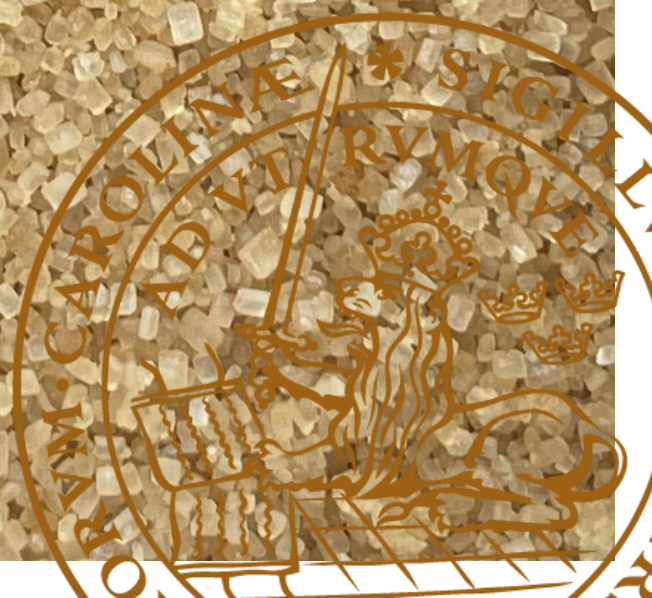


Thin Beet Juice Concentration with Forward Osmosis

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Thin Beet Juice Concentration with Forward Osmosis

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Preface



This degree project was carried out at Lund University in collaboration with Nordic Sugar as part of the Master program in Membrane Engineering for Sustainable Development.

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Generative artificial intelligence tools were used in the preparation of this thesis to improve readability and to assist with language and grammar editing. I have processed the generated text and take full responsibility for the content.



Popular Scientific Summary

The traditional beet sugar production consists of several steps: pretreatment, extraction, beet juice purification by liming, beet juice concentration, and crystallization. This process requires large amounts of heat and energy, especially during evaporation of sugar beet juice. As industries work to reduce energy consumption and greenhouse gas emissions, finding alternative methods for juice concentration has become increasingly important.

One promising technology is forward osmosis, a membrane-based process that can remove water at lower temperatures than conventional evaporation. This project examined the potential use of forward osmosis for concentration of sugar beet thin juice. The main focus of the study was the use of molasses, a byproduct of sugar production, as a draw solution. Reusing molasses could make the technology more sustainable and easier to integrate into existing sugar factories.

Experiments were carried out using different operating conditions and membranes to determine which factors strongly affect process performance. The results showed that forward osmosis can successfully sugar beet juice and that molasses can function as a draw solution. However, several challenges remain. The high viscosity of molasses slows water transport, and the accumulation of dissolved substances near and within the membrane reduces process efficiency. These effects limit the rate at which the juice can be concentrated and highlight the importance of membrane design and process optimization.

Despite these challenges, the results show the potential of forward osmosis as a lower-temperature alternative for juice concentration and industrial byproducts such as molasses can be integrated into membrane-based processes.

Summary

Sugar production is an energy-intensive industry that heavily relies on thermal evaporation for juice concentration, contributing significantly to greenhouse gas emissions. Growing pressure to improve industrial sustainability has driven interest in alternative concentration technologies. Forward osmosis (FO) has emerged as a promising non-thermal membrane process that utilizes osmotic pressure difference rather than hydraulic pressure or heat, enabling concentration at mild operating conditions.

Although FO has been extensively investigated for fruit juice concentration, limited research has focused on its application for sugar beet thin juice concentration. Previous studies mainly focused on achieving high water flux using synthetic draw solutions, while comparatively little attention has been given to the use of industrial by-products as draw solutions. Molasses, a byproduct of sugar production, offers potential as an alternative draw solution due to its compatibility with the sugar production process. Therefore, this study aimed to evaluate the feasibility of FO for thin juice concentration using molasses as a draw solution. The effect of operating parameters including draw solution type, concentration, flow rate, feed-to-draw ratio, temperature were investigated. Experiments were performed with commercially available polymeric membranes for FO, reverse osmosis (RO), and nanofiltration (NF). Process performance was evaluated through transmembrane flux behavior and analysis of juice quality parameters.

The results demonstrated that molasses successfully functioned as a draw solution, and increasing draw solution concentration, crossflow velocity, and temperature enhanced water flux due to improved osmotic driving force. Despite promising results, concentration polarization and fouling were a major limitation that reduced the process efficiency. In the comparative trials of different polymeric membranes, the cellulose triacetate FO membrane exhibited the highest flux. The long-term concentration experiment achieved a volume reduction of 0.5 and increased thin juice concentration from 16.0 to 27.9 %RDS.

These findings show the potential of FO as an alternative concentration technology for sugar production, particularly when integrated with industrial byproducts. Further optimization of hydrodynamic conditions and membrane design are needed to improve process performance and facilitate industrial implementation.

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1 Introduction

Global sugar production plays an important role in the food industry, reaching approximately 180.97 million tons in 2024-2025 and projected to increase to 189.26 million tons in 2025-2026, with Brazil, India and the European Union as leading producers [1]. Approximately 80% of the world's sugar is produced from sugar cane, while the remaining 20% is derived from sugar beets [1]. Sugar production is also highly energy-intensive, largely due to significant steam demand and electricity consumption required for thermal concentration processes [2]. Life cycle assessments of beet sugar production further highlighted the environmental impact of the industry, with greenhouse emissions estimated at approximately 623 kg CO_{2eq} per ton of white sugar [3]. 56% of these emissions were attributed to processing and 44% to beet cultivation [3].

In response to increasing environmental concerns, extensive research efforts have focused on reducing energy consumption and lowering emissions. Among emerging technologies, forward osmosis (FO) has gained attention as a promising non-thermal membrane-based separation process. It utilizes the osmotic pressure difference between a feed solution and a draw solution to drive water transport across a semipermeable membrane. Operation at low temperatures makes FO attractive for juice concentration applications, where preservation of product quality is critical.

Previous studies have demonstrated the potential of FO for concentrating both sugarcane and sugar beet juices, often employing synthetic draw solutions such as sodium chloride due to their high osmotic pressure [4-8]. However, these systems face challenges related to reverse solute flux (RSF), which can compromise final product quality and limit their industrial applicability. As a result, there is a growing interest in exploring more compatible draw solutions.

Molasses, a byproduct of sugar production, presents a promising alternative draw solution due to its high osmotic pressure, availability within the process, and compositional compatibility with sugar streams [9]. Additionally, its reuse within the production chain offers potential for process integration. Nevertheless, molasses also introduces challenges, including high viscosity, complex composition, and limited availability with only 19 kg dry matter per ton of processed sugar beet [3]. Given these opportunities and limitations, further investigation is required to evaluate the practical applicability of FO for thin beet juice concentration.

1.1 Aims

The aim of this project is to evaluate the feasibility and performance of FO as a complementary technology for thin juice concentration in beet sugar production. The study aims to:

1. Investigate the effect of operating parameters such as draw solution type, concentration, flow rate, and temperature on water flux;
2. Compare the performance of different membranes under varying conditions;
3. Assess the suitability of molasses as a draw solution;
4. Evaluate changes in thin juice quality, including pH, conductivity, color, polarimetric sucrose and purity.

2 Background

2.1 Beet Sugar Production

The sugar production process starts with cultivation of sugar beets. They are usually planted in early spring and require six to eight months to grow [10, 11]. Beets are harvested in autumn when the sucrose levels peak [10, 12]. Harvested beets are then transported to the sugar factory.

Upon arrival at the factory, beets undergo a systematic process of reception and quality analysis. Samples are taken for evaluation, cleaned, physically inspected and subjected to chemical analysis to measure sucrose content and other quality parameters [12]. Delivered beets are piled and stored in designated areas [10, 12].

The first industrial step is to wash the beets from soil, stones and debris. It is done with water combined with mechanical separators. Washed beets are then sliced into thin, V-shaped strings called cossettes in the slicing station [10-12]. This shape maximizes the surface area of beets while maintaining the structural integrity [10].

After slicing, cossettes undergo a counter-current diffusion process in the diffuser. Sucrose is locked inside the sugar beets by the cell membrane (protoplasm), which is semipermeable to water but impermeable to sucrose [10, 11]. To extract sucrose, a denaturation process takes place, where heat is applied to denature the protoplasm [10, 12]. In the diffuser, cossettes move from one end to the other, while hot water moves in the opposite direction [10]. As a result of this step, raw (diffusion) juice and pulp as a byproduct are produced [10]. Wet pulp is pressed, dried and sold as animal feed [12].

The next step in the process is purification, where nonsugars, suspended particles and colloids are removed to produce high-purity liquid for subsequent evaporation. Purification consists of a sequence of chemical and thermal treatments using milk of lime and carbonation gas [10, 12]. Lime is added to neutralize acids and precipitate impurities, while carbon dioxide is used to remove excess lime and facilitate further purification [10]. Following this production step, a purified product known as thin juice is obtained. Thin juice is a clear, thermostable liquid with purity of 92-95% and pH 8.8-9.0 [10].

The next production step is evaporation, where thin juice with about 15% dry substance (DS) is concentrated to thick juice with 70% DS. To reduce the boiling point of juice and prevent decomposition of sugars, a flashing evaporation method is utilized, where juice is concentrated under vacuum. Before entering the evaporators, juice is preheated to a temperature near the boiling point, ensuring that energy provided in the evaporation station is used strictly for vaporization. Industrial sugar production relies on multiple effect evaporation systems with three to six individual evaporators in series [12]. High pressure steam is introduced only to the first set of evaporators, and the water evaporated from the thin juice serves as a heating medium for subsequent stages [12].

The final production stage is crystallization, which takes place in cylindrical vessels known as vacuum pans. Concentrated sugar liquor is evaporated until supersaturation is reached, promoting the formation and growth of sugar crystals. The resulting mixture of syrup and sugar crystals, called massecuite, is boiled down to the final dry substance level. Massecuite is then sent to mixers before being sent to centrifuges [10]. Centrifuges apply centrifugal force, which

pushes the liquid liquor through a fine screen while retaining pure sugar crystals. Factories use a three-stage cascading scheme where the runoff syrup is reboiled in subsequent “raw” stages until the remaining liquid is exhausted as molasses [10, 12].

The sugar leaves the centrifuge as “wet sugar” and must be moved to drying and cooling equipment. After the drying process, sugar requires conditioning and maturation for 12 to 48 hours in specialized silos to reach stability [10]. The summary of the production stages is presented in the Figure 2.1.

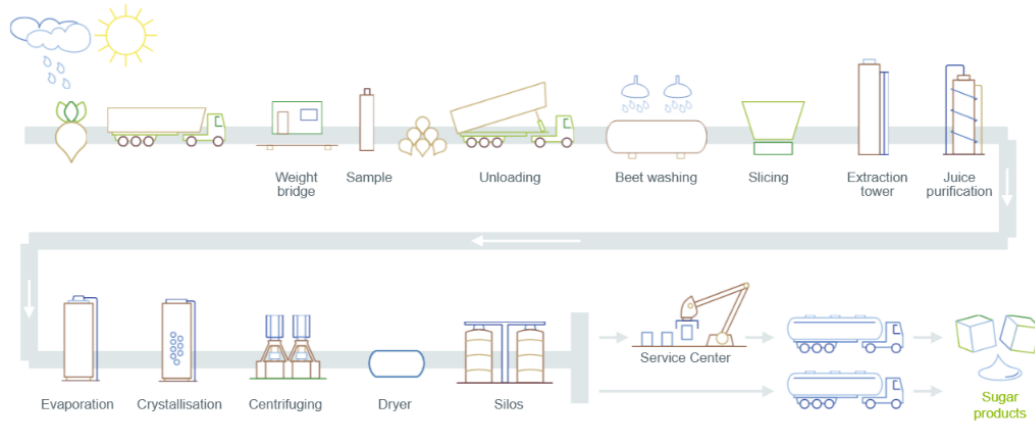


Figure 2.1. Schematic diagram of sugar production. Used with permission of Nordic Sugar AB.

