

Forward Osmosis: An Energy-Efficient Approach for the Treatment and Recovery of Resources from the Dairy Industry Effluent

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The study explores implementing the Forward Osmosis (FO) membrane separation process to recover resources from whey, a waste stream in the dairy industry. A Hollow Fibre FO (HF-FO) membrane with membrane area of 13.8 m² was employed, using aqueous NaCl as the draw solution. The optimization study delves into the impact of operating parameters such as mode of FO, concentration and osmotic pressure of draw solution and Transmembrane Pressure (TMP) on overall process efficiency. In the recirculation mode of FO, 24% (w/w) aqueous NaCl draw solution attained an impressive water recovery of 83.06% at a flux of 2.206 LMH (liters per square meter per hour) without any external TMP. With a slight application of TMP of 0.35 bar, water recovery further increased to 94.41%, concurrently achieving a high concentration factor of 17.87 at a flux of 2.462 LMH. Notably, lactose, a value-added product, is concentrated from 3.95% to 49.5% in the concentrated whey stream. The reported FO process exhibited remarkable energy efficiency, achieving a specific energy consumption of less than 8 kWh per ton of water removed. Thus, this approach is resourcefully and economically beneficial for the dairy industry, presenting a sustainable solution with positive environmental implications.

Keywords: Forward osmosis, Industrial wastewater, Membrane separation, Lactose, Whey

Introduction

The increasing global demand for clean water and the challenges posed by population growth and industrialization necessitate exploring non-conventional water resources. Wastewater recovery and reuse are crucial solutions to address the need for clean water and environmental preservation. The Central Pollution Control Board (CPCB) regulations mandate industries to adopt technologies for effluent treatment and reuse. Owing to these regulations, industries seek more efficient technologies for treating wastewater and recovering clean water.

The water demands of various industries, including textile, thermal power, cement, food processing, beverages, paper, dairy, chemicals and leather are substantial, requiring extensive water usage in their daily operations.¹ Among these industries, the dairy sector stands out as one of the fastest-growing, necessitating a significant amount of fresh water for manufacturing, storage, cleaning and utility operations.¹ Consequently, the volume of wastewater the dairy industry generates is notably high.^{1,2} It is reported that most dairy plants generate between 0.2

and 10 litres of wastewater for every litre of processed milk, with an average of approximately 2.5 litres.^{3,4} In a country like India, which processed about 210 million tons of milk in 2020–2021, it is estimated that around 525 million tons of wastewater is generated during milk processing.⁵ The characteristics of dairy wastewater, differ significantly from wastewater generated by other industries due to the presence of easily degradable organics. The organic load varies based on the source, unit operation and technology used in the dairy industry. The dairy industry typically generates two types of wastewater: (a) Product waste stream: Generated during the manufacturing cheese, yogurt, paneer, butter, ghee, skim milk and ice cream^{6–8} (b) Effluent generated in Cleaning In Place (CIP) units: Resulting from the washing of manufacturing units, silos, containers, milk cans, trucks and floors. The product waste stream is reported to be heavily loaded with organic contaminants, in contrast the CIP stream is highly diluted and contains disinfectants and a small amount of milk solids.⁹ Unfortunately, the current practice in the dairy industry involves a mixing approach where the product waste stream and the CIP stream are combined before treatment and forwarded to an Effluent Treatment Plant (ETP) for further processing.

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This mixing practice presents challenges in efficiently treating and recovering resources from distinct waste streams. Despite the advancements in the treatment technologies, the current practices still face challenges in recovering clean water and value-added products from dairy wastewater. The available methods often come with drawbacks, such as low process efficiency, tedious operations, high costs, energy consumption, generation of secondary streams and by-products like sludge, fouling, the requirement of large areas and the generation of foul odours.^{10,11} The treated water is often limited to non-potable applications within the dairy premises. This complexity underscores the need for innovative and sustainable solutions to recover resources from the entire dairy effluent effectively. Efforts to optimize treatment processes, enhance resource recovery and implement sustainable practices in the dairy industry are essential. Addressing the unique characteristics of dairy wastewater and evaluating the mixing practice can lead to more effective and environmental-friendly wastewater treatment solutions.¹¹

The generation of product waste, such as whey and buttermilk, along with wash water containing milk solids and curd particles, contributes significantly to the organic load in wastewater from dairy operations. Whey, a liquid by-product, is produced while manufacturing cheese, paneer and yogurt. It contains more than 90% water content. It typically has a yellowish tinge due to its high Beta-Carotene content, distinguishing it from the white appearance of dairy wastewater from other operations.¹² Whey is a valuable but challenging waste stream due to its organic nature. It comprises approximately 7–8% of total solids, primarily lactose sugar, proteins, vitamins and minerals.¹² The production of 1 kg of cheese or paneer can generate about 9 kg of whey as a waste stream.^{13,14} Despite its valuable components, whey is considered one of the most polluted waste flows due to high levels of Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), turbidity, oils and fats, suspended solids, phosphorus and nitrogen levels.^{14,15} The environmental risk associated with waste whey is significant if directly released into watercourses. Unlike other milk wastewaters, whey is highly resourceful and bio-degradable. Consequently, treating whey separately without mixing it with other dairy wastewater streams is essential. This approach allows for more targeted and efficient treatment processes that can harness the resource potential of whey while mitigating its environmental impact.

Treating whey separately acknowledges its unique composition and allows tailored solutions to extract value from this aqueous stream.

