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Abstract. Due to the high total dissolved solids (TDS) content in tannery wastewater, industrial wastewater reuse is possible if a reverse osmosis (RO) process is implemented. RO can successfully be applied for TDS removal from tannery wastewater, provided a suitable pretreatment system is employed before RO. Forward Osmosis (FO) could be a good option for replacing RO pretreatment methods like Ultrafiltration (UF) for treating industrial effluents. In this study, the FO process is investigated to concentrate three different types of synthetic secondary treated tannery effluents (FS1, FS2, and FS3) as feed solution and NaCl solution as draw solution. Operating FO at low osmotic pressure gradient ($ΔΠ_{low}$) of 9.33 ± 0.7 bars and high osmotic pressure gradient ($ΔΠ_{high}$) of 20.63 ± 0.7 bars, for the concentration of FS1, FS2, and FS3, respectively, was evaluated in terms of flux (Jw), flux decline ratio (FDR), the percentage increase in feed solution concentration (CF) and solute rejection (SR). Results show that operating FO at ΔΠ_{high} gave higher J_w and lower FDR than operating FO at $ΔΠ_{low}$, irrespective of feed solution composition. Operating FO at $ΔΠ_{high}$ provided higher J_w and lower FDR for FS3 (6.18 LMH and 26.88%) and FS2 (6.16 LMH and 27.84%) as compared to FS1 (5.7 LMH and 32.52%). CF and SR were higher for experiments performed at ∆Πhigh than ∆Πlow, irrespective of feed solution composition. Solutes like magnesium and chromium had good rejection (>80%) due to size exclusion at Δ Π_{high} and Δ Π_{low}, irrespective of FS type. Potassium and Ammonium, having a small hydrated radius, had low rejection at ΔΠ_{high} and ΔΠ_{low} for FS3, FS2, and FS1, respectively. Essentially, by emphasizing the osmotic pressure gradient as a critical component, this study suggested a systematic strategy for researching the FO process.

Keywords: Forward Osmosis (FO), FO Membrane, Feed Concentration, Osmotic Pressure, Solute Rejection, Tannery Effluent

INTRODUCTION

The production of leather involves preparing hides/skins for tanning through pre-tanning operation, permanently preserving skin proteins by tanning, and improving the aesthetic look in the aftertanning stages. For every ton of raw material treated, between 30 and 40 $m³$ of effluents are produced. Many substances, such as lime, fat liquors, sodium and ammonium salts, antibiotics, tannins, colors, and tannins, are employed in leather manufacturing (Kaul *et al.*, 2013). Process chemicals may find their way into wastewater, contributing significantly to pollution. The effluent treatment procedure uses a traditional treatment system consisting of unit biological procedures followed by physical and/or chemical processes. Pollutants include suspended particles, and chemical and biochemical oxygen demand (COD and BOD) can be eliminated with a traditional treatment system. Resource recovery and water reuse are often practiced in the industry to reduce the strength and quantity of wastewater produced. The industry practices biological treatment methods to remove total dissolved organics from wastewater. Even after treatment, the treated tannery wastewaters contain some organic, nitrogenous matter and high total dissolved solids (TDS) (Pophali and Dhodapkar, 2011; Ramteke *et al.*, 2010).

The constituents contributing to TDS in tannery effluents are calcium, ammonium, magnesium, sodium, chlorides, nitrates, and sulphates. Such a treated effluent containing high TDS is not suitable for process and nonprocess reuse applications in the industry (Zhao *et al.*, 2022). The effluent must be discharged to surface water, where strict limits are laid for chlorides and TDS parameters. Further, disposal of treated tannery effluent, high in chlorides and TDS, is reported to affect the fertility of the soil and contaminate groundwater, making the soil unsuitable for agriculture and the water unfit for utilization purposes (Bhardwaj *et al.*, 2023). Reverse Osmosis (RO) has been employed in tanning industries and common effluent treatment plants treating high TDS wastewater from the tannery industries (Ranganathan and Kabadgi, 2011). The secondary treated tannery wastewater is passed through either conventional RO pretreatment steps or Ultrafiltration, Microfiltration, and Nanofiltration membrane systems to improve the feed water quality of the RO system. Conventional pre-treatment processes to reverse osmosis treatment system consist of first pH adjustment next, followed by coagulation/flocculation, disinfection, filters with multimedia, and filtration (activated carbon and cartridge) (Jang *et al.*, 2017; Pramanik *et al.*, 2014; Sweity *et al.*, 2013). In addition, membrane pretreatment technologies like MF, UF, and NF are also practiced for Reverse Osmosis (Kim *et al.*, 2002; Suthanthararajan *et al.*, 2004). All these methods increase the cost of the treatment and chemical usage.