2.2 Forward Osmosis

Forward osmosis (FO) is membrane-based separation technology which utilizes the osmotic pressure gradient as a driving force [13]. During the process, two solutions of different osmotic pressures are separated by a semipermeable hydrophilic membrane. Water moves from diluted feed solution (FS) to a more concentrated draw solution (DS) through the membrane due to the difference in osmotic pressure [13]. Suspended solids are rejected by the membrane, concentrating FS. The performance is evaluated by measuring the permeate flux:

$$J_w = A(\pi_{DS} - \pi_{FS}) \quad (1)$$

where J_w – water flux, A – membrane permeability coefficient, π_{DS} – osmotic pressure of the draw solution, π_{FS} – osmotic pressure of the feed solution.

The membranes utilized in FO are asymmetric, composed of a dense active layer (AL) and a porous support layer (SL) for mechanical stability, as shown in Figure 2.2 [14]. To minimize the mass transfer limitations, AL is fabricated to be as thin as possible [15]. Depending on the membrane orientation, there are two possible operational modes: active layer-facing-feed solution (AL-FS) and active layer-facing-draw solution (AL-DS) [14]. Studies have shown that the AL-FS mode has typically higher transmembrane flux compared to AL-DS mode due to concentration polarization [16].

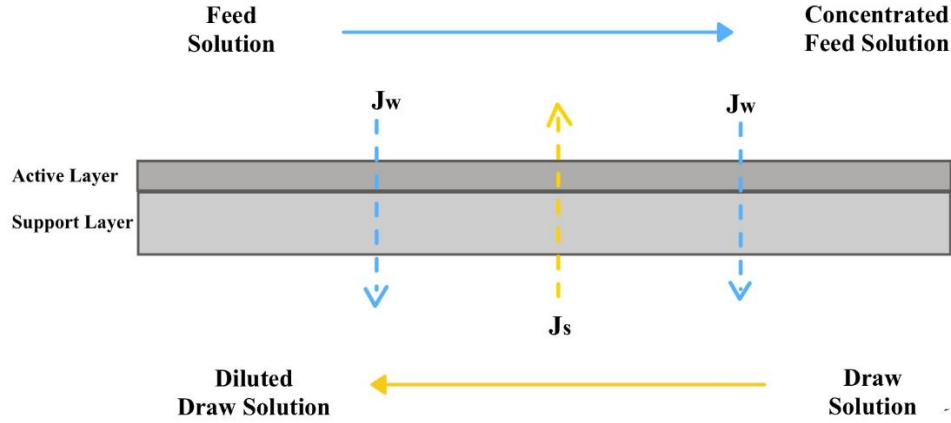


Figure 2.2. FO process in AL-FS mode. J_w – water flux, J_s – reverse solute flux.

2.3 Advantages of Forward Osmosis

Compared to conventional thermal processing and membrane technology, FO exhibits several advantages. It operates at ambient or cooler temperatures, which helps to preserve heat-sensitive bioactive compounds and does not affect color, taste, and aroma of food products [17]. For example, in the concentration study of natural pigments from rose petals, FO showed the lowest degradation of anthocyanin compared to thermal evaporation and freeze drying [18].

Moreover, FO has higher rejection of FS solute compared to reverse osmosis (RO). Xie et al. [19] observed higher rejection of bisphenol A in FO compared to RO at the same values of permeate flux. This was explained by retarded forward diffusion of feed solutes, where reverse salt flux blocks the adsorption and diffusion of feed solutes, leading to high rejection.

It is believed that FO is less prone to fouling due to absence of applied hydraulic pressure. However, this tendency is partly attributed to the low permeate fluxes in the FO processes [20]. The fouling in FO is a result of chemical and hydrodynamic interactions similar to RO, but the fouling layer is less compact which reduces its impact on process performance [21, 22]. Since FO is less prone to irreversible fouling, it can treat FS with high content of foulants and requires less frequent cleaning [22, 23].

Another common misconception about FO is that it requires less electrical energy than pressure-driven membrane processes due to the absence of externally applied hydraulic pressure. However, in most applications, the overall energy consumption of FO systems is comparable to conventional processes because an additional separation step is typically required to regenerate or recover the draw solutions [22]. In contrast, for applications such as juice concentration, where the draw solution does not need to be recovered, the advantage of lower energy consumption in FO can be more realistically realized.

2.4 Challenges of Forward Osmosis

Despite the aforementioned strengths of FO, there are various limitations associated with this technology that hinder its applicability. Acknowledgment and understanding of these challenges is necessary for further improvement.

2.4.1 Draw Solutions

The selection of DS plays a key role in development of the FO process, since the performance of FO is greatly dependent on osmotic pressure exerted by the DS [24]. Osmotic pressure is defined as the minimum pressure needed to prevent the inward flow of a solvent through a semipermeable membrane to a more concentrated solution [25], and it can be calculated using van't Hoff's equation. It is evident that the osmotic pressure is dependent on the solution's solubility:

$$\pi = iMRT = i \left(\frac{n}{V} \right) RT \quad (2)$$

where π – osmotic pressure, i – van't Hoff factor, M – molarity, n – solute moles, V – volume of the solution, R – gas constant, T – absolute temperature.

Aside from high osmotic pressure, there are other criteria for DS: nontoxicity, reasonable molecular size to minimize reverse salt flux (RSF), compatibility with FO membranes, low operational cost during recovery, and chemical stability [26]. The main challenge in selection of optimal DS lies in the conflicting nature of the requirements. The solution has to have strong affiliation between water molecules and solute; however, this strong affiliation becomes an obstacle during DS recovery [27].

In liquid food concentration, inorganic salts are the most employed DS due to their nontoxicity, low cost, and high osmotic pressure [28]. Sodium chloride was successfully used as DS in various concentration studies of pomegranate [29], grape [30], apple [31], beetroot, and pineapple juices [15]. CaCl_2 , KHCO_3 , MgCl_2 , NaHCO_3 were also used as draw solutes in FO [32]. Despite the fact that inorganic salts generate high permeate flux, they have a high back diffusion rate that affects the quality of the final product [28].

Another widely used group of DSs for liquid food concentration are simple carbohydrates. These DS do not affect the quality of the product, but have low water flux because of limitations of osmotic pressure [32]. Increase in concentration also increases the viscosity, which leads to concentration polarization [32]. To address these challenges, binary DS combining inorganic salts and carbohydrates were also tested for fruit juice concentration [26, 33].

Molasses, a by-product of sugar production, has also attracted attention as a potential DS for FO. It contains high concentrations of sugars, minerals, and organic compounds, which provides substantial osmotic pressure for the process. Molasses is also compositionally compatible with sugar streams, which reduces the concerns related to product contamination [9]. However, it presents several limitations. Its high viscosity reduces mass transfer and intensifies concentration polarization. The complex composition of molasses contributes to membrane fouling. Therefore, further investigations are required to evaluate its feasibility as an industrial DS and to optimize operating conditions.

2.4.2 Reverse Solute Flux

RSF is the permeation of draw solute into the FS through the membrane driven by concentration difference [34]. It occurs because of imperfect membrane selectivity and leads to various issues, such as loss of DS, decrease in the osmotic pressure difference, enhanced fouling, and increase in operational costs [35].

Leakage of DS to the feed side is dependent on the intrinsic parameters of the membrane and the solute characteristics [35]. Studies suggest that the use of multivalent ion DSs with lower

permeation rates might reduce RSF and consequently reduce membrane fouling [36, 37]. However, some multivalent ions such as calcium and magnesium interfere with foulants on the feed side, which, in turn, intensifies fouling [38].

Control and mitigation of RSF is crucial for FO processes. There are various strategies involving membrane modifications and selection of less permeable DS for the process [35].

2.4.3 Concentration Polarization

Concentration polarization (CP) is one of the greatest challenges in FO technologies [39]. It refers to the accumulation or depletion of solutes at the membrane's surface or within its structure, which affects the solute concentration gradient and reduces the effective osmotic pressure driving of the process [39]. This phenomenon leads to a reduction in water mass transfer efficiency and permselectivity of the membrane [39].

CP is categorized into two main types: External Concentration Polarization (ECP), which occurs on the outer surface of the membrane, and the Internal Concentration Polarization (ICP), which takes place within the porous support layer of the membrane [28, 39].

ECP occurs because the solute concentration at the membrane-solution interface becomes different from the bulk solution as water permeates through the selective layer [28, 39]. Concentrative ECP happens on the feed side when the membrane rejects solutes and allows water to pass, causing a solute build-up at the surface [28, 39]. On the other hand, dilutive ECP occurs on the draw solution side when incoming pure water flux dilutes the draw solute at the membrane interface [39]. Even though ECP can be partially controlled by increasing cross-flow velocity, enhancing turbulence and using feed spacers to reduce the boundary layer, it remains a significant factor in processes that use highly concentrated draw solutions at low flow rates [28, 39, 40].

ICP is an intrinsic characteristic of asymmetric membranes that occurs within the porous support layer [28, 39]. Unlike ECP, ICP cannot be mitigated by simple hydrodynamic adjustments [39]. The type and severity of CP are heavily influenced by membrane orientation [39]. Dilutive ICP is the predominant mechanism when the support layer faces the draw solution, as water permeates through the selective layer to the draw side, it locally dilutes the draw solution. Porous structure restricts the back diffusion of solutes from the bulk solution, leading to decrease in osmotic driving force [28, 39]. Concentrative ICP takes place when the support layer faces the feed solution, allowing feed solutes to enter the pores along with water and become trapped and concentrated at the internal side of the active layer [28, 39]. Figure 2.3 demonstrates the concentration profiles of FO in AL-FS and AL-DS modes.

