



Treatability of Whey Processing Effluent in an Ultrafiltration Pilot Plant using Hydrophilic Polyethersulfone Membrane

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Treatability study of whey processing effluent was undertaken in a single stage ultrafiltration pilot plant using a commercial hydrophilic polyethersulfone flat sheet membrane. The pure water permeability of the membrane was found to be $1.065 \times 10^{-7} \text{ m}^3 / (\text{m}^2 \cdot \text{s} \cdot \text{kPa})$. The flux of permeation was observed to decrease steadily at all pressures and the maximum flux was recorded to be $10.42 \times 10^{-6} \text{ m}^3 / \text{m}^2 \cdot \text{s}$ at $2.94 \times 10^2 \text{ kPa}$ after around 15 minutes of operation. The initial and final characteristics of dairy wastewater sample were determined in the form of pH, BOD, COD and TDS at an ambient temperature. The BOD, COD and TDS removal were found to be 44 %, 64% and 38% respectively. The results, thus obtained underscore the importance of ultrafiltration as a supplement to the existing wastewater treatment processes.

Keywords: COD, Dairy effluent, Flux, Permeability, Ultrafiltration

Introduction

Wastewater from the dairy processing industry, cheese whey effluent in particular, is featured by a relatively high organic load with a ratio of BOD to COD typically in the range 0.4–0.8.¹ Conventional biological and physico-chemical treatments stand inadequate in view of growingly stringent discharge norms for such effluents. Membrane processes, especially ultrafiltration (UF) and nanofiltration (NF) on the other hand, have become a viable alternative on several counts such as high COD reduction in the range of 74 and 98%, removal of ancillary contaminants and color, and ease of operation with high throughput.^{1,2} UF is a low pressure membrane separation process applied in the separation and fractionation of solutes of varied molecular weight spanning from 5 kDa to 500 kDa or even more depending on the membrane molecular weight cut-off.^{3,4} Luo *et al.*, revealed that an integrated UF-NF treatment could be a worthwhile strategy not only for recycling of nutrients, but also for the reclamation of dairy wastewater.⁵ Furthermore, the process could generate bioenergy. In another study it was reported that UF treatment of dairy effluent led to almost 58% retrieval of clean water using optimum trans-membrane pressure (TMP) and volume reduction

factor (VRF).⁶ Notwithstanding a growing body of literature cites the treatment of dairy effluents using innumerable membrane processes, very few pilot plant studies of UF necessitate the need for more research on several grey areas of membrane chemistry and process improvement. In view of the same, the present study was undertaken in order to assess the potential of UF using a commercial hydrophilic polyethersulfone membrane, in terms of flux and quality of permeate stream. In essence, the aim of the current study is to explore the efficacy of ultrafiltration in the downstream processing of dairy effluent under different experimental conditions.

Materials and methods

Chemicals and Membranes

Polyethersulfone/Hydrophilic ultrafiltration (PES/HFUF) flat sheet membrane used in the present work was purchased from Permionics Membranes Private Limited, Vadodara, India. The molecular weight cut off the membrane was 5 kDa while its maximum operating temperature and pH range being 45°C, and 1–14 respectively. The stock solution of feed effluent was prepared by deionized water having conductivity 15 $\mu\text{s}/\text{cm}$ obtained from an in-house RO system. Reagents and chemicals used in the present study were essentially of AR grade supplied by Merck, India and were used as-received sans any pretreatment.

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Permeability and Flux Measurement of Membrane

The permeability of the used membrane was determined following experimental measurement of fluxes under various operating pressure using deionized water. The slope of the permeation flux versus trans-membrane pressure plot thus obtained represented the membrane permeability. The flux of water J_w ($m^3/m^2 s$) was estimated using Eq. 1.⁷

$$J_w = \frac{V}{A} \Delta t \quad \dots (1)$$

Where V is the volume of the permeate sample (m^3), A is the effective area (m^2) and Δt is the permeation time (s). The trials were conducted at $25 \pm 1^\circ C$ and average of three replicates was reported.

Characterization of Dairy Effluent and Permeate Collected

Biochemical oxygen demand (BOD), chemical oxygen demand (COD), total dissolved solid (TDS) and pH of the feed and permeate streams were evaluated using BOD₅ method, open reflux method, digital TDS meter (H M Digital Inc, Model No TDS3TM) and digital pH meter (Model No 802, Systronics Make) following standard methodologies described elsewhere (APHA 2005).

