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Reclamation of dairy wastewater using ultrafiltration process

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ABSTRACT

Large amounts of water are daily used in dairy industries for different parts of their services such as chilling, cooling, steam production, pasteurizing, etc. This consumption generates a huge quantity of wastewaters which could be reused after performed treatment. In the present work, a real sample of dairy wastewater was treated using ultrafiltration (UF) and process efficiency and permeate quality were improved by operating under optimum conditions of transmembrane pressure (TMP) and volume reduction factor (VRF). More than 99% of retention rate were observed for turbidity and the BDO5, more than 80% for suspended matter, and 95% for proteins with an optimal TMP fixed at 2.5 bar. Moreover, a reduction of 40 and 55% was recorded for conductivity and the total dissolved salts, respectively. Rather important retention rates varying from 95 to 99% for the majority of the analyzed parameters were observed for a VRF range varying from 1.11 to 2.5. A recovery of 58% of the dairy effluent is possible after treatment by UF using the PES-5 membrane. The permeate quality obtained in optimal TMP and VRF allows the industry to reject its effluents into the river without risks of contamination (according to Tunisian standard for wastewater discharge NT106-02) and to reuse or recycle them during the process (according to Tunisian standard for reusing treated wastewater NT106-03).

Keywords: Ultrafiltration; Dairy wastewater; Retention rates; Volume reduction factor

1. Introduction

Tunisian dairy sector is strategic and it plays an important role in the agricultural, economic, and social fields; it occupies 68% of permanent workers employed in the agricultural sector [1]. However, this sector consumes huge amounts of water during the production process and equipment cleaning. In fact, water consumption varies between 1.5 and 5 L per liter of treated milk depending on the type of industry [2–4].

The untreated wastewater issued by units of dairy products may have a high organic content, biochemical oxygen demand (BOD), and chemical oxygen demand (COD). This can cause water and soil pollution and therefore permanent risk of environmental pollution. That is why the requirements of the Tunisian standard for wastewater (NT106-02) oblige

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manufacturers to treat their wastewater before discharging into the environment.

Several techniques have been assayed to reduce the pollutant load of wastewaters generated by food industries in general, and dairy industries in particular where the processes such as flocculation, deep filtration, coagulation, etc. are used. These conventional treatment processes have always presented disappointing results, either because of poor performance or by very high production of sludge [5–9].

Among available technologies for wastewater treatment, membrane technology has been often considered as a promising method especially for water reuse [10–12].

Several investigations have shown that nanofiltration (NF) and reverse osmosis (RO) were adequate for concentration of dairy effluents and production of reusable water. Koyuncu et al. [13] found that NF could reject more than 98% of COD and conductivity of dairy effluents, and a two-pass RO system could produce water of very high quality.

Permeate water obtained from NF/RO treatment of dairy wastewater can be discharged in river or reused, but with the increase in organic solutes and inorganic salts in retentate during a concentration process, concentration polarization and osmotic pressure increase rapidly leading to a large flux decline [10].

Chollangi and Hossain found that UF of dairy wastewater yielded a high permeate flux at low transmembrane pressure (TMP) using regenerated cellulose membranes, but the permeate characteristics were not sufficient for water reuse as it contained high lactose concentration [14]. A two-stage UF/NF process was proposed for utilization of whey protein and lactose, as proteins were retained by UF membrane and lactose in UF permeate was concentrated by NF [15,16].

Concentration of dairy wastewater by NF using a rotating disk membrane (RDM) filtration system was investigated by Luo et al. in order to promote process efficiency based on flux and selectivity [17,18]. A real effluent was treated by shear-enhanced filtration system and its result was compared with that of model effluent. Under extreme hydraulic conditions of highest TMP with high shear rate, the RDM filtration system could produce a better quality permeate and save energy because of its very high permeate flux.

Luo et al. also studied the performances of two-stage ultrafiltration and nanofiltration (UF/NF) process for the treatment of model dairy wastewater to recycle nutrients and water from the wastewater [19]. They concluded that most of the dairy wastewater could be recycled to produce reusable water and substrates for bioenergy production. Compared with the single NF process, this two-stage UF/NF process had a higher efficiency and less membrane fouling.

In this study, in order to evaluate UF process efficiency, a polyethersulfone membrane designated PES-5 was chosen based on molecular weight cut-off and flux, and a real effluent was collected from a Tunisian milk industry after biological treatment.

The effect of the main process parameters such as TMP, recovery rate, and volume reduction factor (VRF) on the quality of permeate obtained was studied.

The objective of this work is to minimize the impact of dairy discharges on the environment by improving the quality of water discharged and to study the possibility of reuse of the treated wastewater using single UF process.

2. Material and methods

2.1. Experimental setup

Membrane experiments were performed in a Millipore Labscale TFF system using PES-5 ultrafiltration membrane (Fig. 1). The main characteristics of the PES-5 membrane are summarized in Table 1.

The solution was pumped through the system using a high-pressure pump. The pressures were regulated using digital pressure gauges.