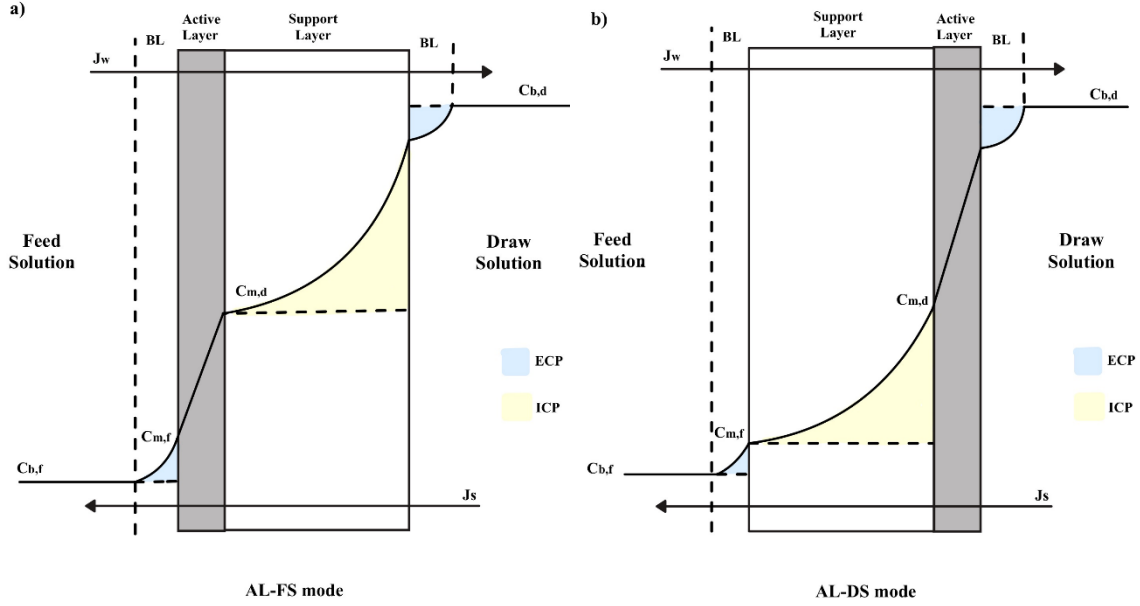


Figure 2.3. Concentration profile of FO process in a) AL-FS mode; b) AL-DS mode. J_w – water flux, J_s – reverse solute flux, BL – boundary layer, $C_{b,f}$ – concentration of solute in feed bulk, $C_{m,f}$ – concentration of solute at feed-membrane interface, $C_{m,d}$ – concentration of draw solute at active layer-support layer interface, $C_{b,d}$ – concentration of draw solute in bulk.

The severity of ICP is mathematically determined by the structural parameter (S), which accounts for the thickness, tortuosity, and porosity of the support layer [39]. A membrane with high structural parameter value will experience more severe ICP [39]. Another critical factor is the solute resistivity (K), which is influenced by the diffusion coefficient of the solute [28], [40]. Temperature also has a significant effect, as higher temperatures reduce solution viscosity and increase solute diffusivity, diminishing the effect of ICP and enhances overall flux [41].

A unique property of FO is the ICP self-compensation effect, which describes a stabilizing relationship between fouling and CP. If foulants accumulate on the membrane surface and increase hydraulic resistance, the resulting decrease in water flux reduces the severity of ICP [39]. This reduction in ICP increases the osmotic gradient across the fouled layer, which moderately compensates for the loss of the driving force caused by fouling [39]. It was reported that this effect is more pronounced in the AL-FS membrane orientation [42]. AL-DS mode is highly susceptible to complex internal fouling and clogging, which makes AL-FS mode more favorable in FO processes [42].

2.4.4 Fouling

Fouling is defined as accumulation of particles or solutes on the surface or within the membrane structure, leading to reduction in performance [43, 44]. Even though FO is less susceptible to fouling and has higher fouling reversibility than pressure-driven processes, it remains a significant operational challenge.

Based on foulant nature, this phenomenon is broadly categorized into four types: biofouling, organic fouling, scaling, and colloidal fouling [43-46]. Biofouling involves the attachment of microorganisms and the subsequent growth of a biofilm enclosed in an extracellular polymer matrix [43-44]. Organic fouling is caused by macromolecules like humic acids, proteins, polysaccharides, the rate of organic fouling is governed by chemical and hydrodynamic interactions

between foulants and the membrane surface. Inorganic scaling occurs through the crystallization of soluble mineral salts, such as calcium carbonate, gypsum, or silica, which precipitate on the membrane surface when their solubility limits are exceeded. Colloidal fouling results from the deposition of fine particles (1 to 1000 nm) like clay, silica nanoparticles, or silt, that form a cake layer at the membrane surface [44].

The location of fouling is heavily dependent on the membrane orientation. In the AL-FS mode, foulants form an external cake layer on the dense surface, which is more reversible through simple cleaning [43-45]. On the other hand, in the AL-DS mode, foulants can penetrate the porous support layer to cause internal fouling or pore clogging, which is significantly harder to remove [43]. A critical mechanism unique to FO is cake-enhanced osmotic pressure (CEOP), where a developed fouling layer traps draw solutes that have migrated via reverse salt diffusion. This leads to an elevated osmotic pressure at the membrane surface, which reduces the driving force more severely than the resistance of the foulant layer itself [43]. Furthermore, the critical flux concept suggests that significant fouling only becomes noticeable once the permeate flux exceeds a specific threshold value [43, 46].

Due to the reduced applied hydraulic pressure in FO, the fouling layer is relatively loose which makes it generally more reversible. Physical cleaning methods, such as hydraulic flushing, air scouring, and osmotic backwashing are often sufficient to restore flux [43, 44, 46]. However, when treating complex feeds or in case of intense fouling, physical cleaning may be ineffective. Studies have employed different chemical cleaning agents such as chlorine, alkali and acid solutions [43].

2.5 Application of Forward Osmosis in Sugar Production

The concentration of thin juice via evaporation is one of the most energy-intensive stages in beet sugar production [47]. FO offers an energy-efficient alternative for partial dewatering prior to thermal evaporation stage. The byproduct of sugar production, molasses is suited as a draw solution due to its high osmotic pressure, with 82 °Brix molasses yielding 317 bar osmotic pressure [9].

The application of FO provides substantial benefits over pressure-driven membrane processes such as reverse osmosis, which requires high operating pressures to overcome the osmotic pressure of feed juice [48]. FO operates at ambient temperatures with limited hydraulic pressures, which ensures that foulants are not forced against the membrane surface, which results in significantly less severe fouling compared to RO. Studies have shown that the concentration of liquid foods by RO is limited up to 25-30 °Brix [48], while FO is able to concentrate liquid foods to 60 °Brix [29]. FO also preserves the sensory and nutritional integrity of the juice, as the low temperature operation prevents the degradation of heat-sensitive compounds.

The resulting diluted molasses from the concentration process can be used for bioethanol production. Molasses naturally contains over 50% fermentable sugars, but its high initial concentration requires substantial dilution with freshwater before it can be effectively fermented by microbes [9, 47]. Dilution of molasses as a result of FO can significantly reduce the need for freshwater in this process. Industrial plant trials have demonstrated that this integrated process can achieve a fermentation efficiency of 90.43%, conserving 9.14 liters of fresh water per every liter of ethanol generated [9]. This configuration facilitates “no waste” production in the sugar industry.

3 Methodology

3.1 Materials

The samples of thin juice, thick juice, and molasses were provided by Nordic Sugar (Örtofta, Sweden). Thin juice was collected before the evaporation step and was softened using ion exchange resin before sampling. Thin juice samples were stored in a frozen state and thawed before each experiment.

When the supply of thin juice from the 2025 campaign was exhausted, thin juice from the 2024 production campaign was used. This juice was stored in a freezer and has undergone multiple freeze-thaw cycles during storage and use. In addition, thin juice from the 2024 campaign that was continuously stored in a freezer room was utilized.

For the long-term concentration study, thin juice was prepared from thick juice by dilution followed by pH adjustment to match the characteristics of industrial thin juice.

3.2 Membranes

Flat sheet cellulose triacetate membranes (FTS H₂O) were used for all parametric studies. The membranes were soaked for 24 hours in deionized (DI) water prior to use according to manufacturer's recommendations.

In addition, reverse osmosis RO98 pHt (Alfa Laval) and nanofiltration NP030 (Microdyn Nadir) membranes were tested under selected conditions for comparative analysis.

3.3 Forward Osmosis Setup

Forward osmosis experiments were carried out using plate-and-frame SEPA module (Sterlitech) with an effective membrane area of 140 cm². All experiments were performed in FO mode, with the membrane active layer facing the feed solution. The system was operated in a closed-loop recirculation configuration for both feed and draw solutions.

The forward osmosis setup is presented in Figure 3.1. Two peristaltic pumps were used to circulate the feed and draw solutions through the membrane module. The mass of solutions was continuously measured using digital balance, allowing the determination of water flux from the rate of mass change over time, assuming a solution density of 1 kg·L⁻¹.

A conductivity meter was used to measure the conductivity of solutions during the process. Pressure gauges were installed on both the feed and draw sides to ensure no hydraulic pressure difference across the membrane.

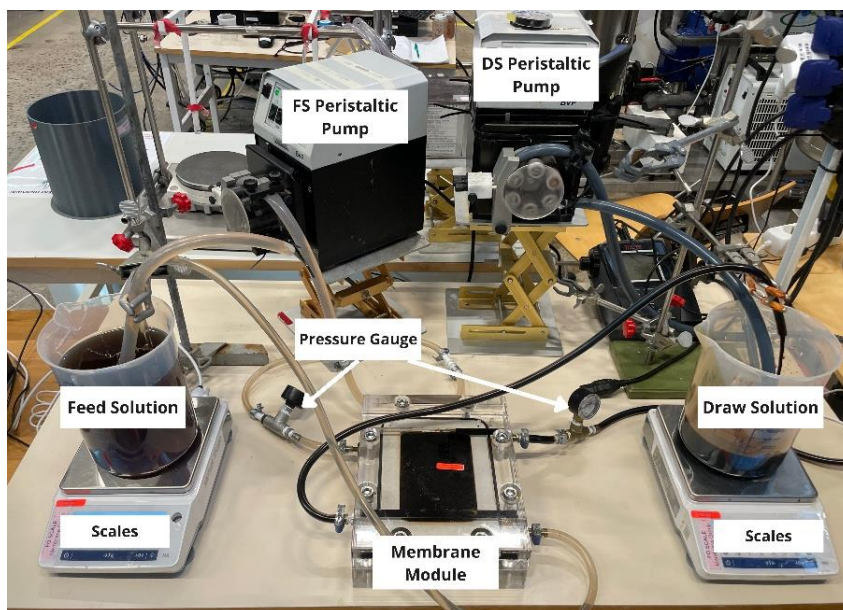


Figure 3.1. A picture of the forward osmosis setup.

The peristaltic pumps were calibrated to relate revolutions per minute (RPM) to the volumetric flowrate. The feed solution pump was calibrated using DI water, while the draw solution pump was calibrated using both DI water and molasses solutions with concentrations ranging from 30 to 60 %RDS. The flow rate was calculated by measuring the change in mass with a digital balance; a hydrometer was used to measure the density of molasses.

After each experimental run, the system was flushed with DI water for 30 minutes. A pure water flux (PWF) test was performed after each experiment, where 1 kg of DI water was circulated on the feed side and 1 kg of 0.5 M NaCl solution was circulated on the draw side at a flow rate of $60 \text{ L} \cdot \text{h}^{-1}$. The mass of the feed solution was measured with 2-second intervals for 20 minutes. The membrane was replaced if PWF decreased by more than 15% after filtration.

3.4 Parametric Studies

Two types of draw solutions, sodium chloride and molasses, were investigated in this study. A series of parametric experiments was conducted to evaluate the influence of operating conditions on forward osmosis performance. Each experimental run was carried out for 3.5 hours.

3.4.1 Draw Solution Flow Rate

The effect of draw solution flow rate was examined by varying flow rate between 55 and $75 \text{ L} \cdot \text{h}^{-1}$ while maintaining the feed flow rate at $65 \text{ L} \cdot \text{h}^{-1}$. 2 kg of thin juice was used as a feed solution and 1 kg of draw solution (1.5 M NaCl, 40 %RDS molasses) was used. Tests were conducted at ambient temperature.