Biological methods can be effective but may have associated challenges, such as converting the lactose into various organic compounds, including greenhouse gases. High-end reactors like anaerobic sequencing batch reactors, anaerobic membrane reactors and granular sludge bed bioreactors have been proposed to treat waste whey.¹⁶ However, these systems can be costly and require skilled operational staff. Additionally, the biological treatment in anaerobic digesters may create a significant environmental footprint and generate secondary digester effluent that requires further treatment before discharge. Protein precipitation and membrane separation are physicochemical methods used to valorise and produce various whey products.¹⁶ Membrane filtration techniques, such as Microfiltration (MF) for removing of fats and bacteria and Ultrafiltration (UF) to fractionate proteins are also studied.¹⁶ High-cost and energy-consuming techniques like Nano Filtration (NF) and Reverse Osmosis (RO) have also been investigated for water separation from whey. The traditional method of processing whey to form whey powder using thermal heating is also mentioned.¹⁷ While these methods have their merits, there is a recognized need for a more cost-effective and energy-efficient technology for recovering water and generating profits from value-added products derived from whey. The challenges lie in finding methods that balance efficiency, cost-effectiveness and environmental sustainability in treating this unique and resource-rich aqueous stream.

Forward Osmosis (FO) indeed presents itself as a promising and energy-efficient membrane separation technology, especially in dairy wastewater treatment. In FO, osmotic pressure gradient is the driving force created by a concentrated draw solution (Fig. 1).⁽¹⁸⁾ This allows movement of water through a semi-permeable membrane from the feed solution towards the draw solution. The ideal draw solution should have high osmotic pressure, low viscosity, cost-effectiveness and be non-toxic and safe. One notable advantage is that FO does not require hydraulic pressure, contributing to its energy-efficient operation as compared to other membrane processes such as UF, NF and RO. FO offers several advantages, including high total solids concentration, excellent

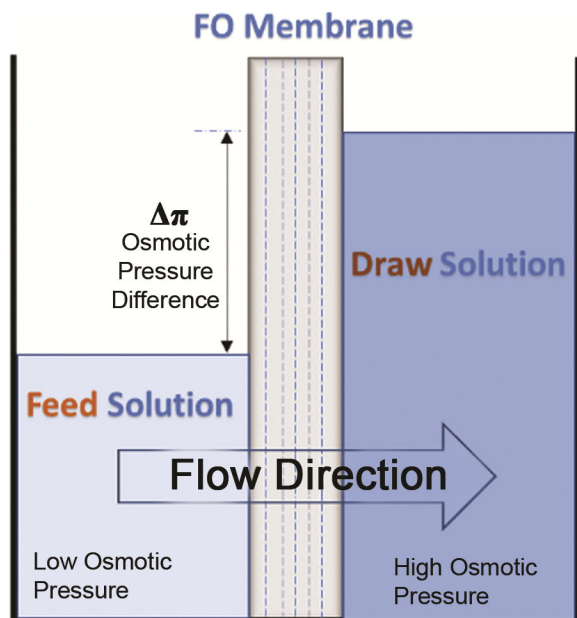


Fig. 1 — Schematic representation of concept of Forward Osmosis

water recovery, enhanced fouling resistance and potentially higher energy efficiency.¹⁹

FO was initially developed for the desalination of seawater.^{20–22} FO has been employed for water reclamation, providing a sustainable approach to treating and recovering water from different sources. FO membranes have concentrated liquid foods and fruit juices.²³ FO's characteristics are advantageous for preserving food's nutraceutical and organoleptic properties. FO has been reported for separating ethanol from water, particularly in producing alcoholic beverages. It concentrates microalgae and removes nitrogen and phosphorus.²⁴ This application is crucial in environmental and wastewater management. FO membrane separation finds applications in concentrating various wastewater streams, sewage, sludge, urine and landfill leachates, showcasing its versatility in managing different types of liquid waste.²⁴ These diverse applications underscore the potential of FO as an efficient membrane separation technology in various fields, addressing water treatment, resource recovery and environmental sustainability challenges. The adaptability of FO to different processes and its ability to handle a range of liquids, makes it a promising technology for addressing complex separation needs across industries and for sustainable and resource-efficient wastewater treatment.

Only some works of application of FO for treatment of whey are reported in earlier research

reports. Implementing FO in whey treatment showcases its potential to concentrate and recover valuable resources from this waste stream efficiently. The literature mentions applying a flat sheet FO membrane and 1.66 M MgCl_2 as the draw solution to achieve four times the whey concentration.²⁵ Utilization of a spiral wound Cellulose Tri-acetate (CTA)-FO membrane and NaCl as the draw solution to achieve a concentration of 6.5–18% for total solids in whey is stated.²⁶ A 24 m² CTA-FO membrane and 48–57 g·L⁻¹ NaCl as the draw solution gave a 2.5 concentration factor and a total solids concentration of 8–21% for skim milk and 5.9–15% for whey.²⁷ Researchers fabricated a Hollow Fibre-Thin Film Composite (TFC) membrane and utilized 0.5 M NaCl as the draw solution to achieve 3.6 times the concentration of whey. The concentration of whey is investigated using the hybrid and integrated FO-RO system and FO-MD system using 2–3 M NaCl as the draw solution.^{28,29} FO-MD achieved eight times the concentration of whey while FO-RO achieved four times the whey concentration and integrated FO-RO achieved 2.1 times the whey concentration.^{29,30} These studies demonstrate the versatility of FO in concentrating whey, with variations in membrane types, draw solutions and achieved concentration factors. The findings contribute valuable insights into the potential applications of FO in the dairy industry for whey treatment and concentration.

