

Forward Osmosis: An Approach to Reclaim Dairy Waste Stream Whey

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Abstract

Recognizing the issues with conventional water resources and stricter wastewater effluent disposal standards, the treatment and recovery from wastewater are gaining impetus. The dairy industry consumes a substantial amount of water and generates a massive quantity of wastewater annually. Whey, which is about 94% water, is a waste stream produced in the dairy industry during the manufacture of cheese, paneer, yogurt, etc. Although various wastewater treatment technologies are available in the market, membrane technologies are considered the most advanced and reliable ones, but they are expensive. In recent years, Forward Osmosis (FO) is looked upon as a potential alternative to these costly and energy intensive pressure driven membrane processes. FO works on the principle of natural osmotic pressure where energy is just required to lift the solutions. The present lab-scale study investigates the partial reclamation of water from whey using FO technology. The Continuous Single Pass (CSP) and Recirculation mode (RC) study is conducted using high osmotic pressure ($\pi = 375$ bar at 298K) saturated aqueous(aq.) NaCl as the draw solution. The aq. NaCl solution is a potential brine stream in the dairy industry and finds applications in the manufacture of paneer, butter, cheese and ice cream eliminating the need for draw regeneration. The back diffusion study of the Hollow Fibre Forward Osmosis (HFFO) membrane revealed about 0.82% back diffusion of solute. The maximum water recovery of $\sim 56\%$ is achieved in CSP mode while 57.6% is achieved for RC mode with Feed/Draw ratio of 4.5:1. For F/D of 10:1, the maximum permeate flux of $\sim 8.7 \text{ kg m}^{-2} \text{ h}^{-1}$ is observed for the CSP mode of operation for 10 minutes of study. Thus, FO is an efficient membrane technique that eliminates the need for draw regeneration and can be applied in the dairy industry.

1. Introduction:

1.1 General

Due to the population explosion and rise in the standard of living, the demand for freshwater is increasing rapidly (Elimelech & Phillip, 2011; Ward & Pulido-Velazquez, 2008). Knowing the issues with the availability of conventional water resources and wastewater effluent disposal standards, the reuse and recovery from wastewater is gaining impetus (Jury & Vaux, 2005; Shon et al., 2015). These non-conventional water resources provide water security and are considered a reliable alternative in many countries (Khawaji et al., 2008).

Industries consume a vast quantity of fresh water and generate a colossal amount of wastewater. The dairy industry, in particular, consumes about $1-10 \text{ m}^3$ of water per m^3 of processed milk (Wojdalski et al., 2013). The effluent of dairy primarily includes milk or milk products particles, by-products of the manufacturing process like whey, contaminants left after washing of cans, tanks, equipment, floors and after Cleaning In Place (CIP) operations (Kolev, 2017). It has high Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Dissolved Solids (TDS), pH and oil as well as grease. Thus letting it out without any treatment creates eutrophication in the environment (Al-Shammari et al., 2015; Kolev, 2017).

1.2 Whey

Whey is the liquid by-product generally produced during the manufacture of cheese, paneer, yogurt etc. It is the liquid remaining after the removal of fats and caseins from the milk (Wit. JN, 2001). Typically, white colour is observed for dairy wastewater and greenish yellow for whey (Duke & Vasiljevic, 2015). The global annual whey production is approximated to be over 10^8 tonnes per annum (Grba et al., 2002). A typical whey has approximately 93–94% water content and around $7.12 \pm 0.52\%$ of total solids content (Kolev, 2017), constituting a high percentage of lactose. In contrast, proteins, fats and salts are significantly less in composition (Healy et al., 2007). This organically loaded whey is not reused in many dairies and is just treated with dairy wastewater. It has high COD and BOD and thus creates a significant load on the dairy wastewater treatment plant (Kavacik & Topaloglu, 2010). Whey can be processed in mainly three ways—valorisation of whey to recover lactose and proteins, biological treatment and physicochemical treatment (Aydiner et al., 2013). Biological treatment, such as hydrolysis of lactose and proteins produces lactose monosaccharides (glucose and galactose), peptides and amino acids. While controlled fermentation can produce lactic acid, ethanol, and hydrogen. Physicochemical treatments include coagulation, flocculation, ozonation, oxidation, precipitation, gasification, etc (Aydiner et al., 2013, 2014). Among the various whey processing techniques stated above, membrane technologies are considered the most advanced and reliable (Haupt & Lerch, 2018; Shon et al., 2015). Since the 1960s, there has been much development in membrane technology because of the high investment in research and development for membranes (Shon et al., 2015). Membrane separation techniques such as microfiltration (MF), ultrafiltration (UF) and nanofiltration (NF) are extensively used in the dairy industry to separate various whey ingredients such as fats, proteins, lactose and minerals. Reverse Osmosis (RO) is also used for the removal of water to attain a certain Total Solids (TS) content before the concentrated stream is fed to a multiple-effect evaporator and eventually to a crystallizer to obtain the crystallized product (Wit. JN, 2001). All these membrane techniques require hydraulic pressure to operate and are also prone to membrane fouling (Shirazi et al., 2010). So the major issues which plague the membrane process are its high cost, high energy consumption and the high tendency of fouling (Shon et al., 2015).

1.3 Forward Osmosis

Amongst these prevailing expensive and energy consuming membrane technologies, Forward Osmosis (FO) is looked upon as a potential alternative in recent years to replace the costly pressure driven membrane processes (Honmane et al., 2020; Mogha, 2020). FO is another membrane-based separation technique that is a thermodynamically spontaneous process, unlike its hydraulic pressure-driven counterparts. In FO, the driving force is the high osmotic pressure (π) gradient generated by the concentrated draw solution to allow fresh water to permeate through the semi-permeable membrane from the feed solution towards the draw solution (as shown in Fig. 1) (Aydiner et al., 2014; McCutcheon et al., 2005; Tang et al., 2010). The products of FO are a diluted draw solution and a concentrated feed solution (Zhao et al., 2012). The phenomenon of FO can be explained by the second law of thermodynamics. In other membrane-based techniques such as RO, entropy is lowered; hence work has to be done on the system (i.e., hydraulic pressure). But in FO, the total entropy of the system is raised; thus no hydraulic

pressure is required. On the contrary, since entropy is generated in FO, some work can be extracted from the process, and that is the principle used in harnessing the salinity gradient energy, also called blue energy.