Ultrafiltration Study of Dairy Effluent

In order to perform the ultrafiltration study of dairy effluent using the PES/HFUF membrane, initially the effluent sample was diluted with distilled water in the ratio of 2:1(two part of effluent sample and one part of distilled water).The ultrafiltration experiment of dairy effluent was carried out in a small scale laboratory unit as shown in Fig. 1. The experimental set up consists of feed tank, plunger pump, pressure gauge and two detachable stainless steel plates to put the membrane. The effective surface area of this flat sheet membrane module was $0.016 m^2$. The diluted feed dairy effluent solution was taken into feed tank

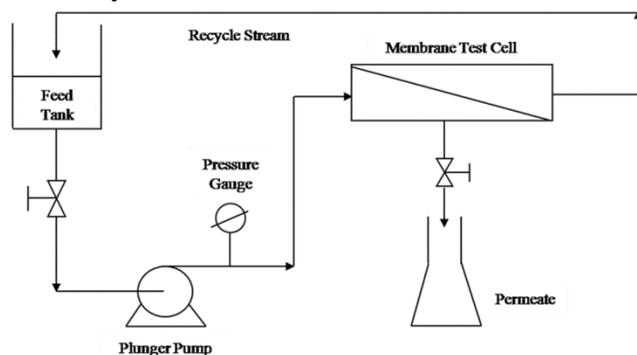


Fig. 1 — Schematic outline of ultrafiltration unit used in the present study

(around 20 litre) and passed through test cell using high pressure (50–400 psi) plunger pump at an ambient temperature ($25 \pm 2^\circ C$). The feed solution was passed from membrane surface at different trans membrane pressure (TMP) ranging from $1.96 \times 10^2 kPa$ – $8.83 \times 10^2 kPa$. The feed flow rate was controlled with rotameter. The retentate stream generating out from the test-cell was sent back to the feed reservoir while permeate stream was collected at regular interval of time and analysed. Each set of experiment was repeated three times with an error of $\pm 2\%$.

FTIR Analysis of Membrane

The purchased commercial Polyethersulfone/ Hydrophilic ultrafiltration (PES/HFUF) flat sheet polymeric membrane (5 kDa) used in the present study was characterized through Fourier transform infrared spectroscopy (FTIR) (Spectrum GX Model; Perkin Elmer, USA). The output from FTIR was logged from a wave number of $400-4000 cm^{-1}$ ($2.5-25 \mu$) at a resolution of $4.0 cm^{-1}$ and was subsequently analysed.

Results and Discussion

Membrane Permeability

The plot of permeate flux against trans-membrane pressure (TMP) to determine the UF membrane permeability using distilled water at ambient temperature established a linear trend. It was evident that the permeate flux increased linearly with increase in TMP. At $1.96 \times 10^2 kPa$ TMP the flux was observed to be $2.63 \times 10^{-5} m^3/m^2s$ which further rose to $10.4 \times 10^{-5} m^3/m^2s$ at $8.83 \times 10^2 kPa$ TMP. This was almost 75% increase with respect to the starting value of permeate flux. Literature has also reported the similar trend for pure water permeability measurement for analogous flat sheet polymeric membrane.^{6,8} The experiment was repeated two times and the average value of pure water permeability for the 5 kDa HFUF membrane used in the present study was found to be $1.065 \times 10^{-7} m^3/(m^2.s.kPa)$.

Permeate Flux

The variation of volumetric permeation flux as a function of run-time at different TMP is presented in Fig. 2. The flux has gradually decreased with time irrespective of any TMP. It was also noted that as the TMP increased from $2.94 \times 10^2 kPa$ to $6.86 \times 10^2 kPa$, the permeate flux was decreased. The highest flux was recorded to be $10.42 \times 10^{-6} m^3/m^2s$ at $2.94 \times 10^2 kPa$ TMP while the lowest was $0.52 \times 10^{-6} m^3/m^2s$ at