This research aligns with the growing interest in sustainable practices within the food and dairy industries. The focus on resource recovery and energy efficiency is crucial for addressing environmental concerns and optimizing the utilization of waste streams. Very few works of application of FO for the treatment of whey are reported to date. This is quite an unexplored research area. The above mentioned literature of the FO treatment of whey, reports very low efficiency in the concentration of whey and no value-added product has been recovered from the waste. The study emphasises on the implementation of FO technology and the subsequent optimization studies provide valuable insights into the potential applications of this approach for treating whey wastewater. It focuses on enhancing the efficiency of dewatering and concentration of whey using FO and importantly, recovering nutrient-rich value-added products from the waste stream and achieving these goals with lower energy consumption. UF pre-treatment of whey was carried out to remove

unwanted contaminants. The pre-treated stream is subjected to FO to concentrate and recover resources such as clean water (in the form of diluted draw solution) and a value-added product. To optimize the process, an investigation of the effects of different factors on FO efficiency was carried out. This study aims to develop a simple, comprehensive and energy-efficient methodology by applying FO membrane technology for treating and recovering resources from dairy wastewater, specifically whey wastewater, which addresses both environmental and economic aspects. Additionally, the recovery of value-added products could have implications for the economic viability of such treatment processes.

Materials, Instrumentation and Methodology

Materials

The various samples of paneer whey wastewater were collected from the nearby dairy industries such as Mahanand dairy in Mumbai, Katraj Dairy in Pune and VIKAS dairy in Jalgaon situated in Maharashtra, India. Aqueous NaCl draw solution was prepared to mimic the brine stream used in the dairy industry using NaCl (99.5% purity) supplied from Ami Chemicals, Mumbai and distilled water. The commercially available surfactants with Sodium Lauryl Sulphate (SLS) purchased from the Ami chemicals were used as the cleaning agents to clean the membrane after every run.

Instrumentation and Analysis

A density meter (Anton Par DMA 5000) was used to measure the density and specific gravity of the feed and draw solutions. The gravimetric analysis used hot air oven and muffle furnace (Thermolyne-F47910-33) purchased from Thermo Scientific, Mumbai. COD was determined using a laboratory COD digester (COD165, 115/230VAC) and spectrophotometer. Nutritional analysis was carried out using the methods mentioned in the Food Safety and Standard Authority of India (FSSAI) manual to analyze foods, such as milk and milk products. A rotatory vacuum distillation system (IKARV 10 digital) evaporates the excess water from the concentrated whey. Freeze dryer (Lyo-works, OS, LABCONCO) was used to lyophilize the concentrated whey. HPLC system (Agilent-1200 series) with RI detector and Bio-Rad Aminex HPX-87 H column (300 mm × 7.8 mm) was used for qualitative and quantitative analysis of lactose in raw wastewater and treated wastewater.

Methodology

Equations used for Experimental Calculations

The average hydraulic pressure difference between the feed and the draw solution of the membrane is defined as the Transmembrane Pressure (TMP). It is expressed by the below Eq. (1)³¹

$$TMP = \frac{P_{Inlet,Feed} - P_{outlet,Feed}}{2} - \frac{P_{Inlet,Draw} - P_{outlet,Draw}}{2} \quad \dots (1)$$

where, $P_{inlet,Feed}$, $P_{outlet,Feed}$, $P_{inlet,Draw}$ and $P_{outlet,Draw}$ are the pressures at feed inlet, feed outlet, draw inlet and draw outlet respectively

Total net Driving Force (ΔP_{net}) is defined using Eq. (2)³¹

$$\Delta P_{net} = TMP + \Delta\pi \quad \dots (2)$$

where, $\Delta\pi$ is the osmotic pressure difference between the feed and the draw solution

Concentration Factor (CF) of the feed was calculated using the following Eq. (3)³¹

$$CF = \frac{M_0}{M_t} \quad \dots (3)$$

where, M_0 and M_t are the initial mass and mass of the feed at the given time 't' during the concentration process

Water Flux (Jw, LMH) through the membrane and **Water Recovery (%)** were determined using below Eqs. (4) & (5) respectively³¹

$$Water\ flux = \frac{\Delta M}{\rho A_m \Delta t} \quad \dots (4)$$

where, ΔM is the water transferred from feed to draw in litres within the time interval Δt , A_m is the area of the membrane and ρ is the density of the water

$$Water\ recovery = \frac{\Delta M \times 100}{M_0} \quad \dots (5)$$

where, M_0 is water in feed inlet

Specific Energy consumed to remove per unit mass of water from the feed E_s (kWh/tonnes water removed) was estimated based on the electric power P_e , consumed by the pumps to lift feed and draw solutions to the membrane and expressed by Eq. (6)³¹

$$E_s = \frac{P_e \cdot t}{M_0 - M_t} \quad \dots (6)$$

where, t is the time required to reduce the mass of the feed solution from M_0 to M_t . P_e is determined using the details specified by manufacturer.

Osmotic Pressure (π) for any solution is calculated by Eq. (7) ³¹

$$\pi = \frac{-RT \ln(a_w)}{V} \quad \dots (7)$$

where, π is the osmotic pressure (atm), R is the universal gas constant = 0.082 L atm·mol⁻¹·K⁻¹ and T is the temperature in Kelvin, a_w is the water activity (Fig. S1 (A) and S2 (A)) represents the variation of water activity along with the concentration of solute for aq. NaCl solution and whey respectively), V is the molar volume of water = 0.018 L·mol⁻¹. Using eq. (7) and eq. (S1) and (S2), relationship between the concentration and osmotic pressure of aq. NaCl solution is represented in Fig. S1 (B) while for whey is represented in Fig. S2 (B).