There have been a few reported works on partial dewatering of whey using FO. Using flat sheet Cellulose Triacetate (CTA) membrane of 140cm^2 and RC operation mode along with 3M and 2M NaCl solution as the draw, the draw regeneration is done using RO and Membrane Distillation (MD) respectively (Aydiner et al., 2013). Using 4M NH_4CO_3 as the draw solution and the CTA membrane, the draw was regenerated using thermal decomposition methods (Seker et al., 2017). The RC mode has been reported with the draw solution of concentration 1M NaCl (Wang et al., 2017) along with Thin Film Composite (TFC) membrane of area 106 cm^2 , 60°Bx Potassium Lactate solution (Menchik & Moraru, 2019) coupled with spiral wound CTA membrane of 0.5 m^2 and 50 gL^{-1} NaCl draw solution for CTA membrane of 12 m^2 (Chen et al., 2019) with no draw regeneration has been reported. The majority of the studies have been carried out using the RC mode of operation along with no draw regeneration or regeneration using some energy-consuming techniques.

In this study, an attempt has been made to propose a continuous process to partially recover water from the dairy waste stream whey by using FO and the value addition to the draw to eliminate the need for regeneration. Sodium Chloride (NaCl) is one such solute that not only finds application in the dairy industry (Wit.J.N., 2001) as the potential brine but is also the potent osmotic agent. So, applicability of the high osmotic pressure aqueous (aq.) NaCl as the draw solution for the partial concentration of whey has been investigated. The study is a novel work carried out to compare the Continuous Single Pass (CSP) mode and Recirculation (RC) mode of FO on commercial FO membrane with an aim to achieve maximum water recovery and permeate flux. Experiments are also carried out to capture the effect of Feed/Draw (F/D) ratio on the performance of tFO process.

2. Materials, Instrumentation And Methodology:

2.1 Materials and Instrumentation

The whey, which is used as the feed of FO in this research was obtained from the local dairy, Shiv Dairy Farm, Guru Teg Bahadurnagar, Mumbai, India. Physiochemical analysis of the dairy whey is carried out further to determine its initial composition. The aq. NaCl solution (24% w/w) having high osmotic pressure ($\pi = 375\text{ bar}$ at 298K) (Honmane et al., 2018) which resembles the dairy brine stream is used as the draw solution. A nylon filtration sheet of 200 mesh (filter cloth) filters the whey and removes any dirt or solid particles prior to the FO process. Two peristaltic pumps supplied by Arya Analytical Services are installed to lift the solutions to the membrane. The aquaporin commercial Hollow Fibre Forward Osmosis Membrane (HFFO) of active area 2.3 m^2 is used for this study. The Anton Par DMA 5000 density meter measures the density and specific gravity of the inlet and outlet feed and draw solutions at the room temperature of 25°C .

2.2 Methodology:

Initially the physiochemical analysis of the whey is carried out to determine the composition of the whey. The multimeter purchased from Hanna is used to measure the pH, electrical conductivity, temperature and Total Dissolved Solids (TDS) of the whey. Total solid content and moisture content is determined using the thermogravimetric method by heating in the oven at 105°C and ash content by heating at 600–650°C in the muffle furnace. Using Anton Par DMA 5000 density meter, density and specific gravity were measured at 25°C, whereas lactose content was determined using the HPLC method.

The back diffusion study of the membrane is carried out to check the diffusion of the solids from the draw to the feed side of the membrane.

The schematic representation of the CSP and RC mode is as shown below in Fig. 2 and Fig. 3 respectively. The CSP has a separate chamber for feed inlet and outlet and draw inlet and outlet, unlike in RC mode where the feed and draw is recirculated continuously. The CSP mode study is carried out for 10 minutes whereas RC mode is conducted for 30 minutes. The Feed Inlet (Feed In) used in this study is the dairy whey whereas the Draw Inlet (Draw In) is saturated NaCl solution. The effect of various F/D on the percentage water recovery and permeate flux is also observed. After the FO process we get concentrated feed and the diluted draw solution.

This diluted draw of aq. NaCl need not be regenerated after the FO and can be directly used to make cheese in the dairy. Salt brining is used in cheese production to inhibit the growth of bacteria and add flavour to it. In the brining process, the cheese is dipped or submerged in the brine solution (20 % NaCl) for some days at 13°C and the same brine can be used for about 1–2 years (Guinee, 2004; Modler et al., 1990). In the manufacturing of butter, 10% (w/w) salt solution is used as the preservative and also added to increase the product shelf life (Hoecker, 1941). The remaining brine can be reconcentrated and reused in the FO itself. Thus the whey and the brine solution both can be eliminated from the dairy wastewater. This will in turn reduce the huge volume of problematic dairy wastewater to be treated.

3. Experimental Study:

3.1 Back Diffusion Study:

The back diffusion study is conducted to understand the permeation of solids from draw to feed side of the membrane. The study is conducted in CSP mode with F/D ratio of 8 with deionised (DI) water as the feed and the 10% (w/w) NaCl solution as the draw solution. The variation in TDS was observed throughout the study. The pattern of reduction of weight of feed was also observed and analysed through graph plotting.

3.2 FO Study:

Calculation of Osmotic Pressure of feed and draw solution for FO:

The osmotic pressure of the feed whey and draw NaCl solution is calculated using equation no (1) adapted from Wolfe et al., 2011.

where, π is the osmotic pressure (Bar), a_w is the water activity co-efficient, V is the molar volume of water = 0.018 Lmol^{-1} , R is the universal gas constant = $8.3145 \text{ J.mol}^{-1}.\text{K}^{-1}$ and T is the temperature in Kelvin scale (Aydiner et al., 2014).

For whey, the experimental value of a_w as a function of total solids (%w/w) at ambient temperature are calculated by equation no (2) adapted from (Kanterewicz & Chirife, 1986). Subsequently, osmotic pressure is calculated from above equation no (1)

The graph 1 below indicates the relationship between osmotic pressure and whey concentration (%w/w).

For aq. NaCl solution, the value of a_w can be applied in the range of 15–50°C (Chirife & Resnik, 1984). The experimental data was fitted to equation no (3) with $R^2 = 1$.

The graph 2 below indicates the relationship between osmotic pressure and concentration of aq. NaCl solution (%w/w) by substituting the values obtained by equation no (3) in equation no (1).

The permeate flux and water recovery are calculated using below equations referred from (Honmane et al., 2018,2020):

The density of the feed and draw solutions at inlet and outlet and of make-up feed solution was checked using a density meter to confirm the permeability of the pure water from the feed to the draw side.

4. Results And Discussion:

The results of the physicochemical analysis of the raw whey are as indicated in the Table 1 below. It stated that the whey is highly acidic and has very high moisture content as compared to the total solids content.