Forward Osmosis (FO) might be a useful substitute for RO pre-treatment when treating secondary processed tannery effluents. According to the literature, the best chance of minimizing RO membrane fouling and scaling is to employ forward osmosis as a pre-treatment before RO (Al-Zuhairi *et al.*, 2015; Korenak *et al.*, 2019; Thiruvenkatachari *et al.*, 2016; Zaviska *et al.*, 2015). This increases the life of the membranes, requires less chemical cleaning of membranes, and causes less damage to membranes during cleaning in the RO process. The RO process currently accomplishes TDS removal from tannery effluents in industries by the RO process. Still, it comes at a high cost because of scaling, fouling, and damage of RO membranes, as discussed. Limited research is available for treating/concentrating tannery effluents with different characteristics with the FO system (Pal *et al.*, 2017). No research is available on the performance of the FO process for concentrating tannery effluents at varying osmotic pressure gradients. Moreover, the researchers have often used cross-flow FO

module configuration, which quite an intensive option, and no research is available on other FO module configurations used for treating secondary treated tannery effluent. In this study, we have evaluated the functionality of the compartment configuration FO system in concentrating synthetic secondary treated tannery effluent. The work intends to explore the performance of the FO process in terms of water flux, flux decline ratio, percentage increase in FS concentration, and solute rejection when concentrating three different synthetic secondary treated tannery effluents as feed solution.

MATERIALS AND METHODS

FO Membranes and Membrane Orientation

Aquaporin-embedded FO flat sheet membranes from Aquaporin Asia, Singapore, were purchased. The membranes were soaked in fresh deionized (DI) water and kept at 4ºC. Once a week, DI water was replaced with fresh water. The membranes were washed in DI water for around 1 hour at room temperature before use. All studies were carried out with the membrane selective/active layer seeing towards the feed solution (FS).

Feed and Draw Solutions

The feed solution was DI water or synthetic secondary treated tannery effluent (SSTTE). Three synthetic secondary treated tannery effluent (SSTTE) solutions (designated as FS1, FS2, and FS3, respectively), simulating secondary-treated real tannery effluents, were prepared using constituents as shown in Table 1. The feed solutions were autoclaved and cooled, and a dose of 6 mg/L ampicillin solution was added

to avoid bacterial growth in the feed solutions during the experimental duration. The pH of the solution was adjusted using a 12 N NaOH solution. The initial volume of the feed solutions was 1250mL for all experiments with SSTTE as FS.

The draw solution consisted of sodium chloride solutions of 14, 20, 24, 28, 34, and 38 g/L, respectively. The initial volume of the draw solution was 1000 mL for all experiments. The chemical reagents were purchased from Aplhachemika Pvt Ltd, Mumbai, and were of analytical grade. Ampicillin was purchased from HiMedia, Mumbai. DI water was supplied via a Merck Millipore water purification system (18.2 MΩ·cm at 25 ºC), and it was utilized to prepare the draw and feed solutions, rinse the FO module after the experiment, and dilute samples for analysis.

Bench Scale FO Setup

Figure 1a shows a schematic sketch of the bench size experimental setup used in this study, and Fig. 1b shows a photograph view of the experimental setup. The experiment was conducted in an air-conditioned room at 23 \pm 1°C. This continuous flow apparatus is used to test the performance of the FO process under continuous feed solution supply and constant concentration draw solution conditions. The feed solution reservoir (1) supplies feed solution to the feed solution tank. The weight of the feed solution reservoir was monitored at time intervals to determine water flux owing to the FO process. Weight was measured manually on a weight balance (14) (Citizen CTG, India).

The aquaporin forward osmosis membrane vertically separates two acrylic tanks or compartments in this setup. The FO membrane was sealed to prevent leakage using a 3mm thick EPDM rubber sheet and 20 no's of 6 mm diameter stainless steel nuts and bolts. Out of the two compartments, the proper compartment (2) was of feed solution (which was deionized water/ wastewater), and the left compartment (3) was of draw solution, which was sodium chloride solution. The feed solution tank dimensions are 18.2cm length x 5.5cm width x 11cm height. The feed solution volume in the feed solution tank was maintained at 1 liter by providing a float valve (6) (MA052, Kerick, India) at the inlet of the feed solution tank. The accountable membrane area was (8.5cm height x 4.0cm width) 34 $cm²$. The feed solution sees the membrane's active layer. As pure water passes through the forward osmosis membrane, the draw solution volume increases, and the feed solution's volume decreases. The float valve controlled the rate at which feed solution was pumped into the feed solution tank from the reservoir.