Pre-treatment of Whey

The process is designed to enhance the probability of recovery of value-added product from whey by removing undesirable components, such as proteins and solids, through filtration and ultrafiltration. The hot raw paneer whey wastewater, with a temperature between 50–60°C, was cooled to room temperature. The cooled wastewater was filtered through a nylon filtration cloth of 200 mesh (Fig. 2 (a) and (b)). This step is aimed at separating suspended solid particles from the wastewater. The cloth-filtered raw whey (Fig. 2 (c)) undergoes further pre-treatment using a UF membrane. This step helps to remove proteins and other dissolved solids that could cause membrane fouling during subsequent processes. The cloth-filtered raw whey was recirculated through an

assembly consisting of a cartridge filter and a dialyzer membrane. A low flux dialyser UF membrane made of Fresenius polysulphone with a polycarbonate housing and membrane area of 1.3 m² and a Molecular Weight Cut Off (MWCO) in the range of 10⁴ to 50×10⁴ Da was used (Fig. 2 (d)). The UF process operates under an external pressure of 2 bar in cross-flow mode. After the ultrafiltration of whey, it was observed that the retentate stream of the UF process was thicker than the feed and permeate (Fig. 2(e) and 2(f)). This thickness of retentate stream indicates the removal of various solids from the whey.

Experimental Setup

The commercial Hollow Fibre Forward Osmosis (HF-FO) membrane procured from M·s⁻¹ Aquaporin, Denmark was supplied feed and draw solutions using the centrifugal pumps purchased from Arya Analytical Services, Mumbai. Valves, connecting pipes, containers and pressure gauges (HYZA: 0–20 bar) were all purchased from the local market. The module (Aquaporin Inside™) used in the present study has active surface area of 13.8 m². The TMP for the membrane is 0.2 bar within the pH range of 3–9 with the maximum allowed operating pressure of < 2 bar on both the feed and draw sides. The pilot scale forward osmosis plant was assembled by using centrifugal pumps (280 W), connecting pipes, joints, a feed tank (100 L), draw tanks (100 L) and membrane module. The overall setup can be operated in two modes (a) Continuous Single Pass (CSP) mode and (b) Recirculation (RC) mode (Fig. 3 (A) and Fig. 3 (B)).



Fig. 2 — Pretreatment of raw paneer whey (a) Filtration using 200 mesh nylon filter cloth (b) Dried suspended matter of whey collected from the filter cloth (c) Permeate of filter cloth filtration (used as feed of UF process) (d) Membrane used for UF and permeate of UF (used as feed of FO process) (e) Reject or Retentate of UF of whey (f) Comparison showing difference in colour and texture of whey after UF process

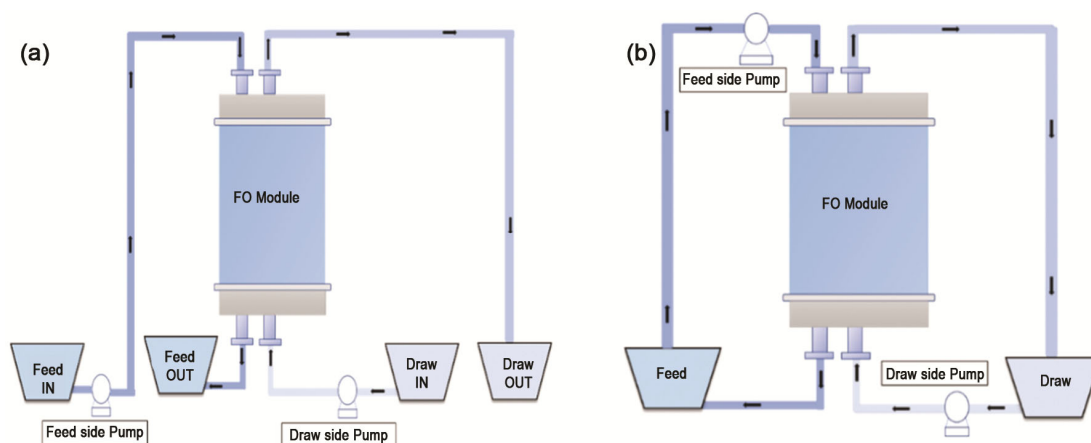


Fig. 3 — Schematic representation of modes of operation of FO (A) Continuous Single Pass (CSP) mode (B) Recirculation (RC) mode

Table 1 — Physicochemical and nutritional analysis of whey samples

Parameter	Unit	Sample-1	Sample-2	Sample-3	Mean \pm SD of sample values
pH	—	4.4	4.2	4.2	4.27 \pm 0.12
Total Suspended Solids (TSS)	%	2.01	1.54	1.35	1.63 \pm 0.34
Total Solids (TS)	%	6.2	5.9	5.95	6.02 \pm 0.16
Ash content	%	1.56	1.37	1.49	1.47 \pm 0.10
Moisture content	%	93.8	94.1	93.4	93.77 \pm 0.35
Electrical conductivity	ms \cdot cm ⁻¹	13.45	10.21	7.69	10.45 \pm 2.89
COD	mg \cdot L ⁻¹	1,20,000	95,000	92,045	102348 \pm 15358
Density	kg \cdot L ⁻¹	1.0325	1.0198	1.0120	1.02 \pm 0.01
Specific gravity	—	1.02854	1.01812	1.01714	1.02 \pm 0.06
Lactose	%	3.7	3.85	3.95	3.83 \pm 0.13
Carbohydrate	g \cdot 100 mL ⁻¹	2.92	3.18	2.85	2.98 \pm 0.17
Energy	kcal \cdot 100 mL ⁻¹	16	19	15	16.67 \pm 2.08
Fat	g \cdot 100 mL ⁻¹	0.62	0.58	0.65	0.62 \pm 0.04
Phosphorous	g \cdot 100 mL ⁻¹	0.006	0.009	0.008	0.01 \pm 0.00
Protein	g \cdot 100 mL ⁻¹	0.83	0.82	0.9	0.85 \pm 0.04
Calcium	mg \cdot kg ⁻¹	1046	1125	1098	1089 \pm 40
Magnesium	mg \cdot kg ⁻¹	105.4	120.6	115.7	113.90 \pm 7.76
Potassium	mg \cdot kg ⁻¹	998	885	904	929 \pm 60
Sodium	mg \cdot kg ⁻¹	376	412	395	394 \pm 18
Zinc	mg \cdot kg ⁻¹	1.3	1.1	1.4	1.26 \pm 0.15