Table 1
Physicochemical characteristics of raw whey obtained from the dairy

Sr.No.	Parameter	Unit	Value
1.	pH	-	2.9
2.	Total Solids	%	5.1%
3.	Total Dissolved Solids	mg L ⁻¹	30,000
4.	Ash content	%	1.17
5.	Moisture content	%	94.9%
6.	Electrical Conductivity	ms cm ⁻¹	7.69
7.	Temperature	°C	30.6
8.	Chemical Oxygen demand	mg L ⁻¹	82,045
9.	Density	g cm ⁻³	1.0128
10.	Specific Gravity	-	1.01714
11.	Lactose	%	1.6
12.	Protein	%	0.03

4.1 Back Diffusion Study results:

The graph 3 below indicates the variation of Total Dissolved Solids (TDS) of Feed along with Time. The calculated TDS of 10% salt solution used for the back diffusion study is 1,38,412 mg L⁻¹, while the highest observed in feed side is 1173 mg L⁻¹. A constant TDS between 1147 mg L⁻¹ and 1173 mg L⁻¹ is observed for the entire study. After 12.6 minutes, the final TDS was 1142 mg L⁻¹ which indicates back diffusion of 0.82%.

The variation of weight of feed vs time during back diffusion study is as shown in below graph 4. It indicates the reduction in feed from 12 kg to 0.37 kg i.e. ninety-six percent feed concentration during the back diffusion study with an average reduction of 91 g min⁻¹.

4.2 FO study results:

The results obtained for various F/D in CSP and RC mode for 10 minutes and 30 minutes study in CSP and RC mode using equation no (4) and equation no (5) respectively are tabulated in Table 2.

Table 2

Results for Flux and Water Recovery for various F/D of whey and aq. NaCl solution in CSP and RC mode

Sr.No.	Feed Inlet (kg)	Draw Inlet (kg)	F/D	Feed Outlet (kg)	Draw Outlet (kg)	CSP mode results		RC mode results for Water Recovery (%)
						Flux (kg m ⁻² h ⁻¹)	Water Recovery (%)	
1	3.448	0.766	4.50	1.63	2.528	4.74	56.09	57.603
2	5.364	0.67	8.005	2.9	3.112	6.42	48.86	50.832
3	7.9	0.79	10	4.565	4.032	8.7	44.909	47.605
4	10.846	0.592	18.32	8.04	3.384	7.32	27.522	29.844
5	11.914	0.342	34.83	9.542	2.7	6.18	21.18	23.805

As observed from graph 5 above, % water recovery is seen to decrease as we approach towards higher F/D ratio. The highest water recovery of 56% is observed for the F/D of 4.5.

The variation in permeate flux along with F/D ratio for the CSP mode is observed in graph 6. The permeate flux increases with increase in F/D from 4.5, 8 and 10 whereas decreases further for F/D of 18 and 34.8. The highest flux of 8.7 kg m⁻² h⁻¹ is observed for F/D of 10.

The observed results of % water recovery in RC mode are displayed in above graph 7. The water recovery observed in RC mode is more as compared to CSP mode. The highest dewatering of 57.603 % was observed for F/D of 4.5.

Table 3
Density Results

Sample location	Density (g cm ⁻³)
Feed Inlet (Raw filtered whey)	1.01280
Feed outlet (Concentrated whey)	1.04506
Makeup Feed	1.01290
Draw Inlet (Saturated 24% NaCl solution)	1.17776
Draw outlet (Diluted NaCl solution)	1.03026

The Table 3 above displays the densities of the various samples obtained after the FO study. According to the dewatering percentage, when same amount of water is added to the feed outlet, approximately similar density (compared to feed inlet) of 1.01290 g cm⁻³ was achieved for the makeup feed which shows the permeation of almost pure water from feed to draw.

5. Conclusion:

Dairy whey contains high moisture content. FO membrane extracts and transfers the water from lower osmotic pressure feed side of the whey to the higher osmotic pressure draw side of the saturated NaCl solution using natural osmotic pressure. The minor back diffusion was observed through the FO membrane. The CSP mode study gives the highest permeate flux of 8.7 kg m⁻² h⁻¹ for F/D of 10 and highest dewatering of 56% is observed for the F/D of 4.5 for 10 minutes of operation. While RC mode gives the highest dewatering of 57.603 % for F/D of 4.5 for 30 minutes of operation. Thus, RC mode is more suitable for achieving higher dewatering at room temperature with more run time than the CSP mode. The analysis through density meter indicated the permeation of pure water through the membrane. This approach is an effective technique to partially dewater whey and helps to reclaim water in higher osmotic agent solution. It also reduces the net quantity of dairy wastewater to be treated. It adds value to the draw NaCl solution, a potential brine in the dairy industry and can be reused for various purposes in the dairy itself, eliminating the need for regeneration.

Declarations:

1. Ethics Approval and Consent to participate: Not applicable
2. Consent for publication: Not applicable
3. Availability of data and materials: All data generated or analysed during the study are included in this published article
4. Competing interests: Authors declare that there are no competing interests in this section
5. Funding: Not applicable

6. Authors Contribution: All authors contributed to the study, conception and design. Material preparation, data collection, investigation and analysis were performed by Vibha Agrawal, Saransh Mogha and Bharat Honmane. All the required resources were made available by Dr. Dilip Sarode. The manuscript was drafted by Vibha Agrawal and reviewed by Saransh Mogha and Dr. Dilip Sarode. All authors read and approved the final manuscript.
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Figures

