

Passion fruit juice concentration using fabricated cellulose triacetate/cellulose acetate forward osmosis membrane

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Abstract

The concentration of juice using an evaporator is an energy-intensive process. Forward osmosis membrane can be used as an alternative for juice concentration at significantly low energy. In this work, fabricated cellulose triacetate (CTA)/cellulose acetate (CA) forward osmosis membrane was applied to concentrate passion fruit juice. The feasibility of the fabricated CTA/CA membrane on concentrating the passion fruit juice was determined. The FO membrane was prepared by fabricating the mixture of CTA/CA with the inclusion of polyvinylpyrrolidone (PVP) as an additive. The CTA/CA forward osmosis membrane was characterized and compared with the fabricated pure CA membrane (with the inclusion of PVP). The synthesized CTA/CA membrane was shown to have a high porosity, desired hydrophilicity, and a reduced impact of ICP, which generally affected the forward osmosis process. The concentration of passion fruit juice was successfully performed at an average of 2.2 LMH of water flux. The latter showed that the fabricated CTA/CA forward osmosis membrane has the potential to concentrate the passion fruit juice. It can be further improved by increasing the concentration of the draw solution (NaCl) or using another type of draw solution that has higher osmotic pressure.

1. Introduction

Fruit juice is a drink that contains high sources of nutrients such as vitamins, minerals, and other beneficial components, which make it essential to the human diet. One of the crucial steps in producing fruit juice is the concentration process conducted to reduce water content, eventually increasing the shelf life and reducing storage and transportation costs. A common technique to concentrate fruit juices is through the evaporation process, separating substances by thermal energy. Evaporation requires steam or hot water to evaporate or remove the water, thus consuming high energy and cost. At the same time, high temperatures will impair the quality of the juices, such as reducing the color and nutritional values (total phenolic content) (Elik *et al.*, 2016).

The membrane separation process has been employed as an alternative to conducting the concentration process. The potential of membrane technology for concentrating fruit juice is varied in terms

of membrane processes such as ultrafiltration (Bhattacharjee *et al.*, 2017), reverse osmosis (Gunathilake *et al.*, 2015; Rastogi, 2018) and forward osmosis (Rastogi, 2020; Wenten *et al.*, 2021), and utilizing the existing or new membrane materials such as using cellulosic based polymers, aromatic polyamides (PAs) (Wang, 2015), and polysulfones (PSFs) (Rashed *et al.*, 2020). The advantage of using forward osmosis for the concentration process is due to low hydraulic pressure, which leads to cost reduction of electrical energy, while ultrafiltration and reverse osmosis use high hydraulic pressure, which consequently leads to high energy and operating costs. The interest in using forward osmosis membrane processes for concentrating juice has increased. This has been shown by several studies on fruit juice concentration that utilize forward osmosis (FO) membrane technology for apple juice (An *et al.*, 2019), grape juice (Kim *et al.*, 2019), orange juice (Li *et al.*, 2021), and many other fruit juices.

FO is a cold concentration process that relies on the

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driving force generated from the osmotic pressure difference between two solutions driven under low hydraulic pressure (Rastogi, 2018). This technique, also known as direct osmosis, operates at ambient or room temperature and does not involve a phase change when concentrating the fruit juices. Thus, it saves energy and reduces the juices' thermal damage. One of the essential criteria for the successful FO process is the selection of a membrane. The best characteristic of FO membrane materials should be high solute rejection, high water permeability and high mechanical strength (Rastogi, 2020).