6.86×10^2 kPa TMP. It was observed that after around 3 h and 10 min of ultrafiltration study with the present membrane, the volumetric permeate flux was diminished to almost zero. Plausibly this was due to blockage of membrane pores by the concentration polarization phenomena on the membrane surface, which might have set a limit in the performance of the ultrafiltration membrane. Literature has also reported the similar kind of observations of permeate flux for hydrophilized polyamide membrane for the removal of dye from dye wastewater.⁸ Another similar kind of study has also indicated that with respect to time permeate flux decreased gradually for the ultrafiltration study of dairy wastewater using polyethersulfone flat sheet membrane of 50 kDa.⁹

Raw Dairy Effluent and Permeate Characteristics

The initial characterizing parameters of the feed dairy effluent, measured at an ambient temperature are presented in Table 1. After measuring the initial characteristics of raw dairy effluent, it was diluted to 2:1 ratio with distilled water in order to make it suitable for UF experimental study with the given

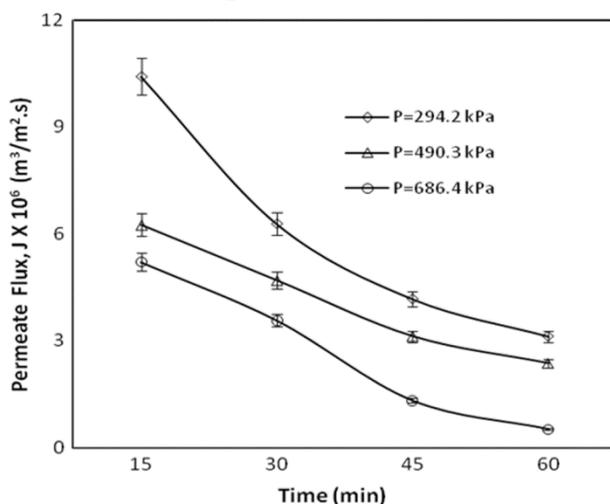


Fig. 2 — Permeation flux profile at different trans membrane pressure

(Feed temp: $25 \pm 2^\circ\text{C}$, Run time: 1 h for each TMP, Feed dilution ratio: 2:1 with distilled water)

membrane. The experiment was performed at different TMP and permeate collected was also characterised and matched. A scrutiny of Table 1 reveals that the values of BOD and TDS were reduced as the TMP was increased from 2.942×10^2 kPa to 6.86×10^2 kPa while pH of the permeate was found to increase indicating alkaline nature of the permeate. The permeate COD was found to reduce much as compared to diluted raw dairy effluent. Similar kind of observation for the dairy effluent from milk industry was reported in literature.⁶ This reduction in BOD, COD and TDS is attributed to the sieving of the larger molecules of proteins, fats and carbohydrates in the membrane pores.

FTIR Analysis of Membrane

The organic functional groups present in the virgin (unused) and used ultrafiltration membranes are provided in Fig. 3. The presented FTIR spectra of virgin membrane in Fig. 3 indicate number of bands or peaks of different shape and length in the wave numbers range of 400 to 4000 cm^{-1} . The assignment of functional group for the PES membrane was done following literature.^{10,12} A very broad band in at the vicinity of 1634 cm^{-1} indicates C–C multiple bond stretching. The bands near $3300\text{--}3500\text{ cm}^{-1}$ range indicate O–H stretching vibrations in oxygenated compounds. The second peak near 2926 cm^{-1} corresponds to intermolecularly hydrogen bonded single bridge compounds in polymeric association. It can also be assigned to stretching vibrations of C = O bonds connected to primary amides of proteins. The polyethersulfone contains two benzene rings, linked by a sulfone group and an ether band. The bands at $1105\text{--}1242\text{ cm}^{-1}$ are attributed to the bands of aromatic ethers and sulfonyl group of PES. A distinct band at 1157.6 cm^{-1} is attributed to the presence of sulfone group. The peak at 1486 cm^{-1} and 2855 cm^{-1} are indicative of C–H bending vibration of alkanes. The increased absorbance at $2800\text{--}2900\text{ cm}^{-1}$ is plausibly due to C–H stretching vibrations of hydrocarbon chromophore particularly aliphatic

Table 1 — Raw dairy effluent and permeate characteristics at an ambient temperature

Sr. No	Parameters	Raw Effluent	Diluted Effluent Ratio (2:1)	Permeate characteristics at different TMP		
				2.94×10^2 kPa	4.90×10^2 kPa	6.86×10^2 kPa
1	pH	4.2	4.9	5.7	6.2	6.8
2	TDS (mg/l)	2730	2140	1500	1450	1330
3	BOD (mg/l)	720	553	350	340	311
4	COD (mg/l)	2920	2033	747	724	738