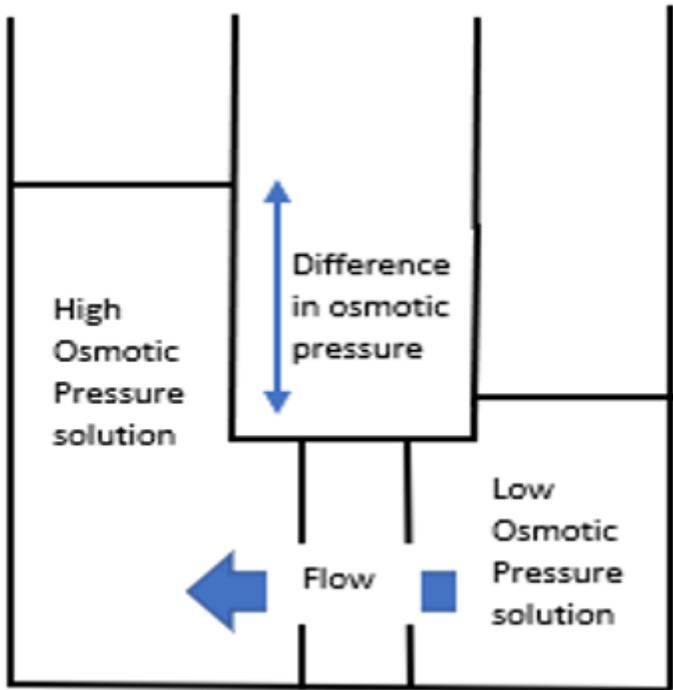


Figure 1

Schematic representation of Forward Osmosis

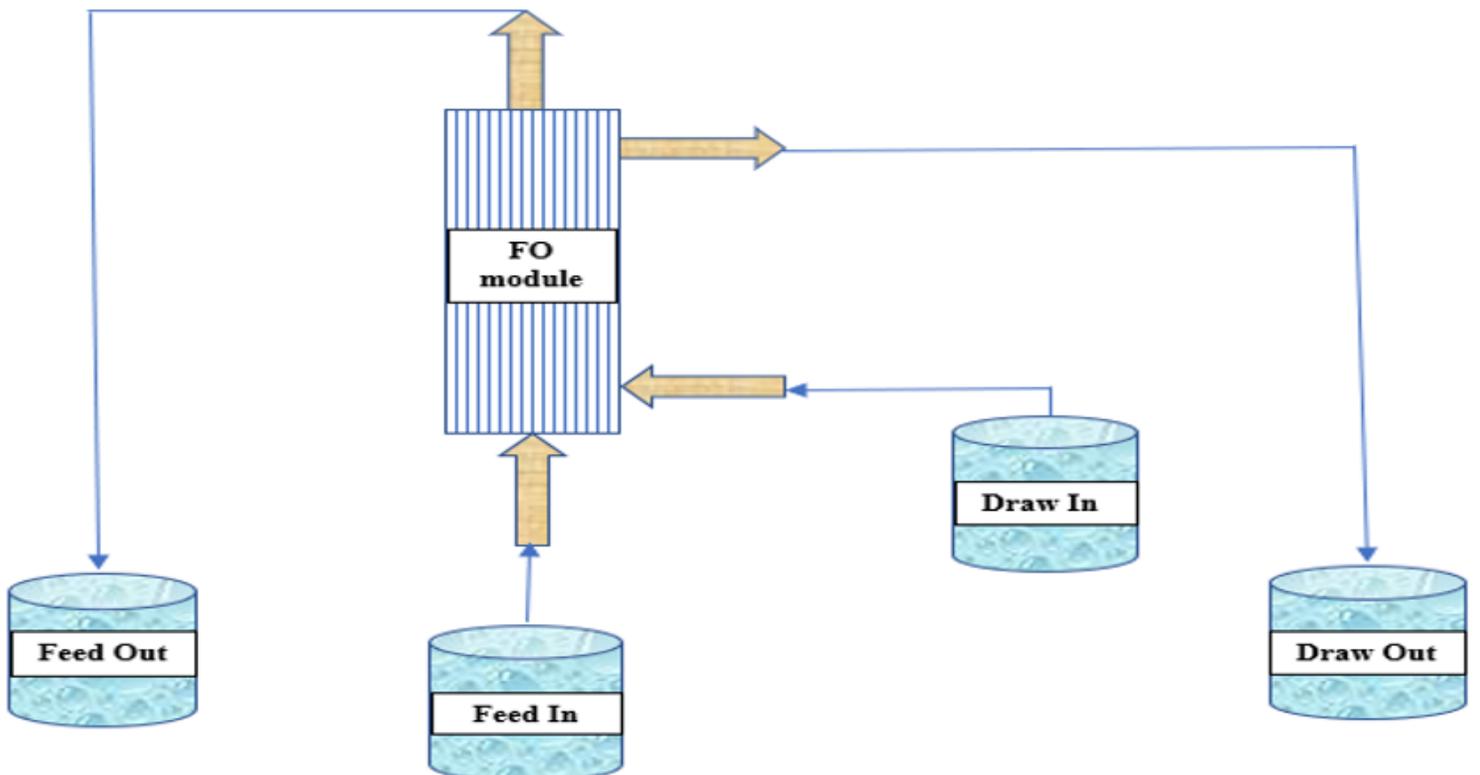


Figure 2

Schematic Representation of Continuous Single Pass (CSP) mode used in the study