Cellulose acetate and/or cellulose triacetate (CTA) is a type of membrane material used for the FO process (Chanukya and Rastogi, 2017; Shang and Shi, 2018; Lakra *et al.*, 2022). The cellulose acetate (CA) membrane is naturally derived with unique qualities such as hydrophilicity, low fouling propensity, mechanical strength, and high-water flux. In contrast, CTA is produced due to a chemical reaction between the natural polymer cellulose and acetic acid. It has a homogeneous membrane structure and can be made with a wide range of permeability (Sunohara and Masuda, 2011). Due to their good film-forming ability, high chemical stability, eco-friendliness, and affordable price, CA membranes are widely employed. However, there has been a lot of interest in the idea of modifying CA by mixing with CTA during membrane fabrication to improve permeability and stability. Nguyen *et al.* (2013) have discussed the CTA/CA-based membrane preparation for forward osmosis. The performance of their membrane, which is cast at a 1:2 ratio of CTA/CA, has produced high water flux and low reverse salt flux with the potential to be used in treating wastewater. Hence, this study would improve the fabrication of CTA/CA by adding PVP (as an additive) in the formulation of membrane solution casting and its potential to be used in fruit juice concentration. Adding the PVP to the casting solution can affect membrane structure and performance. PVP is a water-soluble polymer regularly utilized as a pore-forming agent before the asymmetric membrane preparation using the phase inversion method. PVP modulates the membrane structures and stipulates morphology control related to the thermodynamics and the kinetics of polymer membranes.

Adding PVP could increase membrane selectivity with the relative transport rate reduced at the membrane surface (Rao *et al.*, 2008). Malek *et al.* (2012) showed that adding the PVP as an additive for their FO membrane fabrication had enhanced permeability and hydrophilicity. Another study by Shang and Shi (2018) added PVP as an additive to prepare the FO membrane for anthocyanin solution concentration. They observed

that the water flux was 2.04 LMH with a high rejection rate (98.61%). However, the addition of PVP increased the viscosity of the polymer solution, slowed down the demixing process, and delayed the phase separation (Zhang *et al.*, 2011), which potentially caused the polymer structure to be irregular.

This work studied the feasibility of the fabricated CTA/CA membrane on concentrating the passion fruit juice using the FO process. The FO membrane was prepared by fabricating the mixture of CTA/CA with the inclusion of PVP as an additive. The fabricated CTA/CA forward osmosis membrane was characterized and compared with the fabricated pure CA membrane (with the inclusion of PVP). The results will determine whether the fabricated membrane is appropriate for reducing water content in fruit juice.

2. Materials and methods

2.1 Materials

Passion fruit pulp was purchased from Extra Natural Sdn. Bhd., Kepong, Malaysia. Samples were kept in a refrigerated box, delivered directly to the laboratory, and stored in the freezer at -18°C. CTA (Selectophore) and CA (39.8wt% acetyl, MW 30,000 g/mol) were purchased from Sigma Aldrich. 1,4-dioxane, polyvinylpyrrolidone (PVP-K30), maleic acid, methanol, acetone, and sodium chloride were purchased from a local supplier. No further purification was conducted for all these chemicals. In the coagulation process, deionized (DI) water was used.

2.2 Preparation of cellulose acetate and cellulose triacetate/cellulose acetate -forward osmosis membrane dope

The CTA/CA-FO membrane was prepared by phase inversion following the procedure in Shang and Shi (2018) with some modifications. The CTA/CA membrane casting solution contained 6.5 wt% CA, 6.5 wt% CTA, 45 wt% 1,4-dioxane, 2 wt% maleic acid, 3 wt% PVP, 31 wt% acetone, and 6 wt% methanol. While for the casting solution of the CA membrane contained 13% CA, 45 wt% 1,4-dioxane, 2 wt% maleic acid, 3 wt% PVP, 31 wt% acetone, and 6 wt% methanol. The fully dissolved dope solution was left at an ambient condition for 24 hours to remove air bubbles and inhibit defect formation. This solution was cast on a glass plate surface, and thickness was adjusted at 200 µm using a casting knife (Zechner-Swiss) at a constant speed. The membrane was partially evaporated for 60 s under atmospheric conditions and then immersed in coagulation water. The coagulated membranes were kept in water overnight to extract residual organic solvents from the membrane.

2.3 Membrane characterization

2.3.1 Component analysis

The surface analyses were carried out by Fourier transform spectroscopy infrared with attenuated total reflection (FTIR-ATR) to investigate the functional group present in the fabricated membrane. The FTIR/ATR spectra of the fabricated membrane were determined by a Nicolet IS 10 instrument (Thermo Scientific Nicolet, USA). The samples were dried in a vacuum chamber at 75°C for 24 hrs; before conducting the measurements.