3.4.2 Draw Solution Concentration

The influence of draw solution concentration was studied by selecting concentrations of sodium chloride and molasses that provided comparable osmotic pressures. The osmotic pressure of molasses was estimated indirectly from water activity, which is correlated with refractometric dry solids in sucrose-dominated solutions [49]. Equivalent concentration of sodium chloride was calculated using van 't Hoff equation. All experiments were carried out with feed and draw

flow rates maintained at $65 \text{ L}\cdot\text{h}^{-1}$, using 2 kg of thin juice as feed and 1 kg of draw solution at ambient temperature conditions.

Table 3.1. Concentrations of sodium chloride and molasses used in the parametric study and corresponding osmotic pressure values.

Osmotic pressure (bar)	NaCl concentration (M)	Molasses concentration (%RDS)
54	1.1	30
74	1.5	40
104	2.1	50
158	3.2	60

3.4.3 Temperature

The effect of temperature was investigated using molasses as feed solution. Prior to each experiment, feed and draw solutions were heated in a water bath to the desired temperature. During operation, the temperature of the draw solution was maintained using a hotplate and was monitored continuously using a thermometer. Experiments were conducted at constant flow rate of $65 \text{ L}\cdot\text{h}^{-1}$ for both streams, using 2 kg of thin juice and 1 kg of 60 %RDS molasses. The selected temperature range was limited by the operational range of the membrane reported by the manufacturer.

3.4.4 Feed-to-draw Ratio

To examine the effect of feed-to-draw ratio on the performance, the mass of the draw solution was varied while keeping the feed mass constant. Experiments were carried out with 2 kg of thin juice and 1 kg, 0.5 kg, 0.25 kg of 60 %RDS molasses, with both flow rates maintained at $65 \text{ L}\cdot\text{h}^{-1}$.

3.4.5 Concentration Study

A long-term concentration study was performed to evaluate the system performance over extended operation. 3.5 kg of thin juice and 1.75 kg of 60 %RDS molasses were used as feed and draw solutions, respectively. The system was operated at 45°C for 24 hours. FS flow rate was $65 \text{ L}\cdot\text{h}^{-1}$ and DS flow rate was $55 \text{ L}\cdot\text{h}^{-1}$. The mass of the feed solution was recorded at 10-second intervals throughout the experiment.

3.4.6 Flux Calculation

The water flux was determined from the change in feed solution mass over time. For all parametric studies, the mass was recorded at 2-second intervals. To reduce noise and short-term fluctuations in the measurements, the recorded mass data were averaged over 5-minute intervals, and flux was calculated from the difference between consecutive averaged values.

The calculated flux was normalized by the effective membrane area and expressed in $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$. Data preprocessing was performed prior to calculations, where outliers and mass spikes caused by sample withdrawal or accidental disturbances of the feed container were identified and removed.

3.5 Analysis

Feed and draw solution samples were collected before and after each experiment. All analytical procedures were conducted in accordance with methods provided by Nordic Sugar and followed

the standards of the International Commission for Uniform Methods of Sugar Analysis (ICUMSA).

3.5.1 Refractometric Dry Substance

Refractometric dry substance of thin juice and molasses was measured using sucrose refractometer (HI 96801, Hanna Instruments). DI water was used to zero the instrument. Samples were placed on the prism and the RDS value was recorded directly.

3.5.2 Color Analysis

Color measurements were performed on thin juice samples following pH adjustment and filtration. 50 mL of thin juice was pH adjusted to pH 7 using 0.1 M hydrochloric acid and 0.1 M sodium hydroxide. The pH-adjusted juice was subsequently filtered using a 0.2 µm membrane filter (Thermo Fisher).

The filtered sample was analyzed using a UV-vis spectrophotometer at wavelength of 420 nm. The RDS of the pH-adjusted sample was measured. Color value was calculated using following formula:

$$Color = \frac{100000 * (A_{sample} - A_{blank})}{d * RDS * (0.9857 + 0.00488 * RDS)} \quad (3)$$

where *Color* – color value, *A_{sample}* – sample absorbance, *A_{blank}* – DI water absorbance, *d* – length of the measuring cell, *RDS* – refractometric dry substance of the sample after pH adjustment.

3.5.3 Polarimetric Sucrose Content and Purity

Polarimetric sucrose content was determined using clarified samples of thin juice and molasses. 13 g of thin juice or 6.5 g of molasses was weighed out in 100 mL volumetric flask. In case of molasses, clarification was performed by adding 2 mL of Carrez 1 reagent (zinc acetate, acetic acid) and 2 mL of Carrez 2 reagent (potassium hexacyanoferrate). Both reagents were supplied by Nordic Sugar.

The solutions were then diluted with DI water and filtered through filter paper. The samples were analyzed using a polarimeter at a wavelength of 587 nm. Purity was calculated as the ratio of polarimetric sucrose content to the refractometric dry substance.

4 Results and Discussion

4.1 The Effect of Draw Solution Flow Rate on FO Performance

The FO concentration of thin juice was carried out using sodium chloride as the DS, serving as a benchmark for flux evaluation. The process performance was investigated at different DS flow rates, and the corresponding flux profiles are presented in Figure 4.1.

All flux curves exhibit a consistent trend: an initial rapid decline followed by a more gradual decrease, eventually reaching stabilization. This behavior has also been widely reported in FO studies involving fruit juice concentration [30, 50]. The sharp initial flux drop can be attributed to the combined effects of concentration polarization and membrane fouling due to the deposition of polysaccharides on the membrane surface [30].

An increase in DS flow rate clearly increased the transmembrane flux. This can be explained by the reduction of ECP at high cross-flow velocities. Even though ICP is unaffected by cross-flow velocity, dilutive ECP on the DS side has a significant impact on the process. Increased hydrodynamic conditions improve mass transfer at the membrane surface, therefore maintaining a higher osmotic pressure difference between solutions and reducing the extent of dilutive ECP [51].

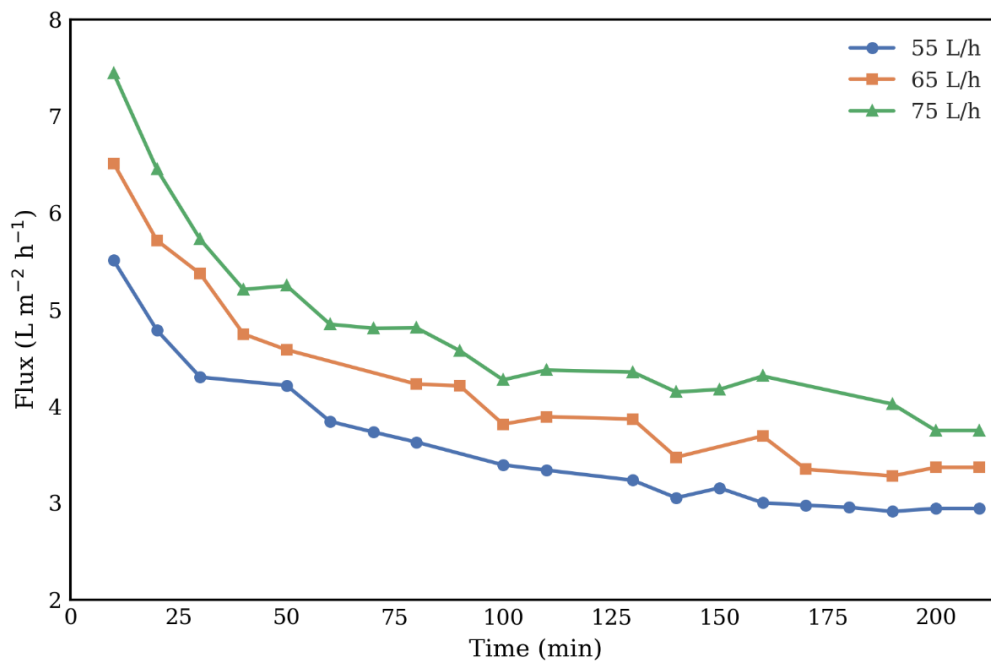


Figure 4.1. Water flux as a function of time at different values of sodium chloride flow rate. The FS flow rate was $65 \text{ L} \cdot \text{h}^{-1}$ for all trials.

During the experimental runs, the refractometric dry substance (%RDS) of FS and conductivity of both FS and DS were measured at 30-minute intervals. As shown in Table 4.1, the highest DS flow rate of $75 \text{ L} \cdot \text{h}^{-1}$ resulted in the greatest increase in thin juice concentration, from 15.8 to 17.7 %RDS, which correlates with the highest observed flux. Similarly, this trial exhibited the largest decrease in DS conductivity of 14.1 mS/cm due to significant dilution caused by water permeation.

The conductivity of FS showed a slight increase across all experiments, which can be attributed to water removal and RSF. A noticeable decrease in both conductivity and %RDS was observed between 0 and 30 minutes. This can be explained by the presence of residual DI water within the system at the start of the experiment, which diluted both solutions.

Table 4.1. Results from conductivity and %RDS measurements of feed and draw solutions during the experimental trial at different values of DS flow rate.

Time (min)	55 L·h ⁻¹			65 L·h ⁻¹			75 L·h ⁻¹		
	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)
0	17.4	3.605	109.3	17.0	3.605	121.4	15.8	3.510	118.6
30	16.4	3.535	108.5	17.3	3.451	114.7	16.1	3.454	117.0
60	17.8	3.560	107.1	17.6	3.476	113.4	16.3	3.475	113.4
90	17.9	3.580	105.5	17.9	3.506	111.6	16.6	3.504	112.1
120	18.2	3.628	104.2	18.0	3.604	110.5	16.8	3.526	110.6
150	18.3	3.625	103.2	18.2	3.610	107.9	17.1	3.545	107.2
180	18.5	3.636	102.1	18.4	3.621	105.3	17.3	3.579	105.5
210	18.8	3.687	101.5	18.7	3.635	104.2	17.7	3.581	104.5
Δ	1.4	0.082	-7.8	1.7	0.030	-17.2	1.9	0.071	-14.1

Thin juice samples were collected before and after the FO process for analysis, see Table 4.2. A decrease in pH was observed in all cases after FO treatment, suggesting that fermentation and production of lactic acid was taking place during the concentration process. Thin juice is rich in sugars, making it susceptible to the growth of microorganisms, particularly during long experimental runs at moderate temperatures.

Table 4.2. Results from analysis of thin juice before and after forward osmosis at different values of DS flow rate.

	55 L·h ⁻¹			65 L·h ⁻¹			75 L·h ⁻¹		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
pH	8.57	8.16	-0.41	7.90	7.49	-0.41	8.89	8.62	-0.27
%RDS	17.4	18.8	1.4	17	18.7	1.7	15.8	17.7	1.9
Color (IU)	1962	2043	81	1975	2009	34	1960	2046	86
Purity (%)	92.1	93.2	1.1	92.8	93.4	0.6	90.8	91.4	0.6

The effect of DS flow rate was further investigated under identical operating conditions using 40 %RDS molasses as DS. The resulting flux profiles (Figure 4.2) exhibit similar trends to those observed for sodium chloride.