The draw solution tank dimensions were 18.2cm x 5.5cm x 11cm. The draw solution volume used in the experiments was 1 L. Pure water flux from the feed solution tank via the FO membrane causes the draw solution volume to rise and dilute. To collect and separate the increased draw solution volume, a 0.5mm diameter outlet port is provided to the draw solution tank at the height of 10 cm. The draw solution's increased volume was collected in a different draw solution tank (4) after passing through the draw solution tank's outlet port. Thus, the draw solution volume in the draw solution tank was maintained constant. A more concentrated draw solution, consisting of 91.2 g/L sodium chloride solution, was fed into the draw solution tank from the concentrated draw solution reservoir (5).

This experiment was done via a peristaltic pump (13) (Ravel, India) that is linked to a conductivity controller (12) (MSCD09, MicroSet, India) to maintain equilibrium in the draw solution tank. The water's conductivity increase in the feed solution tank is measured by a conductivity meter (Inolab Cond720, WTW Germany). A temperature control unit controlled the temperature of the two compartments separated by a membrane. The temperature control unit consists of 2 no's of heating rods (8) (MINI-THERM, Cobalt International, SC) and 2 no's of temperature controllers (11) (XK-W2001, Robocraze, India). Mixing was done in the draw solution tank with the help of an axial impeller connected to a laboratory stirrer (10) (RQG-121-D, Remi Elektrotechnik Ltd, India). Feed solution mixing was carried out by diffusing air with the help of an aerator (16) (Eheim, Germany). The forward osmosis setup is unique in one way: this setup did not utilize pumps for the movement of the feed solution and draw solution.

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Fig. 1: Forward osmosis setup. a) Schematic diagram of the experimental setup, b) Photograph of the experimental setup. 1: Feed Solution Reservoir, 2: Feed Solution Tank, 3: Draw Solution Tank, 4: Separated Draw Solution Tank, 5: Concentrated Draw Solution Reservoir, 6: Float Valve 7: Flow Control Valve, 8: Heating Rods, 9: Conductivity Probe, 10: Stirrer, 11: Temperature Controller Unit, 12: Conductivity Controller Unit, 13: Peristaltic Pump, 14: Weigh Balance, 15: FO membrane Unit. 16: Aerator

FO Experimental Procedure

These experiments applied two different osmotic pressure differences to the FO membrane. For the feed solutions FS1, FS2, and FS3, the draw solution concentrations were 14, 20, and 24 g/L of NaCl solution, respectively, to conduct experiments at low osmotic pressure gradient (ΔΠ_{low} = 9.33 ± 0.7bars). For the feed solutions FS1, FS2, and FS3, the draw solution concentrations were 28, 34, and 38 g/L of NaCl solution, respectively, to allow for experiments to be performed at high osmotic pressure gradient $(Δ\Pi_{high} = 20.63 ± 0.7$ bars). Aeration in the FS

tank was carried out in all tests at a rate of 0.3 mL/min, and mixing in the draw solution tank was done at a constant mixing rate of 75 RPM.

Calculation of Pure Water Flux, Flux Decline Ratio, Increase in Feed Solution Concentration and Solute Rejection.

The pure water flux J_w (LMH) was computed using the Eq. (1) as given by (Dutta *et al.*, 2022);

$$
J_w = \Delta W / (p \times S_m \times \Delta t)
$$
 (1)

where, ΔW is the weight (g) alteration of the

feed solution over a specific time Δt (hours), ρ is feed solution density (assumed 1.0 $q/cm³$), and S_m is the membrane's effective surface area (m^2) . Further, the flux decline ratio FDR (%) was calculated using Eq. (2) as given by (Mondal and De, 2015);

$$
FDR = 100 - [(J_{w, f} / J_{w, i}) \times 100]
$$
 (2)

where, $J_{w,f}$ is the flux recorded after the stop of the experiment and $J_{w, i}$ is the initial recorded flux.

The percent increase in concentration of feed solution CF was calculated as described by Eq. (3) as given by (Andrzejewski *et al.*, 2022);

$$
CF = \frac{C_{f,Final} - C_{f,Initial}}{\frac{C_{f,Final} + C_{f,Initial}}{2 \times C_{f,Initial}}}
$$
(3)

where, C_{ffinal} is the final electrical conductivity (mS/cm at 25 ºC) at the end of the experiment, and C_{f,Initial} is the first electrical conductivity of feed solution (mS/cm at 25 $^{\circ}$ C).

Different ions in the feed solutions have been studied to determine their solute rejection factors. Eq. (4), as given by (Vital *et al.*, 2018), was used to determine solute rejection SR (%).

$$
SR = [(C_{f, initial} - C_{p}) / C_{f, initial}] \times 100
$$
 (4)

where the concentration of the ion on the feed side is represented by C_f Initial and the concentration on the permeate (pull side) by Cp, both expressed in mg/L. Eq. (5), provided by (Vital *et al.*, 2018), is used to calculate the concentration (mg/L) of the permeate C_p .

$$
C_p = (C_d \times V_d) / V_p \tag{5}
$$

where V_d is the volume (L) of the draw solution after the experiment, V_p is the volume (L) of water that percolates from the feed to the draw side, and C_d is the concentration (mg/L) on the draw side.