2.3.2 Membrane surface morphology

The membranes' top surface and cross-section images were observed using Scanning Electron Microscopy (Quanta 200, FEI, USA). A sample of each membrane was dried in an oven at 50°C for 24 hrs to dehydrate it. Followed by mounting the membrane on a specimen with conductive adhesive carbon tape. Surface morphologies of membranes were tested using a Multimode V (Veeco, USA) Atomic force microscopy (AFM) capable of imaging at vertical and lateral resolutions of 0.1 Å.

2.3.3 Pretreatment for clarification of juice

The food material's ability to conduct electricity depends on the product's properties, such as composition, sugar and salt content, pH, and more. Hence, it is important to measure the electrolytic conductivity of the juice in order to observe the optimum time and concentrated juice achieved. Before the concentration process, the fruit pulp was required to be clarified. The clarification method was based on Phung *et al.* (2019) with some modifications. The fruit pulp was filtered through a sieve to remove seeds and the juice through a cheesecloth to get filtered raw juice. The filtered juice was diluted with a ratio of passion fruit juice with distilled water of 2:1. Diluted passion fruit juice was mixed with pectinase enzyme (Sigma Aldrich) to clarify the juice and 250 ppm of pectinase enzyme was added into 1 L of juice. The juice was incubated in the water bath at 50°C for an hour. Then, the enzyme-treated juice was heated up (using a hot plate) to 90°C for 5 mins to deactivate pectinase activity. The enzyme-treated juice was centrifuged at 3000 rpm (using centrifuge Hitech Universal 320, Germany) for 15 mins and filtered through filter paper (110 µm) to get a clear filtrate. The clear filtrate will be used in the following experiment.

2.3.4 Membrane performance for fruit juice concentration

Figure 1 shows the schematic diagram of the laboratory scale forward osmosis membrane system.

This system consists of a peristaltic pump, stirrer, membrane holder, and electronic balance. The fabricated FO membrane was placed on the membrane holder with an effective area of 28 cm². To prevent severe internal concentration polarization and the rough effects of food compounds in the support layer; the membrane was positioned with the feed solution (FS) which is passion fruit juice against the selective membrane layer, and the draw solution (DS) is sodium chloride against the membrane support layer, which the feed and draw solutions were operated in a closed loop.

For the first 60 mins, the membrane was compacted with deionized water to ensure the stability of the FO membrane and to produce a consistent flow. Then, the membrane testing was conducted with water as the FS and 1 M NaCl as DS for an hour at a constant flow rate of 1.4 L/min pumped by peristaltic pumps. The FO process was continued with the passion fruit juice as the FS and DS (2 M NaCl). The amount of juice used was 1 L for each experiment. This performance testing was conducted for 180 mins. The readings for the mass change of DS and the electrical conductivity of FS were recorded at 15 mins intervals with the inclusion of readings taken at the 1st, 5th, and 10th min of the experiment. The transmembrane of water flux (J_w) was calculated based on the below equation (Zhang *et al.*, 2011):

$$J_w = \frac{\Delta V_{draw}}{A_m \cdot t}$$

where ΔV_{draw} is the volume change, A_m is the effective membrane surface area, and t is time. Each experiment was evaluated in duplicate. The FS electrolytic conductivity (EC) values were monitored using an EC meter (Hanna instrument).

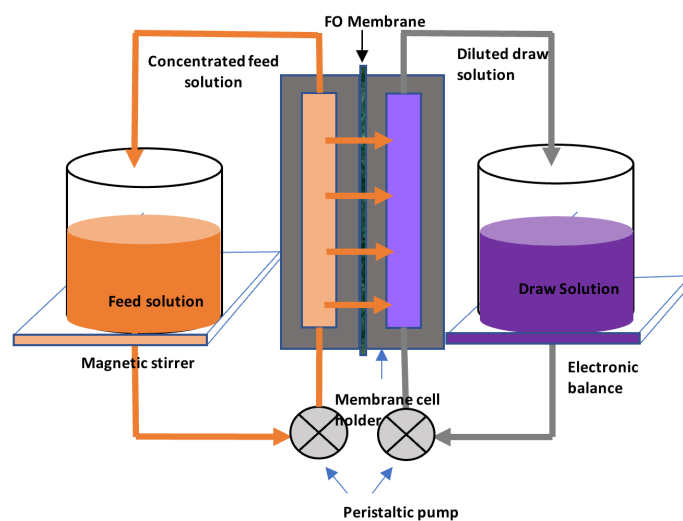


Figure 1. Schematic diagram of the forward osmosis system.