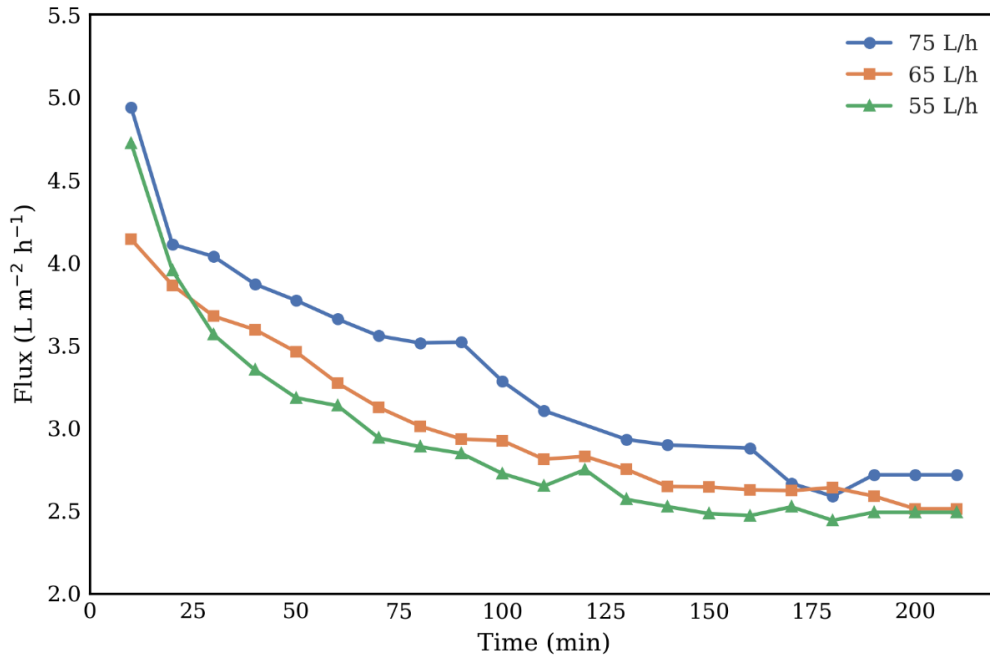


Figure 4.2. Water flux as a function of time at different values of molasses flow rate. The FS flow rate was $65 \text{ L} \cdot \text{h}^{-1}$ for all trials.

A comparison of all trials (Figure 4.3) reveals that despite maintaining similar osmotic pressures of DS, the transmembrane flux obtained with molasses is consistently lower than with sodium chloride across all flow rates. This difference is related to the intrinsic properties of molasses. Unlike sodium chloride, which is a fully dissociated electrolyte with high diffusivity, molasses is a complex mixture of carbohydrates and inorganic salts. The non-electrolyte nature and relatively low diffusivity of sugar molecules significantly limit their transport within the membrane structure [26]. They interfere with mass transfer by inhibiting ion mobility and partially blocking the membrane pores during permeation. Furthermore, molasses solutions exhibit substantially higher viscosity compared to salt solutions, which increases the thickness of the concentration boundary layer and reduces mass transfer coefficient [26]. As a result, both ECP and ICP are exacerbated, leading to lower water flux.

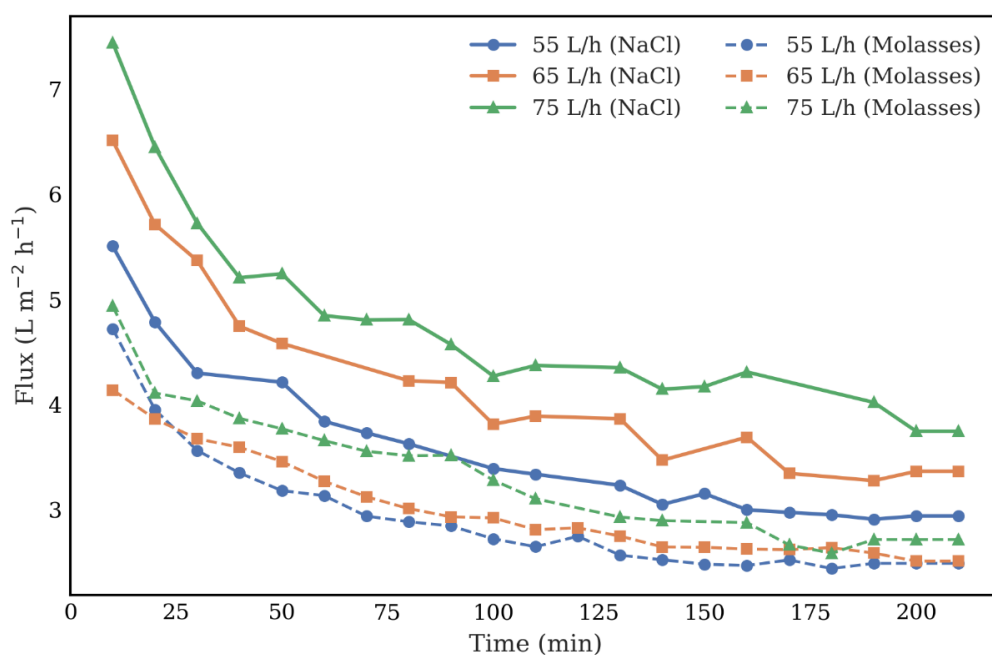


Figure 4.3. Water flux as a function of time at different values of sodium chloride and molasses flow rate.

The analysis of FS and DS properties is consistent with the flux behavior, see Table 4.3. At a flow rate of 75 L·h⁻¹, the thin juice exhibits the highest increase in %RDS and color, corresponding to the highest water flux of 3.3 LMH. The purity of both FS and DS remains relatively stable throughout the experiments, indicating that sucrose degradation is minimal.

Table 4.3. Results from analysis of thin juice before and after forward osmosis at different values of DS flow rate.

Thin Juice									
	55 L·h ⁻¹			65 L·h ⁻¹			75 L·h ⁻¹		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	18.1	19.2	1.1	17.6	18.8	1.2	17.4	18.7	1.3
pH	9.24	8.83	-0.41	9.25	8.85	-0.40	8.95	8.61	-0.34
σ (mS/cm)	3.358	3.501	0.143	3.518	3.608	0.090	3.608	3.652	0.044
Color (IU)	2269	2339	70	1972	2050	78	2012	2095	83
Purity (%)	91.3	90.6	-0.7	90.4	89.1	-1.3	91.3	92.0	0.7
Molasses									
	55 L·h ⁻¹			65 L·h ⁻¹			75 L·h ⁻¹		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	39.2	35.4	-3.8	39.6	35.8	-3.8	39.2	35.6	-3.6
pH	7.00	6.85	-0.15	6.71	7.07	0.36	6.72	7.03	0.31
σ (mS/cm)	18.32	19.74	1.42	18.06	19.69	1.63	18.36	19.79	1.43
Purity (%)	57.9	57.3	-0.6	55.8	55.5	-0.3	56.7	56.6	-0.1

4.2 The Effect of Draw Solution Concentration on FO Performance

The concentration of sodium chloride was varied from 1.1 M to 3.2 M, and the corresponding flux profiles are presented in Figure 4.4. As expected, increasing DS concentration enhances the osmotic pressure difference across the membrane, leads to an increase in the driving force for water transport and results in higher transmembrane flux.

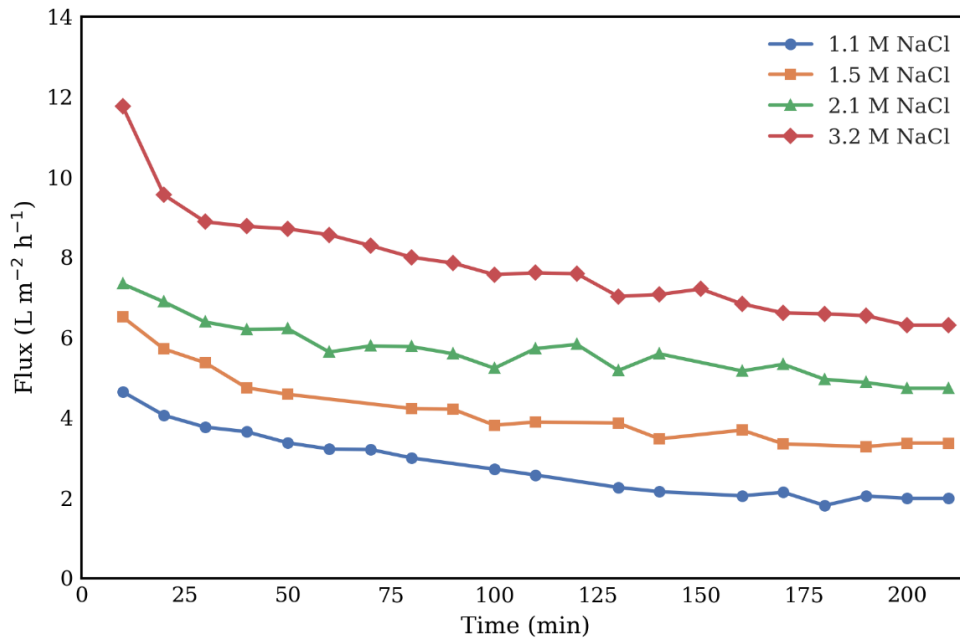


Figure 4.4. Water flux as a function of time at different values of sodium chloride concentration.

This trend is also reflected in the composition of FS. As shown in Table 4.4, the increase in %RDS becomes more pronounced with higher DS concentration. At the highest concentration, significant dilution of DS and increase in FS conductivity was observed. This behavior suggests enhanced RSF, which is more prominent at higher osmotic gradients.

Table 4.4. Results from conductivity and %RDS measurements of feed and draw solutions during the experimental trial at different values of DS concentration.

Time (min)	1.1 M			1.5 M			2.1 M			3.2 M		
	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)	%RDS	σ_{FS} (mS/cm)	σ_{DS} (mS/cm)
0	17.5	3.450	92.30	17.0	3.605	121.4	18.0	3.510	156.9	14.1	3.194	201.8
30	17.1	3.444	90.07	17.3	3.451	114.7	18.4	3.547	150.4	14.3	3.248	197.0
60	17.7	3.499	89.23	17.6	3.476	113.4	18.7	3.607	147.7	14.8	3.304	191.4
90	17.9	3.513	88.32	18	3.506	111.6	19.0	3.638	144.3	15.1	3.374	186.0
120	18.1	3.538	87.47	18.2	3.604	110.5	19.2	3.675	141.1	15.6	3.440	181.3
150	18.2	3.562	86.39	18.4	3.610	107.9	19.5	3.699	138.7	15.9	3.499	176.4
180	18.4	3.586	86.26	18.4	3.621	105.3	20.1	3.701	136.2	16.2	3.571	172.5
210	18.4	3.564	85.39	18.7	3.635	104.2	20.3	3.766	132.3	17.0	3.616	168.9
Δ	0.9	0.114	-6.91	1.7	0.184	-17.2	2.3	0.256	-24.6	2.9	0.422	-32.9

Changes in FS are consistent with the flux behavior. Increasing DS concentration leads to greater color formation likely due to intensified concentration, as shown in Table 4.5.

Table 4.5. Results from analysis of thin juice before and after forward osmosis at different values of DS concentration.