Sampling and Analytical Methods

Feed solution (synthetic secondary treated tannery effluent) in the feed solution tank was collected before the start and after the completion of the experiment. Draw solution samples were gathered after the trial for analysis. The feed solution samples gathered before the start of the experiment were analyzed for Chemical Oxygen Demand (COD), Electrical Conductivity (EC), Total Chromium, cations (Sodium, Ammonium, Potassium, Magnesium, and Calcium), and anions (Chlorides, Nitrates, Orthophosphates, and Sulphates). The feed solution samples gathered after the experiment were analyzed for EC. The draw solution samples were analyzed for Total Chromium, cations, and anions.

RESULTS AND DISCUSSION

To put it briefly, the COD (Aqualytic AL38SC) was measured using a closed reflux colorimetric technique in compliance with Standard Procedures for the Analysis of Water and Wastewater. The absorbance of the digested solution was measured at 600 nm using a Hach DR-6000 UV-Visible spectrophotometer to estimate the COD levels. Ion Chromatography determined cations (Sodium, Ammonium, Potassium, Magnesium, and Calcium) and anions (Chlorides and Sulphates) with chemical suppression of eluent conductivity (Metrohm, 850 Professional IC). Nitrate was measured as Nitrate-N by ultraviolet spectrophotometric screening method (APHA 4500). Phosphate was measured by the stannous chloride method (APHA 4500-P D). Further cation (Magnesium, Potassium, Total Chromium) concentrations in the DS were also examined using an (ICP-OES, Agilent 700) inductively coupled plasma-optical emission

spectrometer. In contrast, Ammonium was examined using Ion Chromatography.

Characterization of Feed Solution and Draw Solution

This study uses synthetic secondary treated tannery wastewater as a feed solution while sodium chloride is the draw solution. The characterization of secondary treated tannery wastewater from individual effluent treatment plants is obtained from the literature and presented in Table 2. Further, a grab sample was collected from the outlet of the secondary sedimentation tank of a Tannery ETP located in Uttar Pradesh, India. The results presented in Table 2 show that the effluent quality exhibit dissimilarities in various parameters. The properties of wastewater from treated tanneries differ between tanneries, locations, and nations. Variations in wastewater characteristics from different tanneries depend upon the type of process employed, water consumption rates, recovery systems, and process modifications adopted to reduce pollution load and hide the processing capacity of the tanneries (Kaul *et al.*, 2013). The TDS in raw tannery influent vary because of the adoption of low salt concentration, desalting of hides, and the adoption of a chilling system for the preservation of hides (Fisher and Pearce, 2009).

Three synthetic secondary-treated tannery effluents were prepared using chemical composition, as shown in Table 1. The characterization of synthetic secondary treated tannery effluents is shown in Table 3. A 6mg/L dose of ampicillin was added to avoid microbial growth in the each feed solution (Bharagava *et al.*, 2014; Shanthi *et al.*, 2012).

The draw solution in this study is sodium chloride (NaCl). NaCl is a more commonly utilized inorganic draw solution due to its great solubility, affordability, and strong osmotic pressure potential (Nguyen *et al.*, 2015), (Dsilva Winfred Rufuss *et al.*, 2023). At increasing concentrations of NaCl, flux increases compared to other draw solutes (Al-Aibi *et al.*, 2016), (Ma *et al.*, 2023).

Parameter	Unit	(Ranganathan & Kabadgi,			(Rodrigues,		This Study
		2011)			2008)		
pH		7.8	8.3	7	7	7.6	9.2
TDS	mg/L	6708	4150	7966	3769	10649	8211.34
BOD	mg/L	260	140	30	55	94.5	31.5
COD	mg/L	300	472	226	302	$- -$	111.35
Chlorides	mg/L	1396	675	1711	1157	3990	2996.31
Sulphates	mg/L	3318	1387	3657	567		2142.26
Sodium	mg/L	2120	1220	2280	1040	2850	2516.51
Potassium	mg/L	18	54	34	18	$- -$	23.33
Calcium	mg/L	160	128	192	39.2	207	354.52
Magnesium	mg/L	67.2	0	16.8	13.92	202	15.27
Ammonium	mg/L		--		--	634	139.57
Total Chromium	mg/L	BDL	2.2	0.06	BDL	0.4	0.02

Table 2. An overview of the composition of real secondary treated tannery effluent.