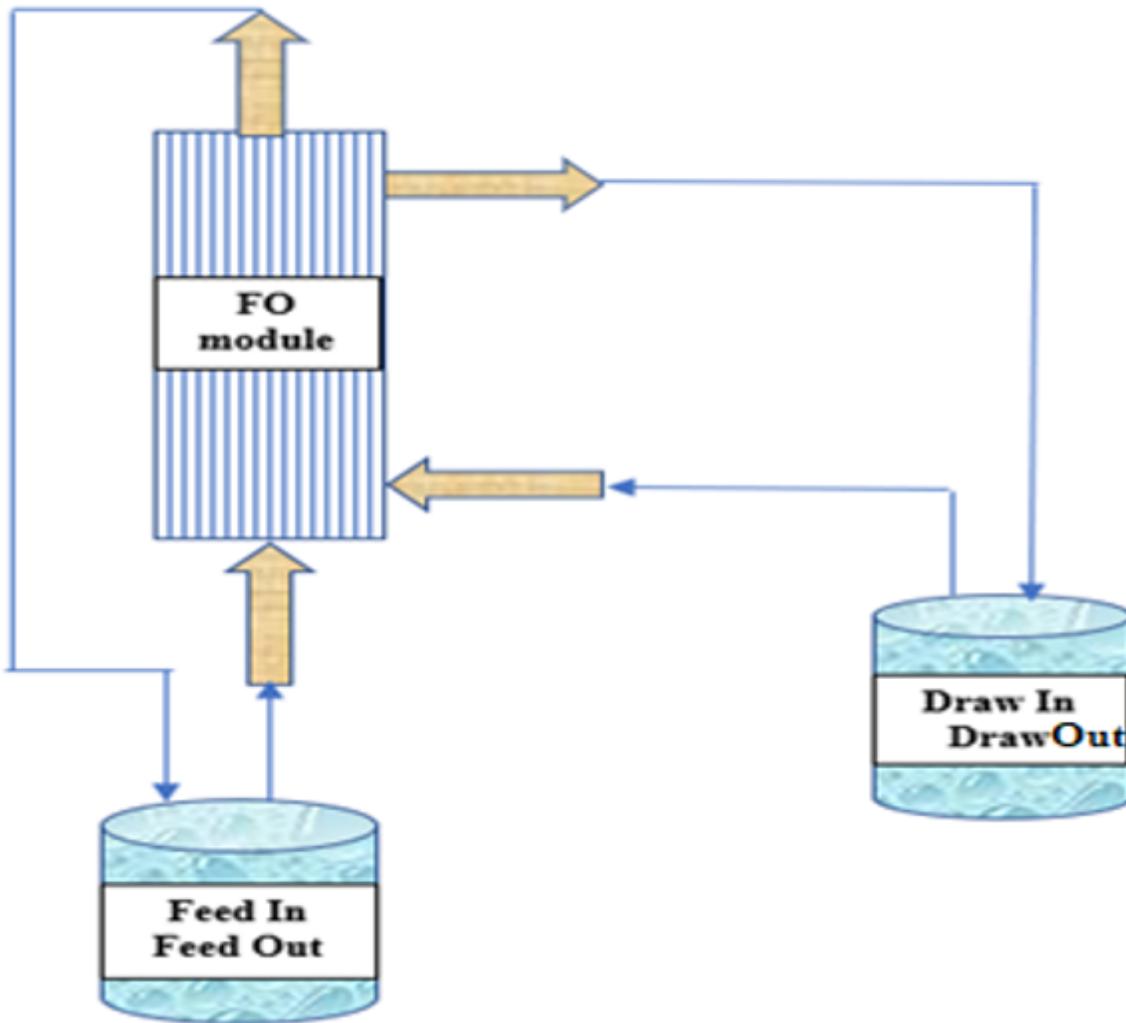


Figure 3

Schematic Representation of Recirculation (RC) mode used in the study

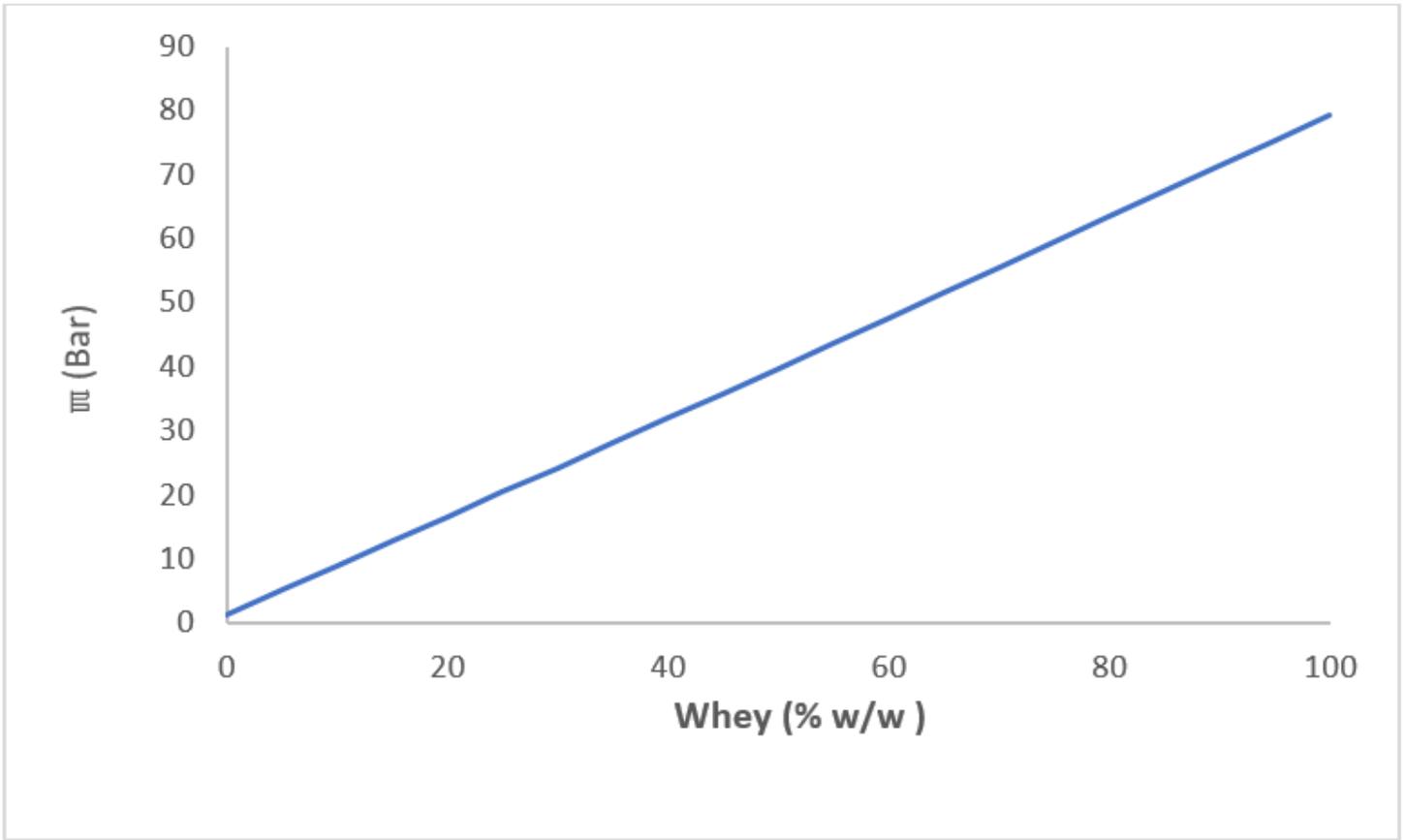


Figure 4

Plot of relationship between osmotic pressure and whey concentration