Results and Discussion

Characterisation and Analysis of Whey

The analysis of the samples of whey effluent collected in different months from the Mahanand dairy industry, Mumbai is shown in Table 1 below. The reported values of Sample-1, Sample-2 and Sample-3 are the average of the 3 values obtained after the analysis of each sample. The methods used to analyse these parameters is shown in Tables S1. From Table 1 below, it is observed that most of the parameters for the samples had similar physicochemical and nutritional constituents. Some variation is observed due to the nutritional variation in

the milk feedstock used for the upstream processing.^{32,33} The nutritional analysis showed that whey has very high moisture content, various water soluble vitamins, minerals and a considerable amount of lactose. The high COD of whey confirms its organic load of dairy wastewater effluent. It is noted that all the analysed samples depicted ~4% lactose content and > 90% moisture content, reflecting its potential for recovering water and lactose as a value-added product.

Pre-treatment of Whey

The proximate and ultimate analysis depicted the significant amount of TS and TSS in the collected

samples. Thus, all particulate matter must be removed to forward it to the next membrane separation unit. The removal of TSS will protect the membrane module from clogging and fouling due to the suspended particles.^{34,35} Thus, removing the unwanted solids is an essential step before approaching forward osmosis. The UF process was selected for the treatment based on the physical and chemical properties of the suspended particles (paneer debris) and dissolved substances (proteins, minerals, vitamins, and lactose). UF membrane processes the whey complex by blocking whey proteins by the size exclusion and leaving the module as the concentrate; lactose and mineral-rich solution leave the UF membrane as permeate.¹⁴ A high quantity of lactose makes it a probable value-added product. This pure protein finds many applications, which is not discussed here as it is out of the scope of this study.

The study removed 2.1% of suspended particles in the whey using nylon cloth filtration. The UF process was run for around five hours without considerable pressure rise observed during the experimentation. Membrane selectivity of 96.15% (calculated based on permeate volume) and water permeate flux (J) of approximately 4.456 LMH were achieved during this work. This UF process was carried out in recirculation mode; hence, the flux obtained in this process was relatively less than that reported in the literature works.³⁶ Lactose moved to the permeate solution while protein was transferred to the reject/ retentate of the UF process. The 30.123 L feed supplied to UF membrane was reduced to 1.158 L in 5 hours. The UF permeate used as a feed to the FO membrane process comprises 4.95% total solids, 3.95 % lactose and 0.2% protein. Obtained results were confirmed by the studies found in the literature stating that the low molecular weight compounds (lactose sugar) were preferably in the permeate of the UF membrane and UF membrane retain the protein molecules.^{14,32}

Experimental Trial of FO Process

The HF-FO membrane of 13.8 m² membrane area was used for the FO process trials. Pre-filtered whey was used as the feed solution and aq. 2.9% (w/w) NaCl was used as the draw solution for the first trial. Initially, a 2.9% (w/w) draw concentration was selected because the same draw concentration was used to calculate the water flux and specific reverse salt flux in the standard manual by the manufacturer. Even in the available literature, the minimum salt concentration used for the draw was observed to be 0.5 M NaCl. Initially the experiments were conducted at room temperature in the CSP mode without any external pressure. In this study, water permeate flux of 0.431 LMH is achieved with dewatering of 10.96 % at CF of 1.123. These results successfully demonstrated the concentration of whey but were significantly less efficient for the process; henceforth, optimization is carried out to increase the efficiency of the process.

Optimization of Parameters

Effect of Mode of Operation of FO

The mode of operation such as CSP and RC, are the critical parameters for the operation of membrane-driven processes.³⁷ The requirement of the area of the membrane and resulting process outputs are the parameters dependent on the mode of operation.³⁸ Thus, the operation mode contributes to the overall process's operating and capital costs. It is an integral part of the membrane process optimization study to select the appropriate mode of operation and study its effect on the process efficiency and the quality of the output products. Thus, in the present study, the FO trials of UF pre-treated whey effluent were initially conducted for performance evaluation under the CSP and RC modes. The influence of the mode of operation on flux and dewatering with variable TMP is shown in Fig. 4. It is noted that there is no