Table 3. Characterization of synthetic

Effect of Osmotic Pressure Gradient on Flux and Flux Decline Ratio

Experiments with varying osmotic pressure gradients between the FS and DS were conducted to identify the effect of osmotic pressure gradients on the performance of the FO process in terms of flux, flux decline ratio, concentration factor, and solute rejection. Experiments were conducted using FS1, FS2, and FS3 as FS and 14, 20, and 24 g/L of NaCl solution as DS to identify the low osmotic pressure gradient effect on the FO process. Experiments were conducted using FS1, FS2, and FS3 as FS and 28, 34, and 38 g/L of NaCl solution as DS to identify the high osmotic pressure gradient effect on the FO process.

Figure 2 depicts the membrane flux about time for different FS and DS concentrations near the same bulk osmotic pressure gradient of 9.3±0.7 bars. Between FS1 and 14 g/L NaCl solution, there was an osmotic pressure gradient of 8.6 bars; between FS2 and 20 g/L NaCl solution, there was a gradient of 9.3 bars; and between FS3 and 24 g/L NaCl solution, there was a gradient of 10.1 bars. The initial flux values for FS1, FS2, and FS3 were 5.79, 5.47, and 4.41 for 14, 20, and 24 g/L of NaCl solution. The initial flux for FS3 is low even when the bulk osmotic pressure gradient is near the same. The possible reason for this might be that the DICP is severe when the DS concentration is higher (Bui *et al.*, 2015), (Idris *et al.*, 2022). The osmotic pressure on the DS side reduces due to the DICP effect, eventually reducing the initial water flux. The flux starts to decline as a function of time. The first rapid decline in flux is mainly because of fouling, while further, the flux declines due to the combined actions of fouling, the concentration of FS, and the back diffusion of DS (Han *et al.*, 2016), (Nguyen *et al.*, 2021).

Fig. 2: Flux as a function of time for low osmotic pressure gradient (9.3±0.7 bars) experiments.

The initial flux decline ratio in the first 3 hours for FS1, FS2, and FS3 is 15.19, 13.97, and 5.21 %, respectively. This shows that the foulant layer was developed for FS1 and FS2, while for FS3, the foulant layer was not yet stabilized. Further, the flux decline ratio at the end of the experiments was 45.35, 40.9, and 36.1 % for FS3, FS2, and FS1, respectively. The fouling propensity was more in descending

order for FS3, FS2, and FS1, respectively, but the initial flux decline ratio was low for FS3 compared to FS1 and FS2. However, at the end of the experiment, the flux decline ratio for FS3 was higher because the feed solution composition of FS3 has a higher fouling propensity than FS1 and FS2 (She *et al.*, 2016), (Nguyen *et al.*, 2021).

Fig. 3: Flux as a function of time for high osmotic pressure gradient (20.6±0.7 bars) experiments.

Figure 3 shows the membrane flux as a function of time for different FS and DS concentrations having a near the same bulk osmotic pressure gradient of 20.6±0.7 bars. The bulk osmotic pressure gradient between FS1 and 28g/L NaCl solution was 19.9 bars, the bulk osmotic pressure gradient between FS2 and 34 g/L NaCl solution was 20.6 bars, and the bulk osmotic pressure gradient between FS3 and 38 g/L NaCl solution was 21.4 bars.

The initial flux values for FS1, FS2, and FS3 were 7.41, 7.5, and 7.44 for 28, 34, and 38 g/L of NaCl solution. The initial flux values of FS1, FS2, and FS3 are near each other, possibly because of the DICP effect. The same net osmotic pressure difference between the FS and DS must have been produced. The flux starts to decline as a function of time. The first rapid decline in flux is mainly because of fouling, while further, the flux declines due to the combined effects of fouling, the concentration of FS, and the back diffusion of DS (Han *et al.*, 2016). The initial flux decline ratio in the first 3 hours for FS1, FS2, and FS3 is 19.43, 9.8 and 9.14 %, respectively. The rapid flux decline ratio for FS1, as compared to FS2 and FS3, might be high because of the reduced osmotic gradient due to DICP and rapid RSF from the DS to the FS side. Further, the flux decline ratio at the end of the experiments was 32.52, 27.84, and 26.88 % for FS1, FS2, and FS3. The fouling propensity was more in descending order for FS1, FS2 and FS3.

Fig. 4: Average flux for low and high osmotic pressure gradient experiments.

From Fig. 4, one can notice that the 7 hours average water flux values for FS3, FS2, and FS1 experiments were 3.54, 4.15, and 4.537 LMH, respectively, at a low osmotic pressure gradient. In contrast, the 7 hours average water flux for FS3, FS2, and FS1 was 6.18, 6.16, and 5.7 LMH, respectively, at high osmotic pressure gradient. Experiments with lower osmotic pressure gradients have lower average flux, while experiments with higher osmotic pressure gradients have higher average flux for FS1, FS2, and FS3, respectively (Camilleri-Rumbau *et al.*, 2019).