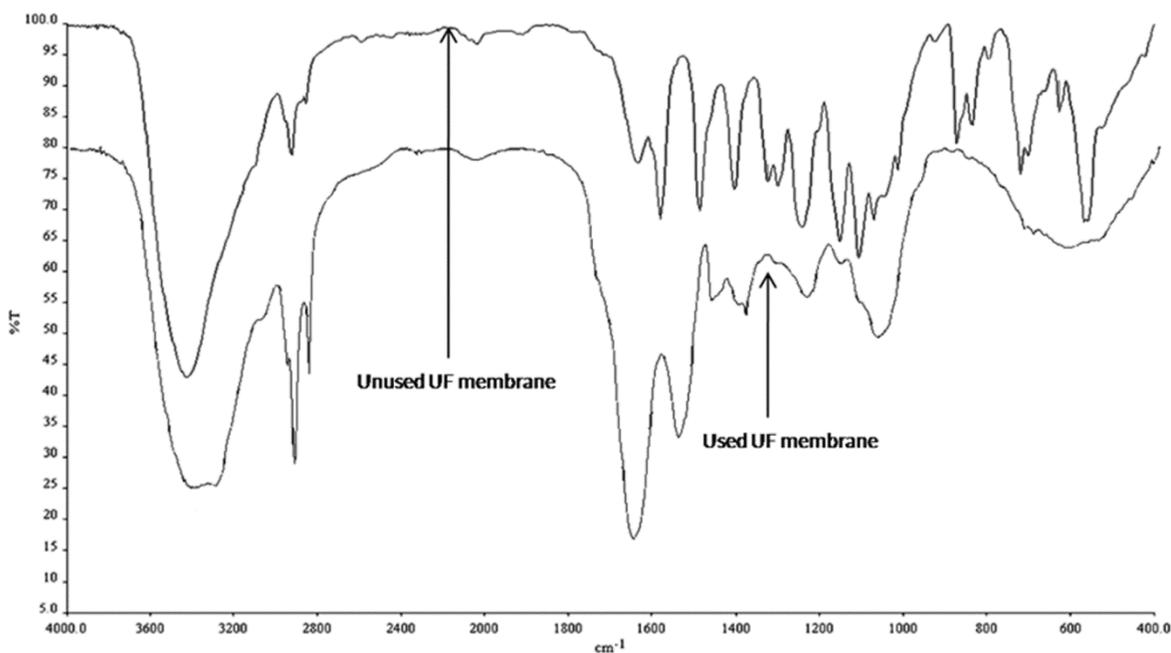


Fig. 3 — FTIR spectra of unused (pristine) and used UF membrane in the present study

compounds.¹⁰ The spectrum analysis of used UF membrane shows less number of peaks than unused pristine UF membrane. The sharp peaks are also not observed and shifted to relatively lower wave number in used membrane. This could be reasoned to the partial blockage of pores on the membrane surface through solute deposition which reduces the permeate flux as the time progresses.⁸

Conclusions

Flat sheet Polyethersulfone/Hydrophilic ultrafiltration (PES/HFUF) membrane of 5 kDa was used for the experimental study of diluted dairy wastewater at different trans-membrane pressure (TMP). The pure water permeability was found to be $1.065 \times 10^{-7} \text{ m}^3/(\text{m}^2 \cdot \text{s} \cdot \text{kPa})$. The flux due to permeation was gradually decreased with time at all TMP. The maximum flux was $10.42 \times 10^{-6} \text{ m}^3/\text{m}^2 \cdot \text{s}$ at $2.94 \times 10^2 \text{ kPa}$ after around 15 minutes of operation. The initial and final characteristics of dairy wastewater sample were determined in the form of pH, BOD, COD and TDS at an ambient temperature. The BOD, COD and TDS removal were found to be 44 %, 64% and 38% respectively. The polyether sulfone flat sheet membrane could be a good candidate in the reclamation of dairy waste water. The overall single stage ultrafiltration can be found suitable for the treatment of dairy effluent. The treated dairy effluent can be recycled or reused or can be discharged into the main stream.

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