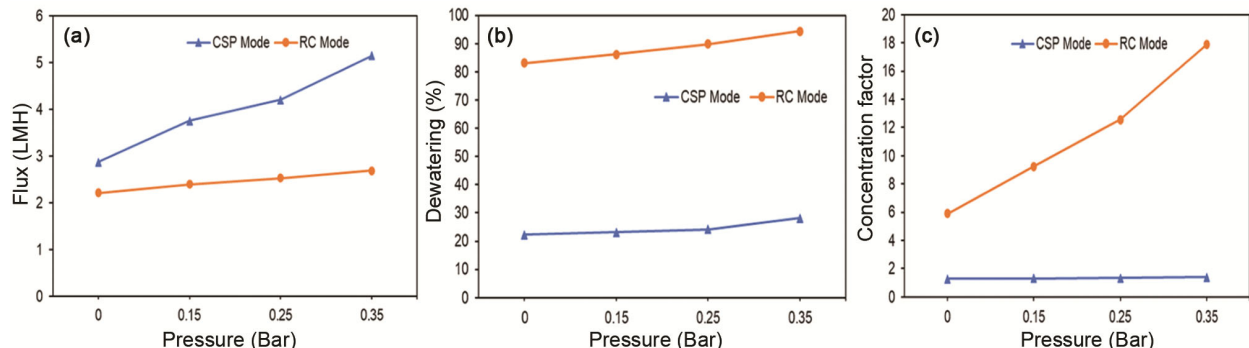


Fig. 4 — Effect of mode of operation on efficiency parameters of FO along with variation in external pressure

remarkable effect on the resulting flux when the trials were conducted in the RC mode of operation with the variable TMP. The RC mode of operation shows > 2.22 LMH flux, while the CSP mode shows > 2.87 LMH flux under the tested conditions (Fig. 4a). It is observed that the flux increases more rapidly with the increase in TMP in the CSP mode of operation than the RC mode. This could be observed because the dilution of the draw solution in the RC mode with the progress of FO reduces the osmotic pressure gradient between the feed and the draw solution. The observed phenomenon agrees with the general operating phenomenon of the FO membrane.³⁹ The analysis of dewatering concerning the mode of operation and TMP depicted higher and more consistent water recovery in the case of the RC mode of operation. The RC mode of operation shows > 83% water recovery, while CSP results in < 30% water recovery under the tested experimental conditions (Fig. 4b). This could be observed due to the higher contact time at constant operating volume with the membrane surface during the RC mode, which tends to extract more water than the CSP mode. The CF increases proportionately along with the feed concentration concerning the TMP in RC mode, rising from 5.9 to 17.88 while in CSP mode, the CF increases from 1.28 to 1.39 only

(Fig. 4c). Thus, to achieve maximum water recovery, RC mode is preferable. Hence, the RC mode of operation is selected for the further process intensification study.

Effect of Concentration of Draw Solution

The concentration of the draw solution directly impacts the osmotic pressure, a significant constraint to achieving the maximum efficiency of the FO process.^{23,39,40} The influence of the concentration of draw solution on flux and dewatering is as shown in Fig. 5. Graphs of the relationship between the osmotic pressure and solute concentration (Fig. S1) are drawn. At room temperature, high osmotic pressure (π) of 325.17 bar is reported at the 24 % (w/w) solubility of NaCl. So, for the FO trials, concentration of draw solution was varied in the range of 2.9% (w/w) to 24% (w/w). At 2.9% (w/w), 10% (w/w) and 24% (w/w) concentration of draw NaCl solution, osmotic pressure varies as 23.30, 92.48 and 325.17 bar respectively (Fig. S1). The whey having ~7 % (w/w) total solids, reports an osmotic pressure of 6.768 bar (Fig. S2). This high osmotic pressure gradient between the feed and draw solution ($\Delta\pi$), drives the FO process to work efficiently along with the other parameters. Even in the RC mode, when the low

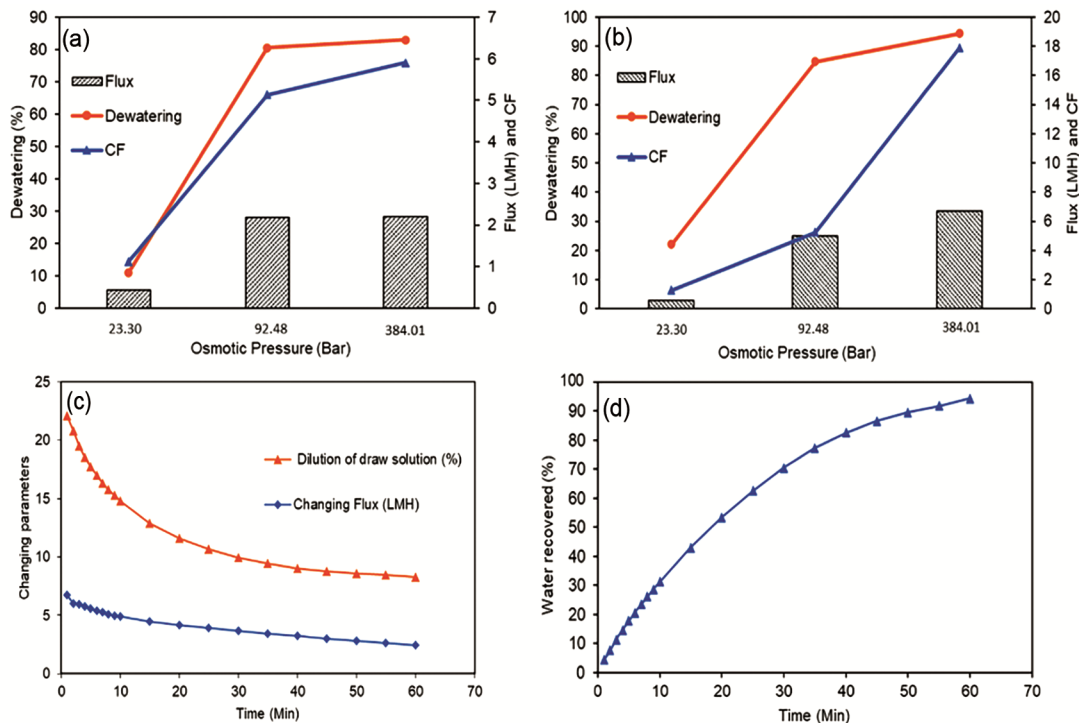


Fig.5 — Effect of varying concentration and osmotic pressure on dewatering and flux of whey (a) Without applying any external TMP and (b) at 0.35 bar TMP, (c) Variation in flux and changing concentration of draw solution along with the progress of FO at 0.35 bar TMP, (d) Variation in water recovery along with the progress of FO at 0.35 bar TMP

osmotic pressure feed whey keeps on concentrating, it does not reach the osmotic pressure as high as the diluted salt solution and this keeps the process going.