The percentage difference between the average water flux of FS1 and FS2 was 8.98%, whereas the percentage difference between the average water flux of FS1 and FS3 was 34.47% for low osmotic gradient experiments. It can be said that when FO is operated for concentrating feed solutions having high concentration at low osmotic pressure gradient, a slight decline in osmotic pressure gradient due to DICP, RSF, and fouling will impact the flux largely (Andrzejewski *et al.*, 2022). The percentage difference between the average water flux of FS3 and FS2 was 0.32%, whereas the percentage difference between the average water flux of FS3 and FS1 was 8.08% for high osmotic pressure gradient experiments. When operated at a high osmotic pressure gradient, the average water flux was near the same for FS1, FS2, and FS3 experiments. It can be said that when FO is operated for concentrating feed solutions having low concentration at high osmotic pressure gradient, the decline in osmotic pressure gradient due to DICP, RSF, and fouling will not impact flux largely (Oymaci *et al.*, 2021).

From Figure 5, it is evident that the flux decline ratio (FDR) for FS1, FS2, and FS3 were 36.1, 40.9, and 45.35 %, respectively, at low

osmotic pressure gradient. In contrast, the flux decline ratio for FS1, FS2, and FS3 was 32.52, 27.84, and 26.88 %, respectively at high osmotic pressure gradient. Experiments conducted with lower osmotic pressure gradients have higher flux decline ratios, while experiments conducted with higher osmotic pressure gradients have lower flux decline ratios for FS1, FS2, and FS3, respectively (Gao *et al.*, 2018). The difference between the flux decline ratio of FS3 and FS2 was 4.45%, whereas the percentage difference between the flux decline ratio of FS3 and FS1 was 9.25% for low osmotic gradient experiments. It can be said that when FO is operated for concentrating feed solutions having high concentration at low osmotic pressure gradient, a slight decline in osmotic pressure gradient due to DICP, RSF, and fouling will increase the flux decline ratio (Ortega-Bravo *et al.*, 2016). The percentage difference between the average water flux of FS1 and FS2 was 4.68%, whereas the percentage difference between the average water flux of FS1 and FS3 was 5.64 % for high osmotic pressure gradient experiments. When operated at a high osmotic pressure gradient**,** the FDR was nearly the same for FS1, FS2, and FS3 experiments. When FO is operated for concentrating feed solutions having low concentration at high osmotic pressure gradient, the decline in osmotic pressure gradient due to DICP, RSF, and fouling will not impact the flux decline ratio largely. In hydraulic pressure driven membrane processes, higher values of flux decline ratio indicate that the membrane has fouled more (Conidi *et al.*, 2019). However, this may not be true in FO processes where hydraulic pressure is not used. In the FO process, though, flux decline is slower in the low osmotic pressure gradient experiment than in the high osmotic pressure gradient

experiment, but operating the FO process at low DS concentration has a greater impact on flux (Gao *et al.*, 2018). A slight decrease in osmotic pressure gradient decreases the flux largely, especially at low osmotic pressure gradient as compared to high osmotic pressure gradient as the flux vs osmotic pressure curve is non-linear (Lay *et al.*, 2012; Oymaci *et al.*, 2021).

Effect of Osmotic Pressure Gradient on Concentration of Feed Solution

Since electrical conductivity (EC) and concentration are directly correlated, the increase in FS concentration was ascertained by measuring FS's EC at the beginning and end of the experiment (Zhang *et al.*, 2020). The percentage increase in EC of the feed solution, CF (%), was calculated using Eq. (3) by measuring the feed solution's initial and final electrical conductivity. Therefore, CF represents the percentage increase in concentration of FS due to the FO process. This increase in concentration during the FO process is due to the addition of FS from the FS reservoir and reverse salt flux from the DS during the FO process (Hickenbottom *et al.*, 2013), (Salamanca *et al.*, 2023).

Figure 6 presents the percentage increase in the FS EC after the experiment for FS1, FS2, and FS3 for experiments conducted at low and high osmotic pressure gradients. The EC of FS1, FS2, and FS3 raised from 6.94, 16.17, and 22 mS/cm to 7.24, 16.88, and 23.2 mS/cm, respectively, after of the experiment, giving 4.23, 4.29, and 5.31 % increase when concentrated at low osmotic pressure gradient. The EC of FS1, FS2, and FS3 raised from 7.3, 16.11, and 22.10 mS/cm to 8.4, 18.06, and 24.5 mS/cm, respectively, after the experiment, giving 14.01, 11.41, and 10.3 % increase when concentrated at high osmotic pressure gradient. The FS was more

concentrated at a high osmotic pressure gradient, than against a low osmotic pressure gradient irrespective of FS composition. This is because when the concentration gradient between DS and FS is more significant, the concentration of FS is greater (Singh *et al.*, 2018).