	1.1 M			1.5 M			2.1 M			3.2 M		
	Start	End	Δ	Start	End	Δ	Start	End	Δ	Start	End	Δ
pH	8.73	8.39	-0.34	7.9	7.49	-0.41	8.89	8.65	-0.24	8.89	8.67	-0.22
%RDS	17.5	18.4	0.9	17.0	18.7	1.7	18.0	20.3	2.3	14.1	17.0	2.9
Color (IU)	2034	2059	25	1975	2008	33	1918	1959	41	2068	2132	64
Purity (%)	91.8	91.0	-0.8	92.8	93.4	0.6	94.8	93.4	-1.4	90.4	91.4	1.0

A similar trend is observed when molasses is used as DS, where the highest flux of 4.2 LMH is obtained at 60 %RDS. However, the relationship between osmotic pressure and flux is clearly nonlinear. Although the osmotic pressure at 60%RDS is approximately three times higher than at 30 %RDS, the corresponding flux increase is not proportional. This indicates that the effective osmotic driving force is reduced at higher DS concentrations. The main reason for this behavior is intensified CP occurring at high DS concentrations. As the molasses concentration increases, a thicker concentration boundary layer (BL) develops near the membrane surface on the DS side. This BL hinders mass transfer and lowers the effective osmotic pressure at the membrane interface compared to the bulk solution [51, 52]. As a result, the increase in water flux becomes smaller than expected despite the increase in DS concentration.

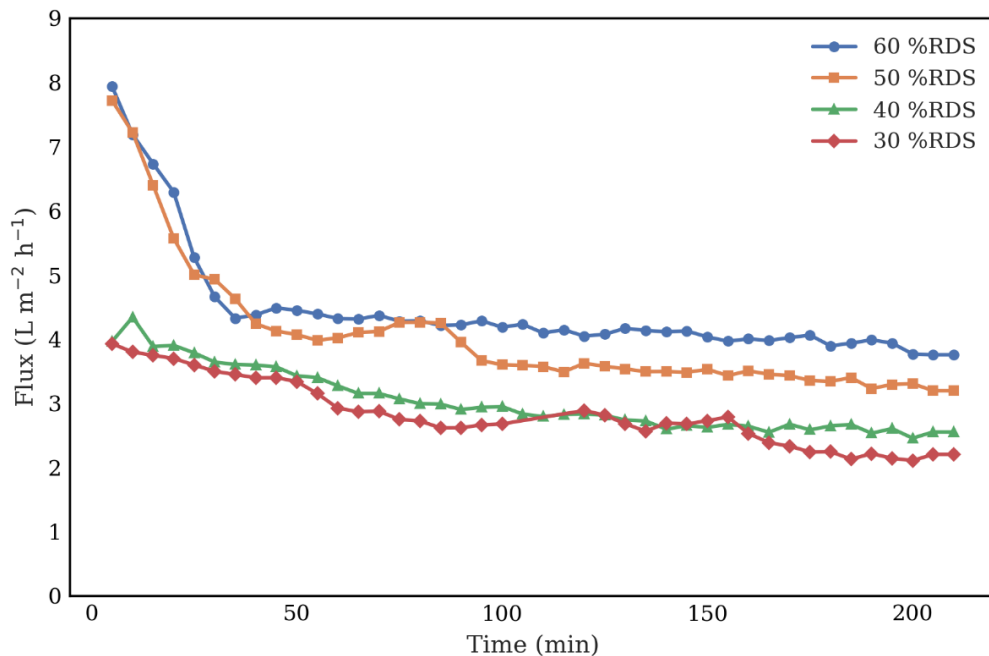


Figure 4.5. Water flux as a function of time at different values of molasses concentration.

Figure 4.6 compares the performance of both DS, it is evident that molasses consistently produces lower flux than sodium chloride due to higher viscosity, lower diffusivity and complex composition.

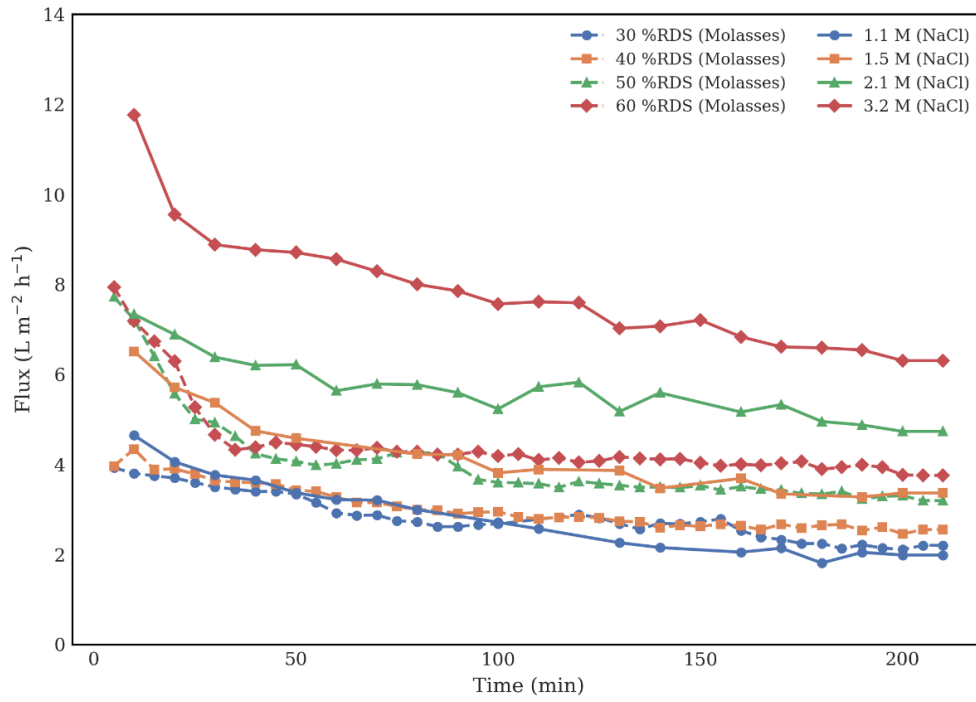


Figure 4.6. Water flux as a function of time at different values of sodium chloride and molasses concentration. Curves sharing the same marker correspond to equal osmotic pressures.

The analysis of FS and DS provides further insight into these observations. As presented in Table 4.6, the %RDS and color formation of thin juice increases with DS concentration. For molasses, the opposite trend in %RDS is observed, reflecting dilution due to water permeation. Purity remains relatively stable across all trials.

Table 4.6. Results from analysis of thin juice and molasses before and after forward osmosis at different values of DS concentration.

Thin juice												
	30 %RDS*			40 %RDS			50 %RDS*			60 %RDS		
	Start	End	Δ	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	17.7	18.8	1.1	17.6	18.8	1.2	18.3	19.8	1.5	17.9	20.0	2.1
pH	6.76	6.38	-0.38	9.25	8.85	-0.4	6.92	6.34	-0.58	8.99	9.02	0.03
σ (mS/cm)	3.328	3.383	0.055	3.518	3.608	0.09	3.131	3.318	0.187	3.442	3.582	0.14
Color (IU)	1518	1573	55	1972	2050	78	1340	1444	104	2341	2453	112
Purity (%)	91.8	91.7	-0.1	90.4	89.1	-1.3	93.3	93.3	0.05	92.9	92.4	-0.5
Molasses												
	30 %RDS			40 %RDS			50 %RDS			60 %RDS		
	Start	End	Δ	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	29.9	27.7	-2.2	39.6	35.8	-3.8	49.3	43.9	-5.4	59.6	51.8	-7.8
pH	8.62	7.93	-0.69	6.71	7.07	0.36	8.52	8.45	-0.07	8.23	8.11	-0.12
σ (mS/cm)	19.83	20.03	0.2	18.06	19.69	1.63	12.81	16.27	3.56	6.737	12.57	5.833
Purity (%)	61.1	61.5	0.4	55.8	55.5	-0.3	61.2	61.1	-0.1	59.1	58.3	-0.8

* Thin juice from the 2024 campaign; stored frozen and repeatedly thawed/refrozen.

4.3 The Effect of Temperature on FO Performance

Subsequent experiments were carried out using molasses as DS to evaluate the effects of temperature on FO performance. The flux profiles presented in Figure 4.7 show a clear increase in water flux with rising temperature from 25 to 45°C. This can be attributed to improved mass transport properties at elevated temperatures. Specifically, higher temperature reduces the viscosity of both FS and DS, facilitating water transport across the membrane. Moreover, the diffusivity of solutes increases, which improves mass transfer and reduces the effect of ICP and ECP [5, 53]. Increased temperature also enhances the water permeability of the membrane, contributing to higher flux [53].

Another important observation is the difference in flux decline over time. At ambient temperature, the flux decreases more sharply, whereas at 45°C the decline is more gradual. This behavior can be explained by the reduced severity of CP at higher temperatures. Improved diffusivity and lower viscosity lead to more efficient diffusion of solutes from the surface of the membrane, minimizing concentration buildup and contributing to a more stable flux over time.

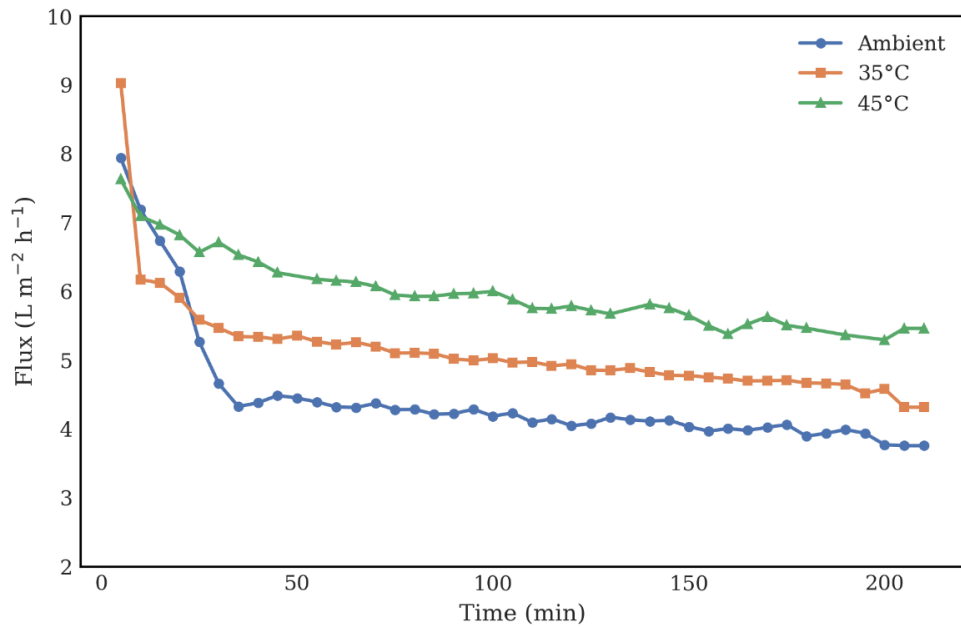


Figure 4.7. Water flux as a function of time at different values of temperature.

As shown in Table 4.7, the increase in %RDS and color formation is more pronounced with temperature, confirming enhanced water removal. Conductivity changes are moderate across all temperatures, indicating limited solute leakage. For molasses, decrease in %RDS becomes more significant with increasing temperature. A more pronounced decrease in pH is also observed at higher temperatures. Even though purity remains largely unaffected.

Table 4.7. Results from analysis of thin juice and molasses before and after forward osmosis at different operational temperatures.