It is reported that the FO-driven processes are favoured mainly by the concentration gradient. However, the externally applied pressure can also provide additional driving force for higher mass transfer.⁴¹ So, in the present study observations are made without applying any external TMP and at slight external TMP of 0.35 bar along with the increasing concentration of draw solution. Without applying any TMP (Fig. 5a), dewatering increased from 10.96 to 30.53, reaching up to 83.06%. At the same time, CF varies as 1.123, 1.439 and 5.903 and flux rises from 0.431, 0.83 up to 2.206 LMH with increasing concentration of draw solution of 2.9% (w/w), 10% (w/w) and 24% (w/w) respectively (Fig. 5a). Similar trend is observed after applying the slight external pressure of 0.35 bar with dewatering efficiency rising from 14.09 to 94.41%, CF rising from 1.164 to 17.889 and flux varying from 0.56 LMH to 2.462 LMH along with the increasing concentration of the draw solution (Fig. 5b). Thus, higher concentration of draw should be used to achieve the maximum water flux.

In this scenario, the draw solution was in contact with the membrane's support layer. As the FO process continues, the water permeating from the feed side dilutes the draw solution, leading to a decrease in flux, as shown in Fig. 5(c). Conversely, this dilution results in an increase in water recovery, as depicted in Fig. 5(d). The higher water recovery reduces the effective draw solution concentration at the membrane interface, which in turn lowers its contribution to the overall osmotic driving force. This also causes Internal Concentration Polarization (ICP) at the membrane surface. Consequently, the theoretically estimated high osmotic pressure is not fully realized in practice. Therefore, even with a significant osmotic pressure difference between the 10% (w/w) and 24% (w/w) salt concentrations, the increase in efficiency does not scale proportionately with the osmotic pressure rise. Nevertheless, due to its maximum efficiency, the saturated 24% (w/w) draw solution concentration was selected for further study.

Effect of Transmembrane Pressure (TMP)

The total driving force, ΔP_{net} , can be adjusted by varying either the osmotic pressure difference ($\Delta\pi$) or the TMP as described in Eq. (2). $\Delta\pi$ is modified by

changing the concentration of the draw solution, while TMP is altered by applying external pressure to the membrane. Under standard conditions, an osmotic pressure gradient drives clean water across the FO membrane without the need for external hydraulic TMP. However, when pressure is applied on the feed side of the membrane, the driving force for water transport through the membrane increases, leading to a higher water permeate flux. Although applying a small amount of hydraulic pressure requires additional energy, this can be offset by lower capital investment costs such as reduced membrane surface area, smaller circulating pumps, and fewer connecting pipes and further savings in the subsequent process.³⁸ However, applying hydraulic pressure has both positive and negative impacts on membrane performance. While increased flux can enhance performance, it also exacerbates Internal Concentration Polarization (ICP). Additionally, pressurizing the membrane with feed water can reduce the porosity and interconnectivity of the membrane pores, thus decreasing fresh water transport. Therefore, in this study, only a small amount of TMP is applied to assess its impact on performance efficiency.

As TMP increases from 0 to 0.35 bar (in increments of 0, 0.15, 0.25, and 0.35 bar), dewatering efficiency correspondingly rises from 83% to 94.41% (Fig. 6a). Simultaneously, the flux increases from 2.2 to 2.462 LMH, and the Concentration Factor (CF) elevates from 5.9 to 17.88 (Fig. 6A). The water flux is calculated based on the draw solute concentration and hydraulic pressure, demonstrating that both water flux and draw volume increase with applied pressure over time for a constant feed volume (Fig. 6c). However, this relationship is not linear due to the impact of ICP. As the feed become more concentrated, the effective pressure on both sides of the membrane decreases, leading to a reduction in water flux (Fig. 6b). Pressure-Assisted Osmosis (PAO) enhances water flux, but this increased flux reduces the effective draw solution concentration at the membrane interface, thereby diminishing its contribution to the overall osmotic driving force. This study highlights the significant role TMP plays in achieving maximum dewatering efficiency, flux, and CF. The findings suggest that increasing TMP by raising the feed side pressure, while maintaining a constant osmotic pressure difference ($\Delta\pi$), significantly boosts flux and water recovery.