Fig. 6: Increase FS concentration for low and high osmotic pressure gradient experiments.

When FO is operated at a low osmotic pressure gradient, the CF is lower for FS1 (4.23%) and FS2 (4.29%) than for FS3 (5.31%). This emphasizes the significance of draw solution concentration selection when operating the FO process for different wastewater strengths. It is concluded here that when FS concentration is low as like FS1, operating FO at a low osmotic pressure gradient will not be fruitful as FS concentration will also contribute to reducing the osmotic driving force along with DICP, RSF and membrane fouling (Song *et al.*, 2018). A slight decrease in osmotic driving force for FO systems operated at low DS concentrations will severe impact flux as the flux vs DS concentration profile is nonlinear (Lay *et al.*, 2012), (Ma *et al.*, 2023). Even though FS2 and FS3 are operated at low osmotic pressure gradients, the CF is near the same. This is because the draw solution concentration was higher for FS2 and FS3 than for FS1, and so the DICP did not impact flux, but the composition of FS2 and FS3 majorly impacted the flux. On the other hand, if FS1 is operated at a high osmotic pressure gradient, it will be fruitful as DICP, RSF, and membrane fouling will not reduce the osmotic driving force compared to FS2 and FS3. An increase in FS concentration due to osmosis will not impact the osmotic driving force majorly if FO is operated at a high osmotic pressure gradient (She *et al.*, 2016), (Dsilva Winfred Rufuss *et al.*, 2023). Figure 6 shows that when FO is operated at a high osmotic pressure gradient, the concentration percentage is higher for FS1 (14.01%) than for FS2 (11.41%) and FS3 (10.3%). Even though there is a slight decrease in osmotic driving force for FO systems operated at high DS concentrations, this will not impact flux as the flux vs DS concentration profile is nonlinear. But for FS2 and FS3, when operated at high osmotic pressure gradient, the CF was lower because the feed solution composition had impacted membrane fouling, resulting in lower flux (Hickenbottom *et al.*, 2013), (Salamanca *et al.*, 2023).

Effect of Osmotic Pressure Gradient on Solute Rejection

Various possible explanations exist for the FO system's rejection of the ions. Convective motion has a negligible impact on ion conveyance because no stimulation is used in the FO process. The Donnan equilibrium effect may also be bestowed upon the big refusal under the FO process.

Figure 7 shows the rejection of Chromium and Magnesium by the FO membrane when FS as FS1, FS2, and FS3 were concentrated at low and high osmotic pressure gradients. The rejection for Magnesium ion for FS1, FS2, and FS3 was 82.35, 84.17, and 94.56 % when DS was 14, 20,

and 24 g/L, respectively. Magnesium rejection was more significant at high FS concentrations. Due to size exclusion, magnesium is effectively rejected since it is a divalent cation with a hydrated radius bigger (0.428 nm) than the FO membrane's pore size (Coday *et al.*, 2015; Gao *et al.*, 2018). Magnesium rejection is possibly low when it is low in concentration in the FS, and this is because of the Donnan equilibrium effect. Further, the rejection for magnesium ion for FS1, FS2, and FS3 was 88.92, 95.71, and 98.13 % when DS was 28, 34, and 38 g/L, respectively. It is seen that rejection increases as the FO process is operated at high osmotic pressure compared to low osmotic pressure. One explanation is that the presence of an impermeant ion for electroneutrality at high osmotic pressure prevents additional solutes from passing through the membrane, hence increasing solute rejection.

Fig. 7: Solute (Chromium and Magnesium) rejection for low and high osmotic pressure gradient experiments.

The rejection for Chromium ion for FS1, FS2, and FS3 was 78.9, 82.46, and 86.23 % when DS was 14, 20, and 24 g/L, respectively. Chromium rejection was more significant at high FS concentrations. The hydrated radius of chromium ion is 0.461 nm, and, therefore, is well rejected by the FO membrane. Possibly, Chromium rejection is low when it is low in

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concentration in the FS, and this is because of the Donnan equilibrium effect (Roy *et al.*, 2016), (Sanahuja-Embuena *et al.*, 2021). Further, the rejection for Chromium ion for FS1, FS2, and FS3 was 84.24, 96.22, and 97.44 % when DS was 28, 34, and 38 g/L, respectively. It is seen that rejection increases when a high osmotic pressure gradient is used during the FO process compared to a low osmotic pressure gradient. The possible reason might be that at high osmotic pressure, the availability of impermeant ions for electroneutrality might avoid the passing of other solutes through the membrane, thus increasing the solute rejection.