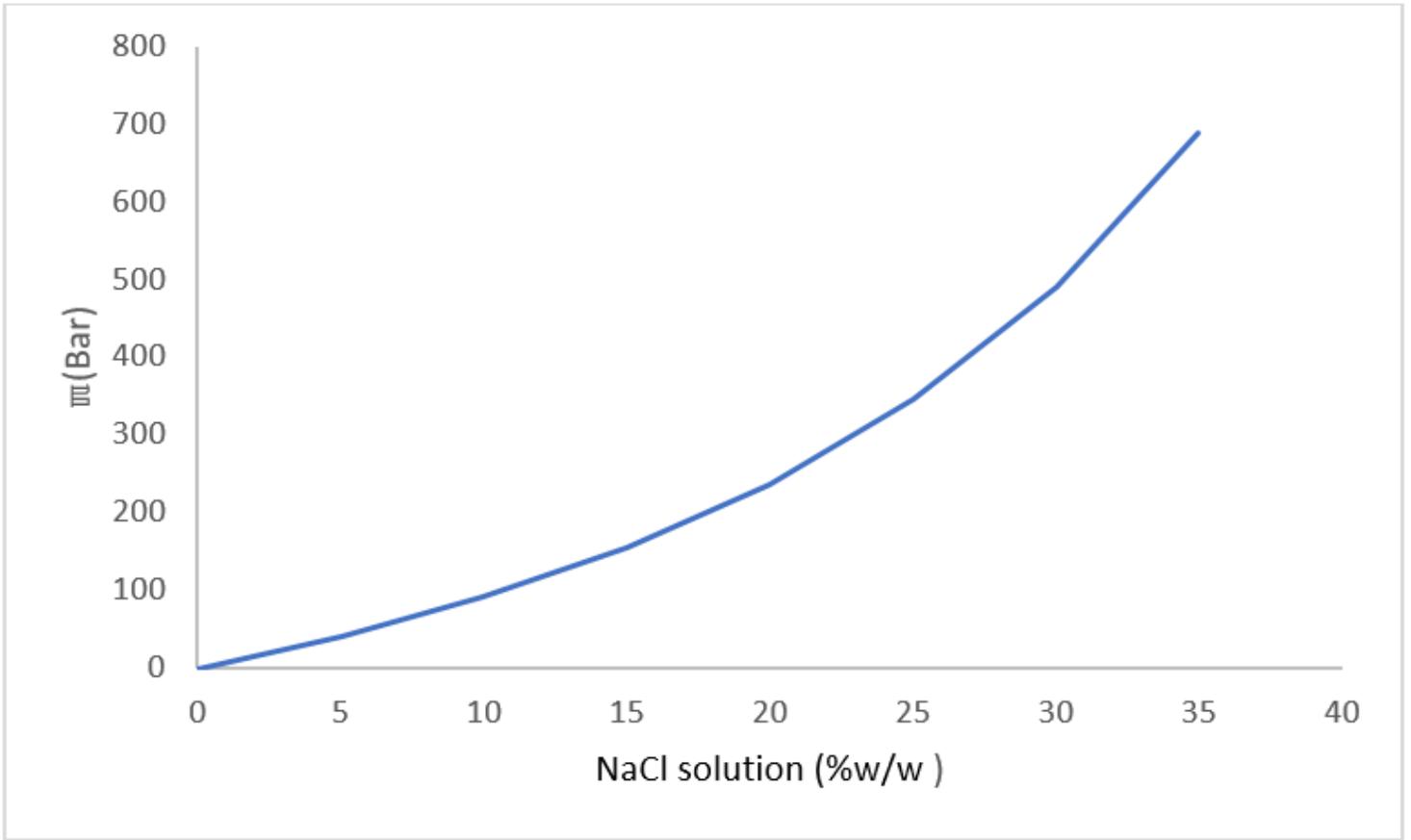


Figure 5

Plot of relationship between osmotic pressure and concentration of NaCl solution

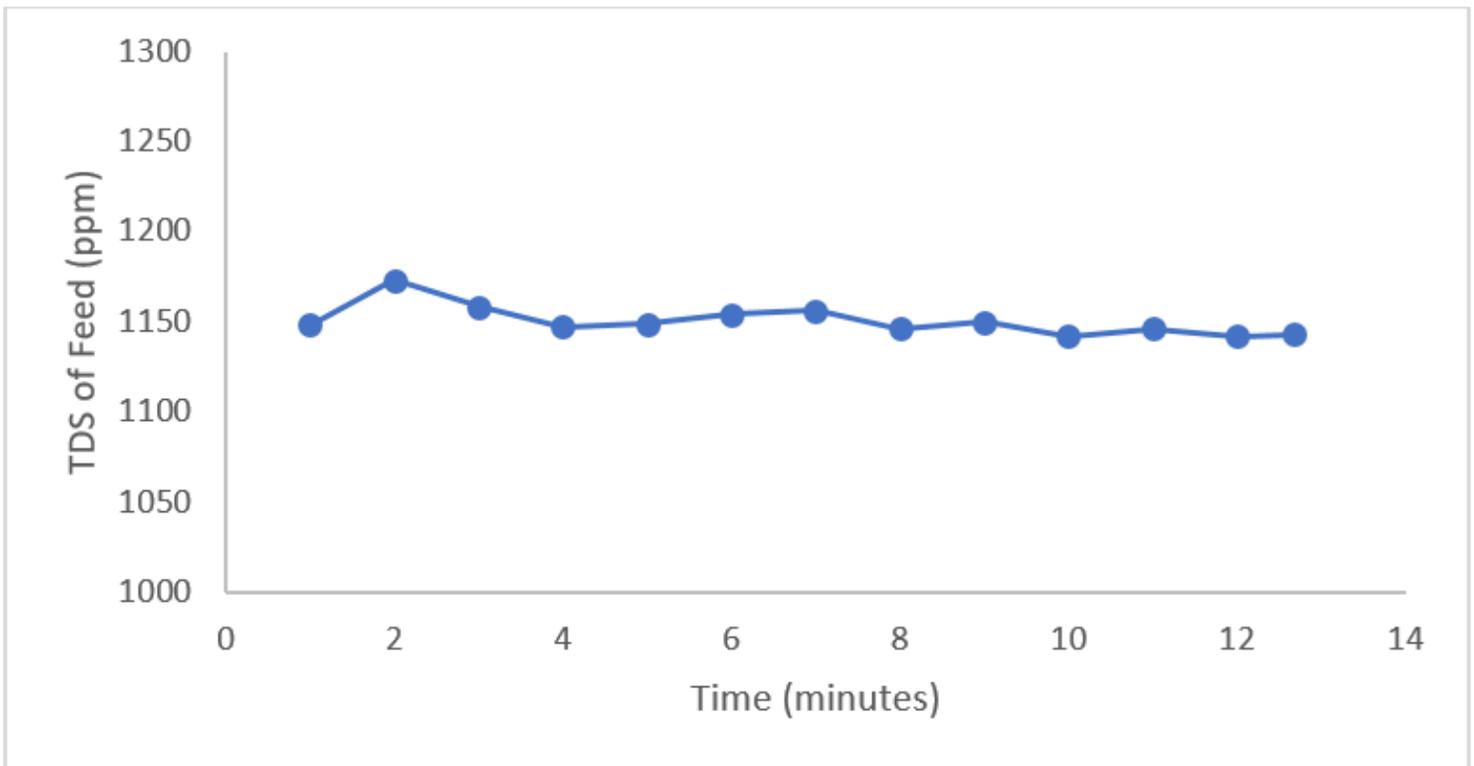


Figure 6