3. Results and discussion

3.1 Cellulose triacetate/cellulose acetate membrane characterization analysis

FTIR analysis was conducted to analyze the functional group in the fabricated membranes. Figure 2 shows the FTIR spectrum of the fabricated mixed CTA/CA membrane and compares it with a pure CA membrane. A broad band in the range of 3400-3550 cm^{-1} was observed, associated with the stretching vibration of the O-H group (Shang and Shi, 2018; El-Ghaffar *et al.*, 2020), indicating strong hydroxyl characteristics. The attributes of strong carbonyl (C=O stretching) for both membranes were observed at peaks of 1746 cm^{-1} (CTA/CA) and 1744 cm^{-1} (CA). The presence of carbonyl and hydroxyl characteristics verified the existence of an acetate group for both membranes (CTA/CA and CA). In comparison, medium stretching of vibrations C=C compound was observed at peaks 1645 cm^{-1} (CTA/CA) and 1633 cm^{-1} (CA), which might belong to PVP (Shang and Shi, 2018). The peak located at 1233 cm^{-1} (CTA/CA)/ 1232 cm^{-1} (CA) shows the carboxylate stretching of C-O (El-Ghaffar *et al.*, 2020). Sharp peaks were observed at 1048 cm^{-1} (CTA/CA)/1047 cm^{-1} (CA), corresponding to C-O-C stretching. The stretching of C-O and C-O-C may be related to the ether bond of CTA/CA. The peaks between 1369 -1432 cm^{-1} for both membranes correspond to the bending vibration of C-H (Shang and Shi, 2018). Overall, the absorption bands of peaks for both types of membranes are almost similar because of the similarity in the structure of CTA and CA. This indicates that the newly fabricated CTA/CA membrane can perform the concentration process like the CA membrane.

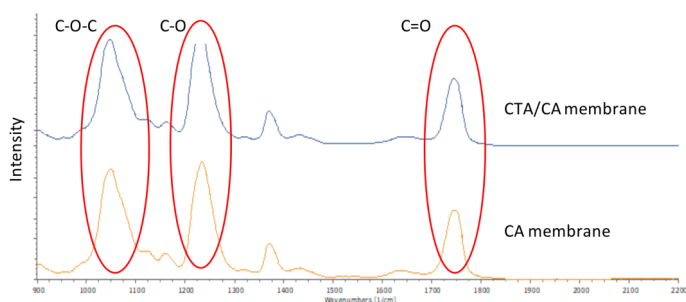


Figure 2. FTIR spectrum of the CA dan CTA/CA FO membrane. The red circle corresponds to the wavelength of the major functional groups.

The surface morphology and cross-section of mixed CTA/CA membranes were analysed using SEM. Figure 3(a) shows a detailed topography of the top surface (active side) of the CTA/CA membranes, in which pores are visible under a magnification of 5000. The pores seem uniformly distributed across the top membrane sheet with a porous structure on the surface. The agglomeration occurrences were not observed on the

membrane surface, which indicates good dispersion or blending between CTA and CA nanoparticles. Figure 3 (b) and (c) show the cross-section images (1000 magnification) of mixed CTA/CA and pure CA membrane, respectively. The ultimate properties of the membrane are significantly influenced by the uniformity of pore size. When fruit juice is concentrated by forward osmosis, uniform pore size creates a smooth selective layer that could improve the diffusion of juice molecules.

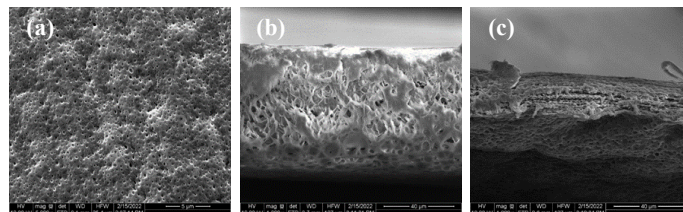


Figure 3. (a) Surface morphology of CTA/CA (5k magnification), (b) cross-sectional of CTA/CA membrane (1k magnification) and (c) cross-sectional of virgin CA membrane (1k magnification).