Figure 8 shows the solute rejection for Potassium and Ammonium. The rejection for Potassium ion for FS1, FS2, and FS3 was - 17.23, 14.24, and 14.17 % when the osmotic pressure difference was 8.6, 9.3, and 10.1bars, respectively. Further, the rejection for Potassium ions for FS1, FS2, and FS3 was - 18.46, 38.36, and 30.26 % when the osmotic pressure difference was 19.9, 20.6, and 21.4 bars, respectively. The rejection of potassium was negative for FS1, representing that potassium ions can easily be transported to the draw solution when the initial potassium concentration is low in FS. However, when the

concentration of potassium increases, as in FS2 and FS3, the rejection of potassium ions is seen to be increased compared to FS1. However, potassium, a monovalent ion with a low hydrated radius (0.331 nm) and more excellent water permeability is less rejected by FO membranes irrespective of the FS concentration (Hancock *et al.*, 2011). It is seen that rejection increases as the FO process is operated at high osmotic pressure compared to low osmotic pressure. The possible reason might be that at high osmotic pressure, the availability of impermeant ions for electroneutrality might avoid the transport of other solutes through the membrane, thus increasing the solute rejection.

Ammonium was determined by Ion Chromatography and at high dilution ratios. The rejection for Ammonium ion for FS1, FS2, and FS3 was 100, 42, and 46.15 % when the osmotic pressure difference was 8.6, 9.3, and 10.1 bars, respectively. Further, the rejection for Ammonium ions for FS1, FS2, and FS3 was 100, 100, and 81.16 % when the osmotic pressure difference was 19.9, 20.6, and 21.4 bars, respectively. The possible reason for the 100 % rejection of ammonium for FS1 when concentrated using low and high osmotic pressure is that the initial concentration of ammonium in FS1 was low (Ortega-Bravo *et al.*, 2016), and while analysis of the impact of dilution of the DS might have made it below detection limit of the Ion Chromatograph. For FS2, the rejection of ammonium was 42% and 100 % when concentrated using low and high osmotic pressure. This shows that at a low osmotic pressure gradient, the rejection of ammonium was lower than at a high osmotic pressure gradient (Volpin *et al.*, 2019). Likewise, in Potassium for FS3, the rejection of Ammonium was lower and was 46.15% and 81.16% when DS was 24 g/L and 38 g/L of NaCl solution, respectively, because, in FS3,

the initial concentration of ammonium was higher compared to FS1 and FS2 (Gao *et al.*, 2018). Ammonium, a monovalent ion with a low hydrated radius (0.331nm) and more excellent water permeability is less rejected by FO membranes irrespective of the FS concentration (Hancock *et al.*, 2011; Roy *et al.*, 2016) (Almoalimi and Liu, 2022). However, it is notable that the rejection of Ammonium was higher when FS3 was concentrated at high osmotic pressure.

CONCLUSIONS

The effect of osmotic pressure gradient on the performance of the membrane was investigated to concentrate different tannery effluents that have undergone secondary treatment. Flux was higher for treated tannery effluents when concentrated at a high osmotic pressure gradient as compared to a low osmotic pressure gradient, irrespective of their composition. FS1 and FS2 flux was higher for FS3 when operated at high osmotic pressure gradient whereas flux was higher for FS1 when compared to FS2 and FS3 when operated at low osmotic pressure gradient. FDR was near same for FS1 when operated at low and high osmotic pressure gradient. In contrast, FDR was higher for FS2 and FS3 when operated at low osmotic pressure gradient as compared to high osmotic pressure gradient experiments. Percentage increase in FS concentration was near the same for FS1, FS2, and FS3 when operated at low osmotic pressure gradient, whereas the percentage increase in FS concentration was higher for FS1 as compared to FS2 and FS3 when operated at a high osmotic pressure gradient. Chromium and Magnesium rejection for FS1, FS2, and FS3 was higher when experiments were conducted at high and low osmotic pressure gradient, and their

rejection was higher at high osmotic pressure gradient than low osmotic pressure gradient. Potassium rejection was very low for FS1, FS2, and FS3 when operated at low and high osmotic pressure gradients. Potassium rejection was negative for FS1 when operated at low and high osmotic pressure gradients. Potassium rejection was higher for FS2 and FS3 when operated at high osmotic pressure gradient as compared to low osmotic pressure gradient. Ammonium rejection was good for FS1 when operated at low and high osmotic pressure gradient. Ammonium rejection was low at a low osmotic pressure gradient for FS2 and FS3 as compared to the high osmotic pressure gradient.

The idea of osmotic pressure difference can be used to control the concentration of draw solution during an FO process, which could be helpful tool in process design and optimization. These results will help develop achievable efficacious osmotic concentrations of varied treated tannery effluents that can produce higher dewatering rates while requiring less solute addition. The design and validation of the osmotic pressure difference as a tool for optimizing an FO concentration process for industrial wastewater applications requires more research.

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