Permeate fluxes (J_v), retention rates (R), and volumic reduction factor (VRF) were measured during experiments according to the following Eqs. (1–3):

$$R = 100 \times \left(1 - \frac{C_p}{C_f}\right) \quad (1)$$

where C_p and C_f are permeate and feed concentrations, respectively;

$$J_v = \frac{Q_p}{S} = \frac{V_p}{t \times S} \quad (2)$$

where V_p is the recovered volume of permeate, t is the time necessary to collect this volume, and S is the effective membrane surface;

$$\mathrm{VRF} = \frac{V_i}{V_R} \quad (3)$$

where V_i and V_R are the initial and the retentate volumes, respectively.

A rinsing step with distilled water was applied after each experiment without applying any pressure

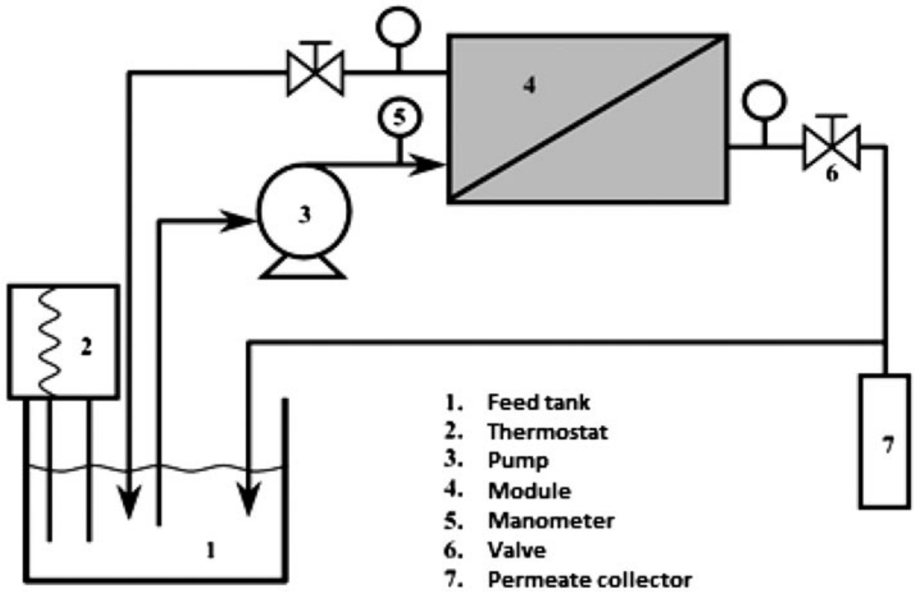

Fig. 1. Membrane filtration experimental setup.

Table 1
PES-5 UF membrane characteristics

Parameter		Value/characteristic
MW CO	(KDa)	5
Geometry		Flat sheet
Composition		Polyethersulfone
Active surface	(cm^2)	50
Temperature max.	(°C)	45
Pressure max.	(bar)	5.6
pH range		1–14

and increasing the feed flow rate in order to eliminate the organic and inorganic substances that clogged the membrane. After that, the membrane permeability was re-evaluated in order to check the membrane fouling state.

2.2. Wastewater and permeates analysis

The values of pH were determined by means of a GLP 22 pH-meter (Crison). The conductivities were measured by a Crison (GLP 32) instrument-type conductivity meter. Both conductivity and pH sensors used for these analyses allowed automatic and continuous correction of the values by taking into account the sample temperature. Turbidity was measured with a DINKO D-112 turbidimeter according to the ISO 7027:1999. Turbidity and conductivity were measured with accuracies of ±2 NTU and ±1.0%, respectively. The COD concentrations were obtained using a Spectroquant Nova 60 from MERCK (Germany)-type COD meter.

The main ions (sodium, potassium, magnesium, calcium, sulfate, and chloride) and the total dissolved solids were determined according to the Standard Methods [20]. Kjeldahl nitrogen was analyzed according to the NF EN 25663 standard, and the protein concentration was measured according to the Bradford method.

2.3. Dairy wastewater characteristics

Table 2 summarizes the physico-chemical characteristics of dairy effluent collected at the outlet of the biological treatment plant in the milk industry.

The results of physico-chemical analysis of the effluent collected show that the parameters that exceed the limits of the standard NT106-02 are COD, BOD5, SM, the content of oils, and fats, sodium, chloride, total phosphorus, and Kjeldahl nitrogen. All remaining parameters are consistent with the standard. We also note that conductivity, turbidity, and dissolved salts are relatively high. Indeed, the conductivity of the effluent exceeds 1,500 µS/cm, which proves excessive mineralization of the wastewater. For turbidity, the value is greater than 50 NTU, proving that the analyzed water is cloudy.

This preliminary analysis of the effluent allows us to optimize the parameters to be analyzed for the continuation of this work.

