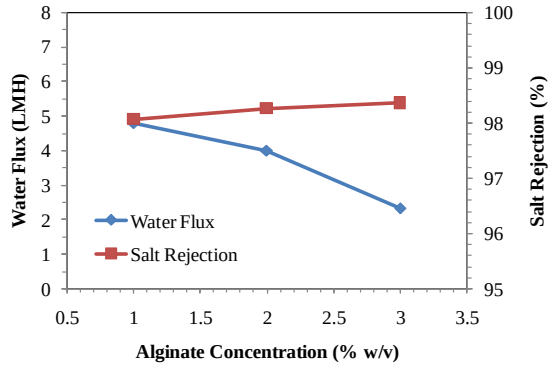


Figure 9 Effect of alginate concentration on forward osmosis performance of modified BC membrane at 50°C impregnation temperature and 3-hrs cross-linking

Figure 10 shows the effect of impregnation temperature in the performance of the modified BC membrane. It was observed that as the impregnation temperature increases, salt rejection decreases while the water flux shows to be highest at 50°C. This behavior is attributed to the membrane thickness which provides resistance in the transport of water and salt molecule within the dense membrane.

The decrease in water flux can also be attributed to the internal concentration polarization occurring inside a composite membrane [23]. The build-up of draw solution salts in the support layer of the modified BC membrane causes internal concentration polarization that significantly reduces the available osmotic driving force [24]. The DS salts deposited within the membrane prevented the passage of water across the membrane resulting to the decrease in water flux. In addition, the formation of active layer, through modification with sodium alginate, decreased the rate of water permeation, which contributed to the reduced water flux as well as the enhanced rejection of the feed salts.

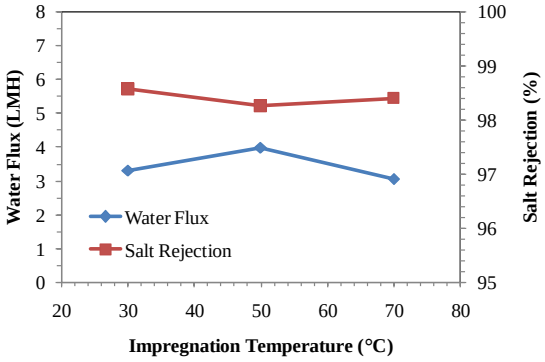


Figure 10 Effect of impregnation temperature on the performance of the modified BC membrane with 2% (w/v) alginate solution and 3-h cross-linking

4.0 CONCLUSION

Bacterial cellulose/alginate forward osmosis (FO) composite membranes were fabricated from *Acetobacter xylinum* modified by sodium alginate and cross-linked with calcium chloride. The membranes were characterized in terms of its surface morphology, surface structure, and hydrophilicity. The employment of sodium alginate in pristine BC membrane increased its thickness, improved its hydrophilic properties and changed its structure from porous to a dense membrane. The salt rejection by the BC membrane increased up to 98.57% due to the modification of the BC sheet with sodium alginate, which makes the material suitable as FO membrane. However, it has lower water flux (optimum 4.80 LMH) compared to 9.283 LMH of the pristine BC membrane.

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