Plot of variation of TDS of Feed vs Time during back diffusion study

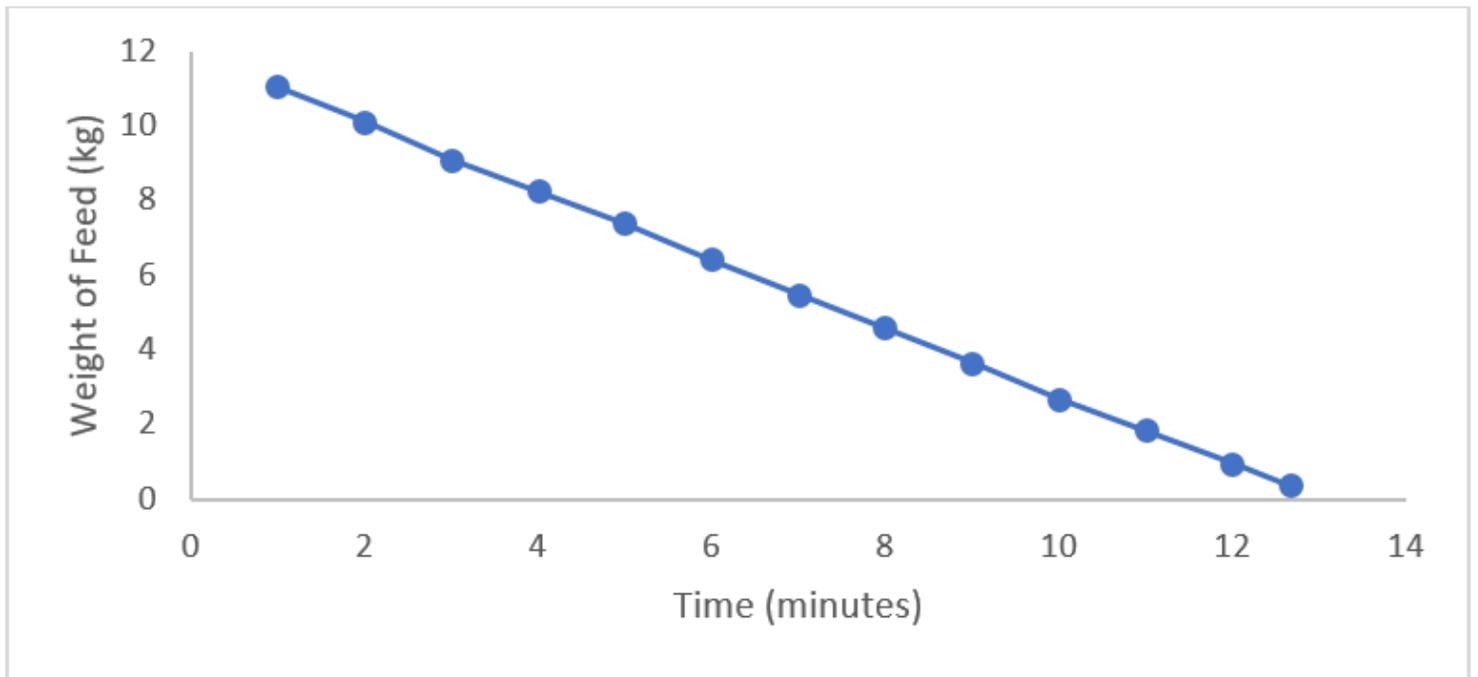


Figure 7

Plot of Weight of Feed vs Time during back diffusion study

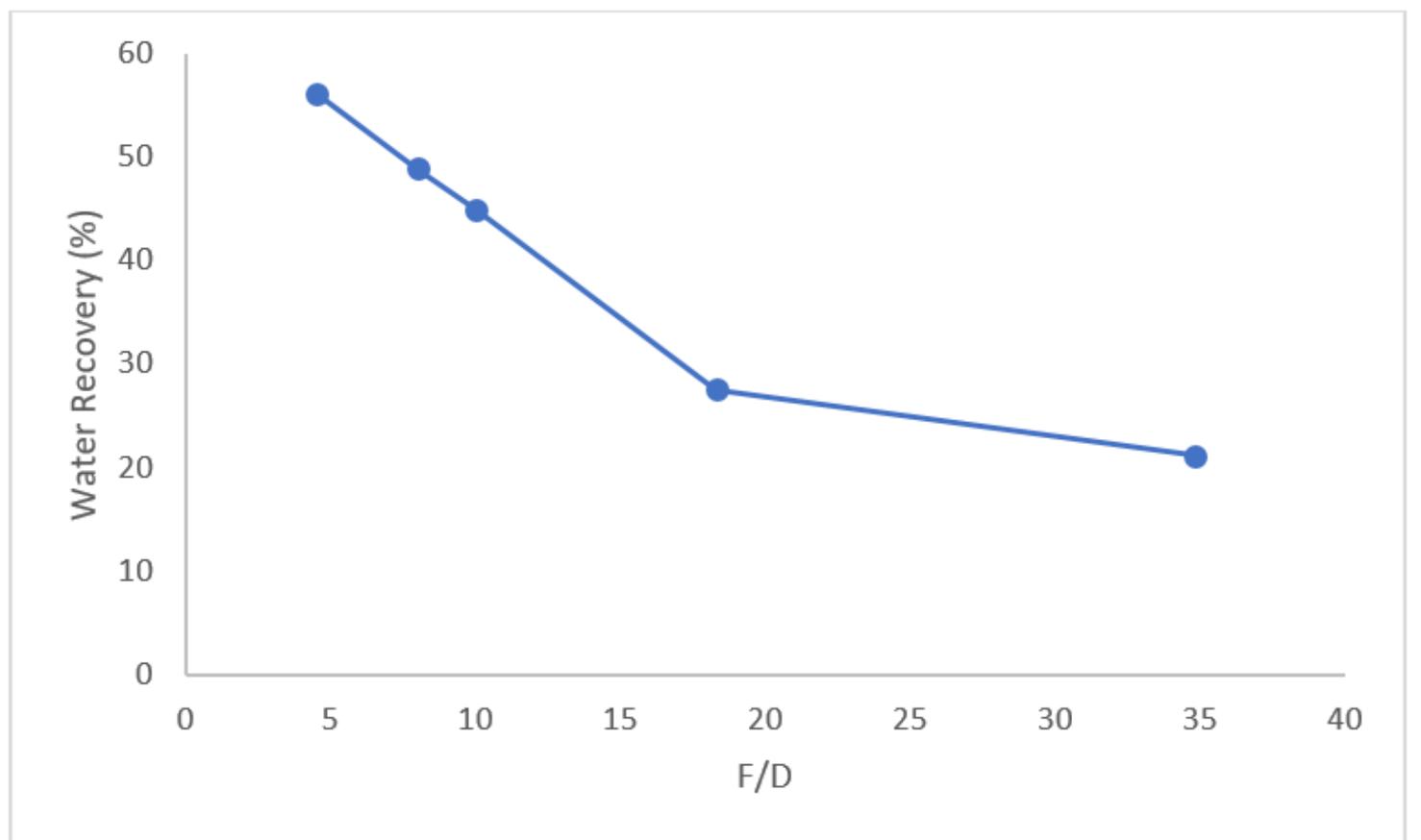


Figure 8