Thin Juice									
	Ambient			35°C*			45°C*		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	17.9	20.0	2.1	16.9	19.5	2.6	16.4	19.5	3.1
pH	8.99	9.02	0.03	9.73	9.62	-0.11	9.11	9.03	-0.08
σ (mS/cm)	3.442	3.582	0.14	2.611	2.657	0.046	2.859	2.985	0.126
Color (IU)	2341	2453	112	1500	1616	116	1944	2083	139
Purity (%)	92.9	92.4	-0.5	93.0	93.1	0.1	93.0	93.1	0.1
Molasses									
	Ambient			35°C			45°C		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	59.6	51.8	-7.8	58.5	49.8	-8.7	59.4	49.6	-9.8
pH	8.23	8.11	-0.12	8.72	8.27	-0.45	8.89	7.40	-1.49
σ (mS/cm)	6.737	12.57	5.833	9.786	16.38	6.594	10.24	17.48	7.24
Purity (%)	59.1	58.3	-0.8	63.1	63.05	-0.05	62.7	60.1	-2.6

* Thin juice from the 2024 campaign; continuously stored in the freezer conditions.

4.4 The Effect of Feed-to-draw Ratio on FO Performance

To evaluate the feasibility of reducing the amount of molasses used in the concentration process, experiments were conducted at different feed-to-draw (F/D) ratios of 2, 4, 8 while keeping the FS mass constant and decreasing the amount of DS. The resulting flux profiles (Figure 4.8) show the expected trend: increasing the F/D ratio leads to a decrease in flux. This behavior is connected to the smaller volume of DS available at higher ratios, which becomes diluted more rapidly during operation. As dilution progresses, the osmotic driving force is reduced, leading to a decline in flux.

In the case of the F/D = 8 experiment, an unstable flux profile is observed during the first 90 minutes. This instability is caused by the very low initial volume of DS, which resulted in incomplete filling of the system. Bubbles of molasses were created on the surface of DS in the container, and they were circulated through the system. This leads to irregular contact between the membrane and DS, reducing the effective mass transfer area. As the process continues, water permeation increases the volume of DS, restoring continuous liquid contact with the membrane and resulting in a more stable flux profile.

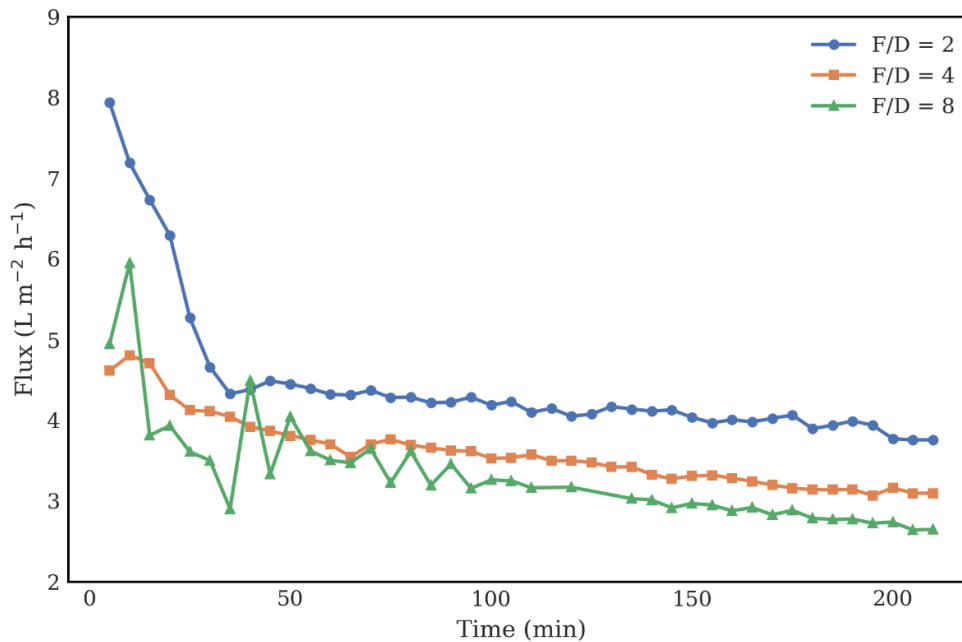


Figure 4.8. Water flux as a function of time at different values of feed-to-draw ratio.

Faster dilution of molasses at high F/D ratio is also evident in analysis of samples, as shown in Table 4.8. This is accompanied by a substantial increase in conductivity due to the smaller volume of DS.

Table 4.8. Results from analysis of thin juice and molasses before and after forward osmosis at different F/D ratios.

Thin Juice									
	F/D = 2			F/D = 4*			F/D = 8**		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	17.9	20.0	2.1	17.9	19.8	1.9	16.7	18.6	1.9
pH	8.99	9.02	0.03	5.53	5.48	-0.05	9.41	9.65	0.24
σ (mS/cm)	3.442	3.582	0.14	3.441	3.476	0.035	2.400	2.518	0.118
Color (IU)	2341	2453	112	- ***	- ***	- ***	1560	1647	87
Purity (%)	92.9	92.4	-0.5	88.5	87.4	-1.1	92.5	91.7	-0.8
Molasses									
	F/D = 2			F/D = 4			F/D = 8		
	Start	End	Δ	Start	End	Δ	Start	End	Δ
%RDS	59.6	51.8	-7.8	58.6	47.0	-11.6	59.1	38.5	-20.6
pH	8.23	8.11	-0.12	8.70	7.89	-0.81	8.57	7.82	-0.75
σ (mS/cm)	6.737	12.57	5.833	6.318	16.53	10.212	7.376	19.60	12.224
Purity (%)	59.1	58.3	-0.8	59.8	59.2	-0.6	57.9	57.6	-0.3

* Thin juice from the 2024 campaign; stored frozen and repeatedly thawed/refrozen.

** Thin juice from the 2024 campaign; continuously stored in the freezer conditions.

*** The sample could not be filtered through membrane filter.

4.5 The Effect of Membrane Type on FO Performance

The suitability of different membranes was evaluated under FO conditions at ambient temperature and the highest allowable operating temperature for each membrane. Figure 4.9 presents the flux profiles obtained with RO membrane RO98pHt at ambient conditions and at 60°C. An increase in temperature results in slight improvement in flux from 0.4 LMH to 0.9 LMH, which is consistent with enhanced mass transfer.

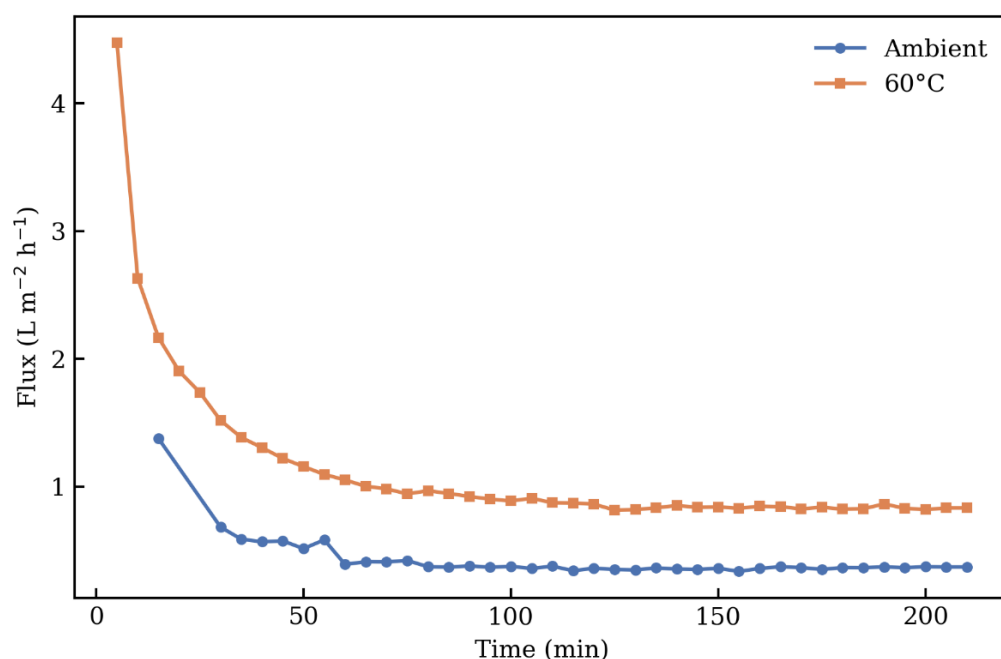


Figure 4.9. Water flux as a function of time at different values of temperature for RO98 membrane.

The compositional analysis revealed only minor differences between the two operating conditions, as shown in Table 4.9. For thin juice, the increase in %RDS is slightly higher at 60°C than at ambient temperature. Other parameters also experienced slight variations between two conditions. Similarly, for molasses, the decrease in %RDS is identical under both temperatures. It is important to note that no pH decrease is observed at 60°C, where microbial and fermentation activity is suppressed. This suggests that the pH reduction observed in the other runs is likely associated with fermentation occurring during the process.

Table 4.9. Results from analysis of thin juice and molasses before and after forward osmosis at different operational temperatures with RO98 membrane.

Thin juice						
	Ambient*			60°C*		
	Start	End	Δ	Start	End	Δ
%RDS	16.1	16.4	0.3	15.1	15.8	0.7
pH	9.0	9.3	0.3	8.58	8.58	0
σ (mS/cm)	2.878	2.929	0.051	2.828	3.078	0.25
Color (IU)	2360	2404	44	2216	2274	58
Purity (%)	91.7	91.9	0.2	94	94.1	0.1
Molasses						
	Ambient			60°C		
	Start	End	Δ	Start	End	Δ
%RDS	58.5	57.9	-0.6	60.3	59.7	-0.6
pH	8.42	8.55	0.13	8.62	8.57	-0.05
σ (mS/cm)	7.028	7.886	0.858	8.160	9.120	0.96
Purity (%)	58.3	58.1	-0.2	62.0	60.8	-1.2

* Thin juice from the 2024 campaign; continuously stored in the freezer conditions.

A similar set of experiments was conducted using the NF membrane NP030 at ambient temperature and 75°C, see Figure 4.10. The effect of temperature was more pronounced than for RO membrane, with the average flux increasing from 0.4 LMH to 3.2 LMH. This is likely a result of the greater temperature difference and higher permeability of NF membranes.

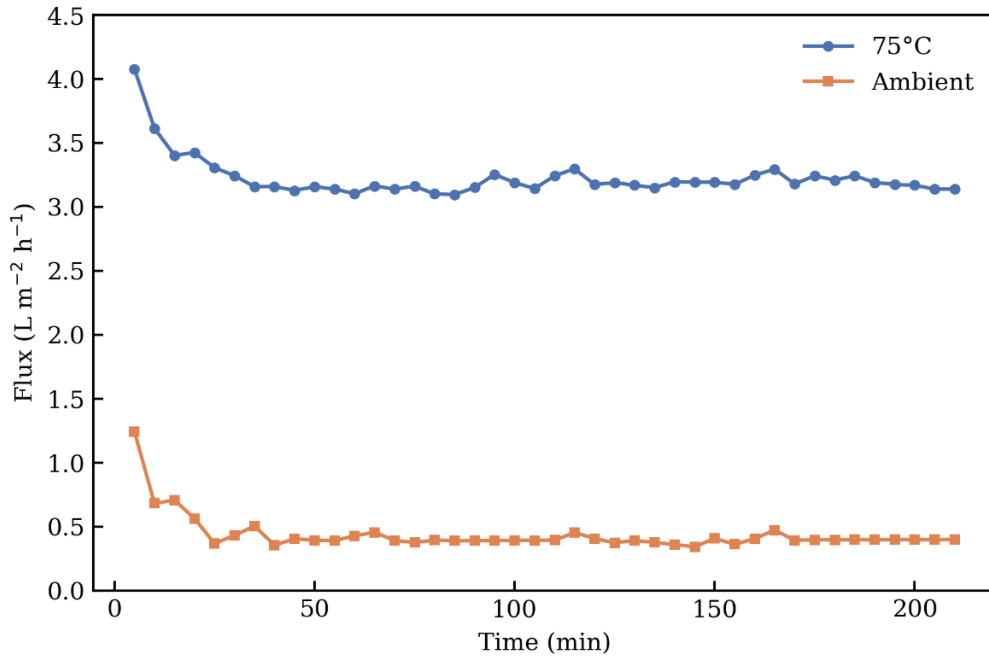


Figure 4.10. Water flux as a function of time at different values of temperature for NP030 membrane.