Surface roughness frequently affects membrane performance due to structural nodules produced from polymer aggregates. Surface roughness could be dependent on the choice of monomer, diffusivity, concentration, reactivity, and solubility (Mohammadifakhr *et al.*, 2020). The surface of the CTA/CA membrane appears highly and finely distributed. This indicates that the CTA/CA membrane surface is rougher. The surface of the CA membrane is smoother. As surface roughness rises, flux and permeability increase. More voids and networks between the layers were observed from the CTA/CA membrane's cross-section image and the nodule structure of the membranes was found to have a heterogeneous distribution of nodule agglomerates. In addition, the nodules are also slightly smaller in size. This is also evidence from the AFM image (Figure 4(a)) that it has finer features, compact surface roughness, and a void between the surface. It seems that the polymer matrices are well bound for the CTA/CA membrane. In contrast, for the CA membrane, the structure between the layers was denser or compact (Figure 3(c)), and the top layer appeared to have a smooth surface roughness (Figure 4(b)).

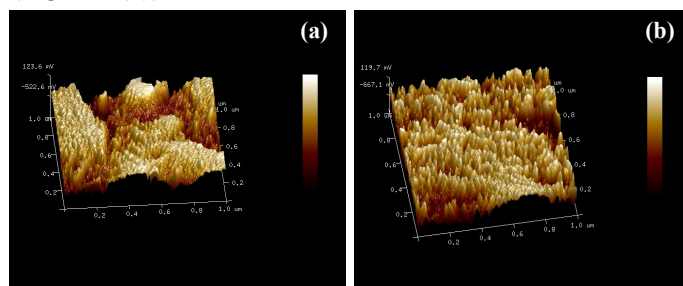


Figure 4. AFM images of active membrane surface before filtration (a) CTA/CA membrane and (b) pure CA membrane.

3.2 Concentration using fabricated cellulose triacetate/cellulose acetate forward osmosis membrane

The feasibility of concentrating the passion fruit juice using the new fabricated membrane of CTA/CA with the inclusion of PVP was investigated by observing the water flux performance. The material properties of the membrane dominate the performance of a FO process. As shown in Figure 5, the initial water flux was $5.8 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (LMH) and decreased to 0.6 LMH at 180 mins with an average of 2.2 LMH. The synthesized CTA/CA membrane was shown to have a high porosity, desired hydrophilicity, and a reduced impact of ICP, which generally affected the forward osmosis process. In contrast, Shalini and Nayak (2016), who used a CTA membrane to concentrate sugarcane juice, obtained an initial water flux of approximately 8 LMH. They have used a high molarity of DS (NaCl - 6 M) compared to this study using low DS (NaCl - 4 M). Another study by Chanukya and Rastogi (2017) had an initial water flux of approximately 9 LMH for concentrating sweet lime juice with 6 M NaCl using a CTA FO membrane assisted with ultrasound. High concentrations of draw solutions will aid in higher transmembrane flux. Thus, the water flux obtained in this study was much lower than those studied, but it can be further improved by increasing the molarity of DS. The properties of juice could also affect the water flux. For example, the pectin content in the juice could influence and create discrepancies in the results. Pectin molecules have the characteristic of forming a thin layer of gel on the membrane surface that has the potential to cause resistance to flux and decrease the mass transfer of water (Bhattacharjee et al., 2017).

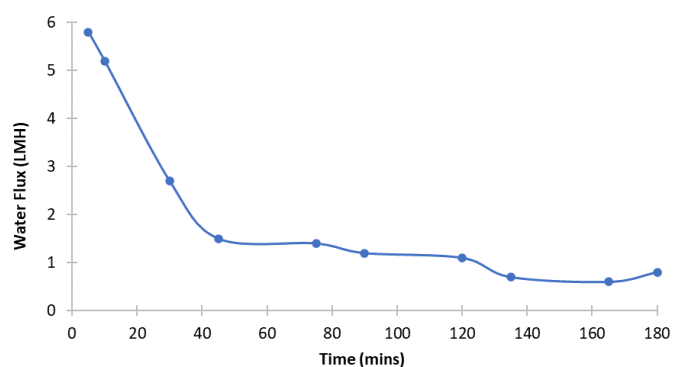


Figure 5. Water flux over time.