Plot of CSP mode- Water Recovery vs F/D

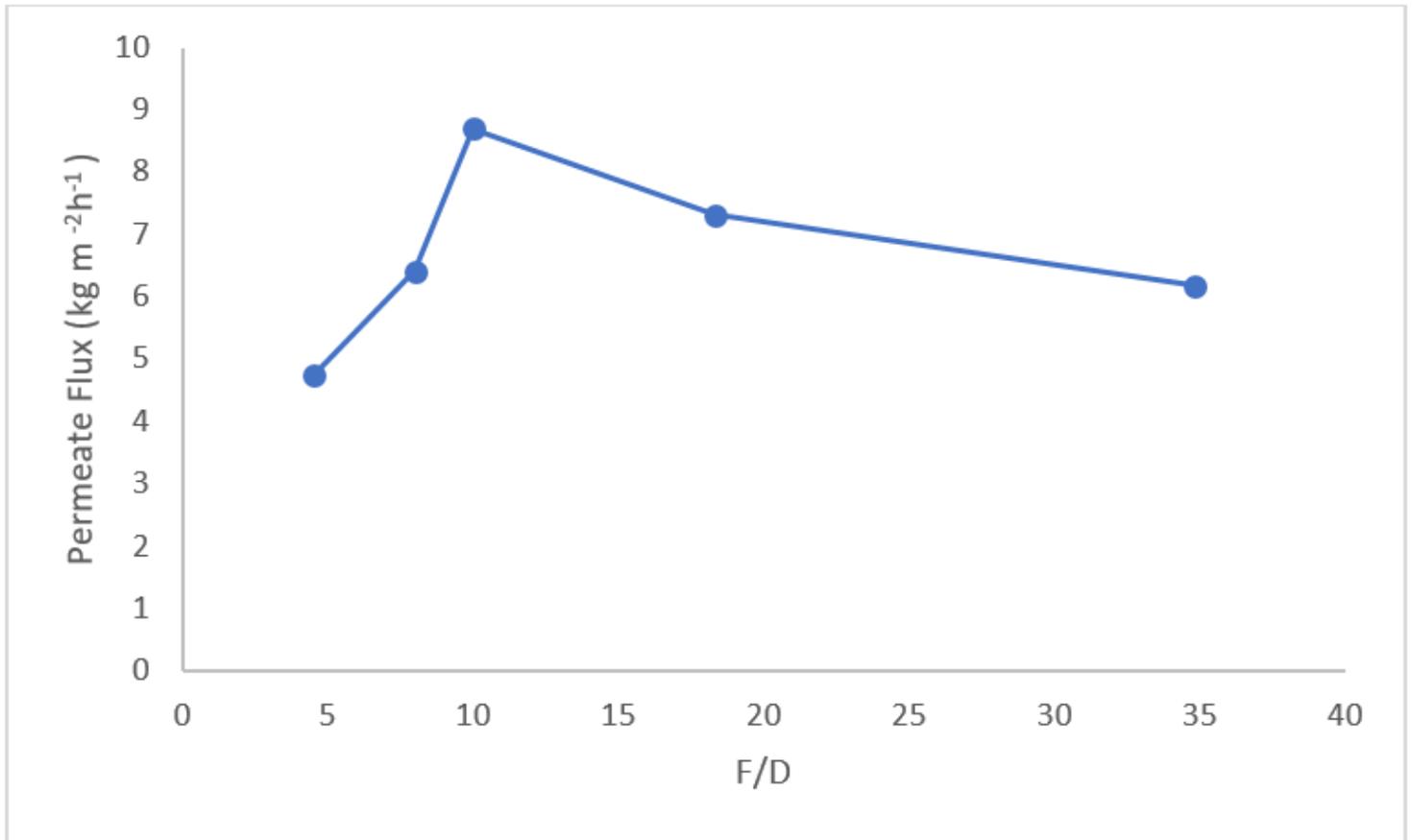


Figure 9

Plot of CSP mode- Permeate Flux vs F/D

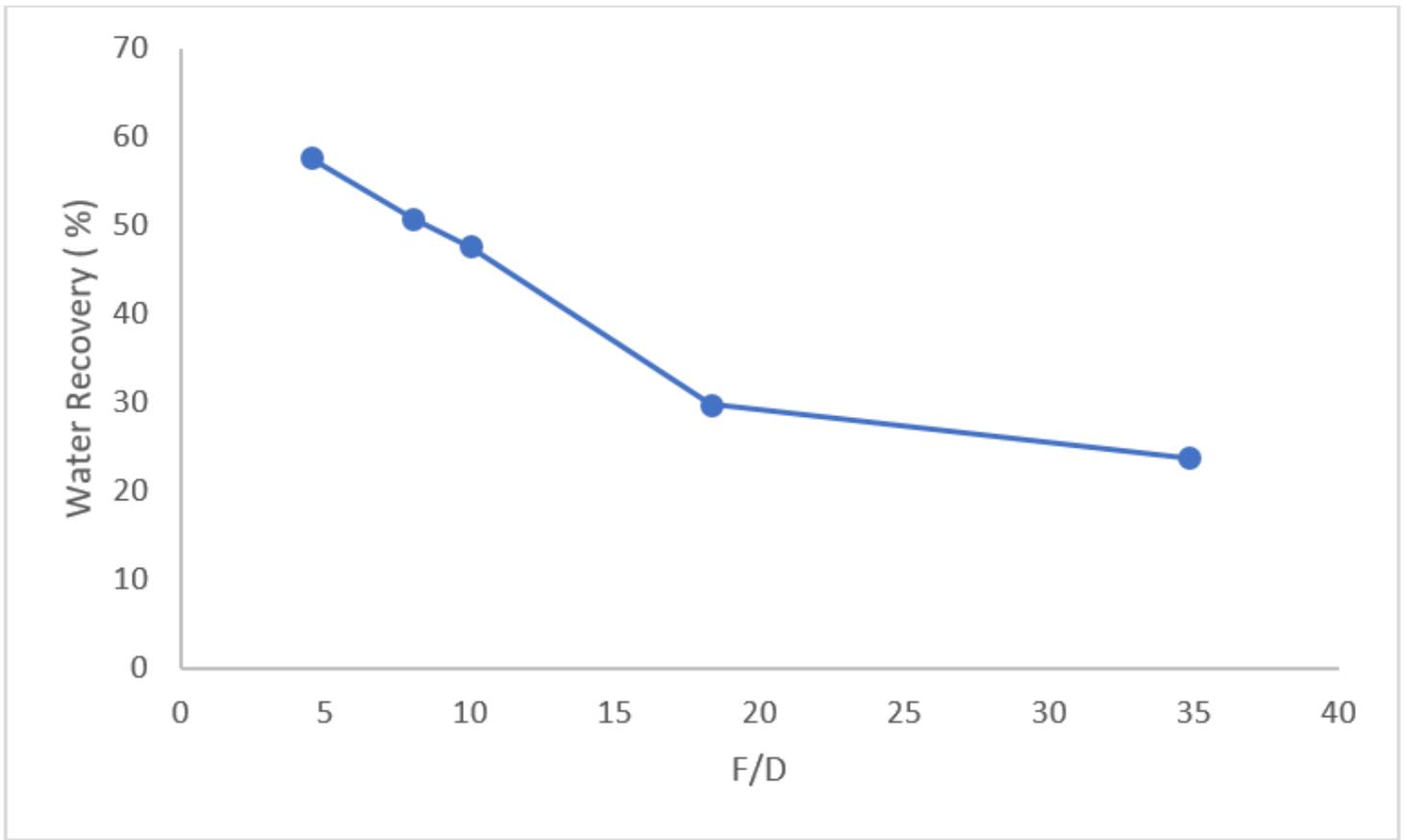


Figure 10

Plot of RC mode- Water Recovery vs F/D