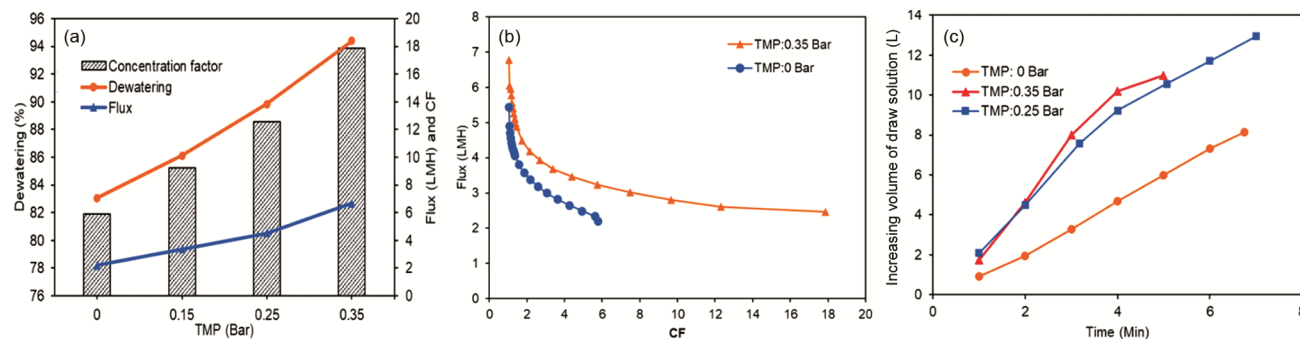


Fig. 6 — (a) Effect of TMP on variation of CF, dewatering and flux in RC mode for 24% (w/w) NaCl solution (b) Variation in water flux observed for different CF of whey at TMP of 0.35 bar and 0 Bar (c) Variation observed in volume of draw solution by varying TMP at 0, 0.25 and 0.35 Bar

Cleaning of Membrane and Analysis of Products of FO

A cleaning protocol was established to clean the membrane, an important aspect of the membrane separation process. After experimental trials, the membrane was thoroughly cleaned with non-harsh bleach-free detergent to restore its performance. It was passed through both, the shell and lumen sides of the membrane. The change in consistency and smell of the detergent solution observed as it passed through the shell side of the membrane was found to be negligible. After 20–25 minutes of the cleaning procedure, alternative cleaning with lukewarm water (< 40°C), detergent (pH: 2–7) and freshwater, the smell of whey from the outlet water of the lumen side disappeared. The TDS of the feed outlet and draw outlet water were almost equal to the fresh inlet water. In addition, similar water flux (11 LMH) of the system was observed before and after cleaning the system. This made it evident that, unlike other membrane processes, foulants form a sparse and loose layer in FO, which can be removed by following proper cleaning protocol so that the membrane is ready to use again.

Diluted Draw- The outlet draw, i.e., diluted NaCl solution, can be used as food processing ingredient and so must satisfy the stringent quality constraints of the food industry. Hence, any contamination of the draw solution can be deleterious. The density was checked to validate that only pure water permeated from the feed whey to the salt draw solution. HPLC analysis was conducted to check the presence of macromolecules of any lactose permeated through the feed. No change in the draw colour was observed after the FO run, indicating that no solute was transferred from the feed to the draw side. The density of the 24% (w/w) NaCl solution used as a draw solution was 1.177 kg·L⁻¹, while the reported density

of the diluted draw solution was 1.06270 kg·L⁻¹. The HPLC analysis of the diluted draw ensured that significantly very low 0.06% lactose moved from the feed to the draw side of the membrane.

Concentrated Feed- The raw whey with 3.95% lactose had a density of 1.0120 kg·L⁻¹, while the concentrated one had density of 1.1884 kg·L⁻¹. When the same amount of extracted water is added back to the concentrated feed, the makeup feed whey density was 1.0125 kg·L⁻¹, similar to the raw whey. This shows that only pure water migrated from the feed to the draw, excluding the other macromolecules like lactose.

Recovery of the Value-added Product

It was noted that the concentrated feed depicted the presence of lactose, which can be a value-added product and can be reclaimed from the stream. So, the concentrated feed was then dried and converted into a powder form using the process of lyophilisation. The analysis of the cream coloured dried powder confirmed all the contents of this recovered powder (Table S3). The HPLC analysis of this dried concentrated whey confirmed that the initial ~3.95% lactose is concentrated to 49.2% (Table S2, Fig. S3A and S3B). The results indicate that the recovered powder has a high percentage of lactose along with calcium (4170 mg·kg⁻¹), potassium (4821 mg·kg⁻¹) and other minerals. It can find potential applications in dairy, pharmaceutical and confectionary industries as per the required application.

Energy Consumption

Specific energy consumed in the FO step in this process is calculated as less than 8 kWhr/tonnes of extracted water from the whey as per Eq. (6). This energy consumption is lower than reported in the literature as explained in Table 2 below.

Table 2 — Energy consumption involved in various processes of whey treatment

Process	Energy consumption	Reference
Vacuum evaporation (33%) conversion efficiency of thermal to electrical energy)	To remove one tonne of water, it typically requires between 80 kg and 330 kg of steam, which is equivalent to 17 kWh to 70 kWh of electrical energy.	42
RO	~9–20 kWh/tonnes of water removed	43
FO	<10 kWh/tonnes of water removed	31
FO	<8 kWh/tonnes of extracted water from the whey	This research study

Conclusions

In this study, pre-treatment of dairy waste whey using filter cloth and UF membrane reduces the total solids and successfully recovers clean water and value-added product from it using forward osmosis process. This FO process extracts the water from whey and converts it into a value-added product with 49.5% lactose content, high in calcium and potassium content which can find application in various industries or animal feed. Most efficient dewatering of 94.14% and flux of 2.426 LMH is obtained on application of slight external TMP of 0.35 bar to the FO membrane. The consumption of the minimal specific energy of less than 8 kWh/tonnes of water extracted from the whey makes this an energy efficient process. This process, in turn, benefits the entire economics of the dairy industry by reducing the quantity of wastewater generated and to be treated in the ETP, affecting the water and energy consumption of the dairy industry and indirectly providing various environmental benefits.

Supplementary Data

Supplementary data of this article is available at <https://nopr.niscpr.res.in/handle/123456789/63168>

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