Figure 4.11 shows the flux performance of FO, RO and NF membranes under both temperature conditions. The FO membrane consistently exhibits the highest flux across all conditions. This can be explained by fundamental differences in membrane structure. Both RO98pHt and NP030 are thin-film composite (TFC) membranes with a polypropylene support layer, designed for pressure-driven processes. These membranes require a relatively thick and mechanically robust support structure, which increases the resistance to mass transfer. In contrast, the FO membrane is made out of cellulose triacetate and lacks this polypropylene layer. The hydrophobic nature of polypropylene can also hinder full wetting of the support structure, reducing effective water transport. As a result, TFC membranes experience more severe ICP, which limits flux despite their higher temperature tolerance. Similar findings were also reported in another study, where RO TFC membranes produced consistently lower flux than FO membranes due to much thicker support layer confirmed by SEM analysis [5].

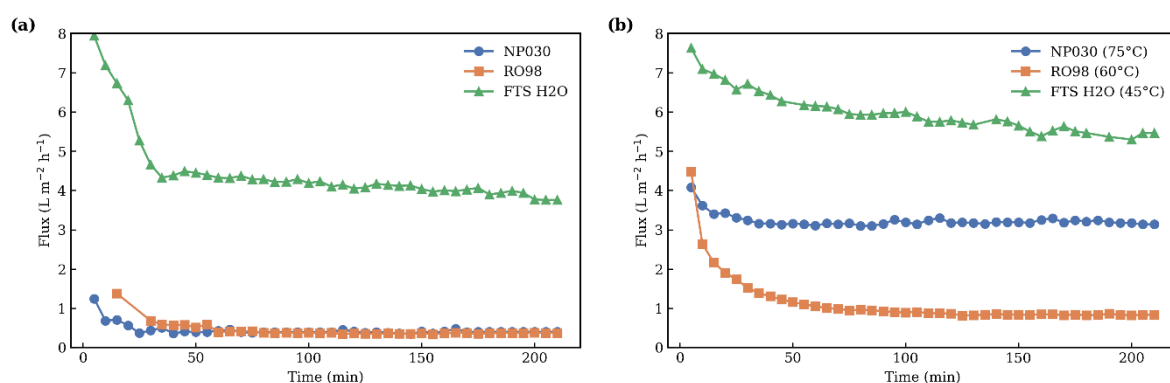


Figure 4.11. Water flux as a function of time for NP030, RO98, FTS H₂O membranes at (a) ambient temperature; (b) highest operating temperature.

Analysis of FS and DS at elevated temperatures further highlights differences between membranes, see Table 4.10. A notable observation for the NP030 membrane at 75°C is the significant rise in conductivity and increase in color for thin juice. This behavior is likely related to the higher molecular weight cut-off (MWCO) of the NF membrane. At elevated temperatures, membrane pores are increased in size, facilitating the passage of smaller organic compounds. Combined with higher flux, this can lead to the transfer of colorants from molasses into the feed solution, resulting in increased color formation. This is less pronounced in FO and RO membranes due to their tighter structure and lower solute permeability.

Table 4.10. Results from analysis of thin juice and molasses before and after forward osmosis at different operational temperatures with RO98 membrane.

Thin juice						
	Ambient*			75°C*		
	Start	End	Δ	Start	End	Δ
%RDS	17.1	17.4	0.3	17.2	18.5	1.3
pH	9.29	9.26	-0.03	9.03	8.80	-0.23
σ (mS/cm)	3.200	3.314	0.114	3.408	3.934	0.526
Color (IU)	2374	2404	30	2281	2702	421
Purity (%)	92.0	91.5	-0.5	94.4	93.3	-1.1
Molasses						
	Ambient			75°C		
	Start	End	Δ	Start	End	Δ
%RDS	59.3	58.4	-0.9	60.3	55.8	-4.5
pH	9.04	9.05	0.01	8.55	8.86	0.31
σ (mS/cm)	6.767	7.960	1.193	10.11	15.26	5.15
Purity (%)	59.3	59.7	0.4	59	60.1	1.1

* Thin juice from the 2024 campaign; continuously stored in the freezer conditions.

4.6 Concentration Study

A long-term concentration experiment was conducted over 24 hours to evaluate the stability and performance of the FO process. The FTS H₂O membrane was selected due to its superior flux performance, reaching 6 LMH at 45°C in previous trials.

The flux and volume reduction profiles are presented in Figure 4.12, a volume reduction of 0.5 was achieved during the experiment. The flux decreased from 5.8 to 3.4 LMH, which is consistent with typical FO behavior during extended operation. This decline is attributed to a reduction in the effective driving force, as the FS becomes more concentrated while DS is diluted. In addition, CP effects become more pronounced over time further reducing the net osmotic pressure difference.

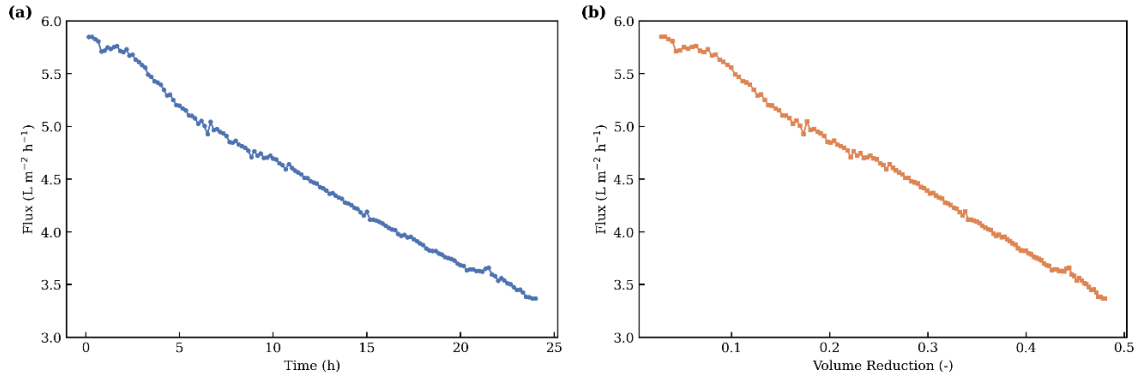


Figure 4.12. Water flux as a function of (a) time; (b) volume reduction.

The analysis of thin juice shows significant concentration from 16 to 27.9 %RDS, as shown in Table 4.11. A notable decrease in pH was also observed, likely due to fermentation of the feed, which could affect thermal stability of juice. The %RDS of molasses decreased from 59.8 to 48.4, confirming significant dilution. The %RDS values of FS and DS did not reach equilibrium by the end of the experiment, the process had not yet reached its thermodynamic limit. This suggests that further concentration could be achieved with continued operation.

Table 4.11. Results from analysis of thin juice and molasses before and after forward osmosis for 24 hours.

Thin Juice*			
	Start	End	Δ
%RDS	16.0	27.9	11.9
pH	8.25	6.04	-2.21
σ (mS/cm)	3.048	3.937	0.889
Color (IU)	1989	2153	164
Purity (%)	95.7	93.3	-2.4
Molasses			
	Start	End	Δ
%RDS	59.8	48.4	-11.4
pH	8.93	8.53	-0.4
σ (mS/cm)	9.070	17.41	8.34
Purity (%)	59.5	56.8	-2.7

* Thin juice prepared from thick juice.

5 Conclusions

This study demonstrated the feasibility of concentrating beet sugar juice using FO and showed that process performance is strongly influenced by operational parameters. Increasing DS flow rate improved flux by reducing ECP and maintaining a higher effective osmotic driving force. Similarly, higher DS concentrations also increased water flux, although the relationship was nonlinear due to enhanced ICP at elevated concentrations. Another key factor was operating temperature, which significantly enhances flux through reduced solution viscosity and increased diffusivity.

The choice of draw solution was also found to play a critical role. Comparison between sodium chloride and molasses revealed consistently lower flux values despite comparable osmotic pressure because of higher viscosity and complex composition of molasses. Nevertheless, molasses remains a highly attractive DS due to its direct compatibility with sugar production processes and potential for industrial integration.

Among the membranes evaluated, the FTS H₂O membrane consistently exhibited the highest water flux under all operating conditions. At 45°C, average permeate flux of 6 LMH was achieved, demonstrating its superior suitability for FO applications. In contrast, the RO and NF membranes exhibited substantially lower fluxes, primarily due to more severe ICP caused by their support layer structures. Although these membranes have wider operational range, structural limitations restricted their suitability for efficient FO operation.

The long-term concentration study further demonstrated the technical potential of the process. Stable operation was maintained over 24 hours, achieving a volume reduction factor of 0.5. Significant concentration of thin juice from 16 %RDS to 27.9 %RDS was achieved. Furthermore, osmotic equilibrium between the feed and draw solutions was not reached by the end of the experiment, suggesting that further concentration could be achieved with process optimization and extended operation times.

Overall, the results confirm that FO using molasses as DS represents a promising complementary concentration technology to conventional thermal evaporation for thin juice concentration. Future work should focus on improving industrial relevance and performance of the FO process. In particular, higher cross-flow velocities and larger F/D ratios should be investigated to better represent realistic operating conditions. Additional strategies to mitigate CP should also be explored, such as use of feed spacers to enhance turbulence, or ultrasonication to reduce fouling. Optimization of DS composition, including hybrid systems, could help balance osmotic pressure, viscosity and RSF.

List of Abbreviations

Abbreviation	Explanation
AL	Active layer
AL-DS	Active layer-facing-draw solution
AL-FS	Active layer-facing-feed solution
BL	Boundary layer
CEOP	Cake-enhanced osmotic pressure
CO _{2eq}	Carbon dioxide equivalent
CP	Concentration polarization
DI	Deionized
DS	Draw solution
ECP	External concentration polarization
F/D	Feed-to-draw
FO	Forward osmosis
FS	Feed solution
ICP	Internal concentration polarization
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
K	Solute resistivity
LMH	$\text{L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$
MWCO	Molecular weight cut-off
NF	Nanofiltration
PWF	Pure water flux
RDS	Refractometric dry substance
RO	Reverse osmosis
RPM	Revolutions per minute
RSF	Reverse solute flux
S	Structural parameter
SEM	Scanning electron microscopy
SL	Support layer
TFC	Thin-film composite
UV-vis	Ultraviolet-visible

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