Deionized water was used as a feed solution for 60 mins. This testing was to ensure the stability of the FO membrane and to produce a consistent flow. The AFM image (Figure 6(a)) of the CTA/CA membrane after the filtration of water (as FS) with 4 M NaCl (DS) shows a thinner peak with a better distribution on the membrane surface comparatively before filtration (Figure 4(a)). However, Figure 6(b) shows that the peak surface roughness was damaged or destroyed after the juice

concentration. A thick cake layer was formed at a particular position on the membrane surface. The forming of this cake layer might be due to pectin molecules or insoluble solids in the passion fruit juice, and it has been trapped on the membrane surface that eventually creates the cake layer.

The electrical conductivity of food material is a function of product characteristics (composition, sugar and salt content, pH, etc). Increasing electrical conductivity values of the juice following a rise in total soluble solids (TSS) might be caused by the loss of water molecules during the forward osmosis process. Figure 7 shows the passion fruit juice's electrical conductivity (EC) during the FO process. The EC increased linearly with time, ranging from 3800-6200 $\mu\text{S}/\text{cm}$. The increase in EC can be correlated with the concentration of ions present increasing inside the juice as the water inside the juice declines over time. As the concentration of ions increase, it will induce a greater flow of electric current (Shrestha et al., 2017) as time passes. This indicates that the juice concentration increased over time and demonstrated that the FO process managed to dewater the fruit juice. EC has several applications in the food, fruit, and vegetable industries. However, much work is still expected to utilize its high-potential application.

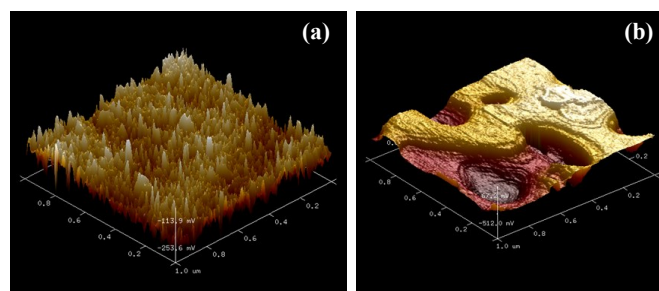


Figure 6. AFM images of CTA/CA membrane (a) after filtration of water (FS) for 60 mins using 4 M NaCl (DS) (b) after concentration of juice (FS) for 180 mins using 4 M NaCl (DS).

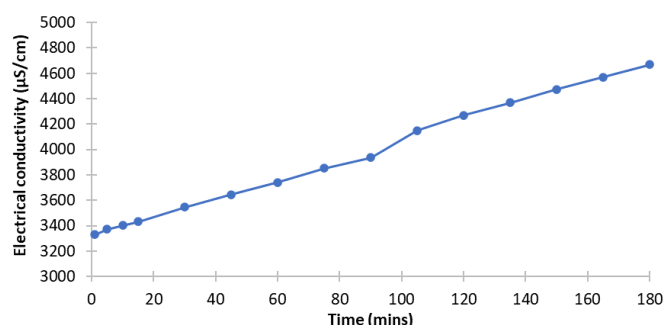


Figure 7. The juice's electrical conductivity over time.

4. Conclusion

CTA/CA forward osmosis membrane was fabricated with the inclusion of PVP as an additive. The functional groups (C-O-C, C-O and C=O) for the newly fabricated

CTA/CA membrane were similar to the CA pure membrane. The synthesized CTA/CA membrane was shown to have a high porosity, desired hydrophilicity, and a reduced impact of internal concentration polarization (ICP), which generally affected the forward osmosis process. The concentration of passion fruit juice was successfully performed at an average of 2.2 LMH of water flux. Although the water flux value was not significantly higher, it has shown that the fabricated CTA/CA forward osmosis membrane has the potential to concentrate the passion fruit juice. It can be further improved by increasing the concentration of the draw solution (NaCl) or using another type of draw solution with higher osmotic pressure.

Conflict of interests

The authors declare no conflict of interest.

Acknowledgments

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