Table 2
Physico-chemical characteristics of the dairy wastewater

Parameter	Unit	Experimental value	NT 106-02 standard
pH	–	7.3	6.5< pH <8.5
Suspended matter (SM)	mg/L	**110.7**	30
Total dissolved salts (TDS)	mg/L	**2670**	–
Turbidity	NTU	**68.5**	–
Conductivity	S/cm	**4430**	–
Chemical oxygen demand (COD)	mg O_2/L	**346**	90
Biochemical oxygen demand (BOD)	mg O_2/L	**50**	30
Chloride	mg/L	**804.5**	600
Sulfate	mg/L	394.7	600
Magnesium	mg/L	52.13	200
Potassium	mg/L	26.31	50
Sodium	mg/L	**839.7**	300
Calcium	mg/L	52.42	500
Fluoride	mg/L	2.41	3
Nitrate	mg/L	40.8	50
Nitrite	mg/L	> 0.1	0.5
Nitrogen	mg/L	**42**	1
Phosphor PO ou P total	mg/L	**3.3**	0.002
Fat and oil	mg/L	**72**	10
Anionic detergents such alkylbenzene	mg/L	**0.54**	0.5
Copper	mg/L	0.034	0.5
Zinc	mg/L	0.018	5
Iron	mg/L	0.052	1
Mercury	mg/L	0.0003	0.001
Cadmium	mg/L	**0.035**	0.005
Protein	mg/L	9.3	–

3. Results and discussion

3.1. Pure water permeability

Pure water flux was measured at the beginning of the experiment. The flux increased linearly with TMP within the tested range, 1–3 bar. The PES-5 membrane permeability value was 55.09 L/h m^2 bar as shown in Fig. 2.

3.2. Effect of TMP

In the present study, 500 mL of dairy effluent was introduced into the feed tank and 25 mL of permeates were recovered at different TMPs ranging from 1 to 3 bar, and we followed the evolution of the permeate volume collected (V_p) over time (t) and we calculated the permeate flux (J_v) according to Eq. (2) and retention rate (R) according to Eq. (1) corresponding to each TMP.

The results of permeate analysis are shown in Table 3. Fig. 3 shows the evolution of permeate flux J_v as a function of TMP ΔP for both pure water and dairy effluent.

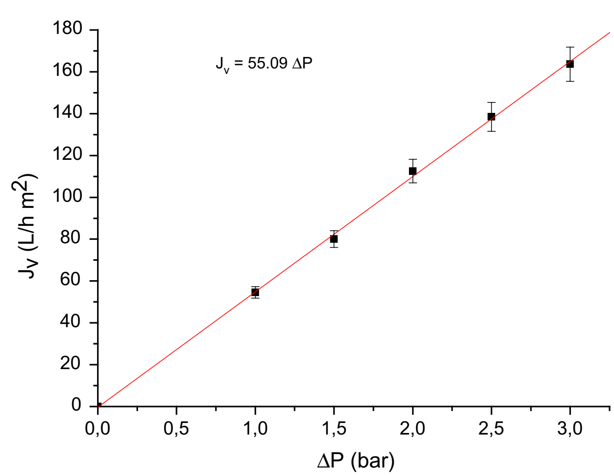

Fig. 2. Pure water permeability of PES-5 membrane at room temperature 25°C.

We note that the permeate flux varies linearly with the TMP and that permeability coefficients were similar in the case of ultrapure water and the dairy effluent. It would therefore be preferable to increase the

Table 3
Effect of ΔP on physico-chemical characteristics of permeates

ΔP (bar)	1.0	1.5	2.0	2.5	3.0
Conductivity (μS/cm)	2,700	2,730	2,710	2,530	2,830
Turbidity (NTU)	0.15	0.17	0.18	0.15	0.19
TDS (mg/L)	1,220	1,270	1,230	1,140	1,170
SM (mg/L)	10	10	9	11	12
COD (mgO_2/L)	16	20	18	16	32
BOD_5 (mgO_2/L)	0	0	0	0	10
Protein (mg/L)	0.74	0.54	1.06	0.33	1.03
Oils and fats (mg/L)	2.4	1.5	2	1.57	2.11

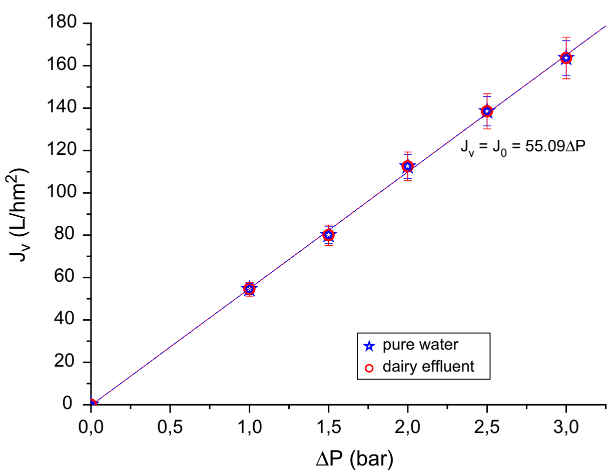

Fig. 3. Permeate flux variation with TMP using PES-5 membrane at room temperature 25˚C.

TMP to get a good permeate flux without reaching much excessive pressure to minimize energy consumption. The compromise is to operate at moderate pressure, which ensures obtaining good permeate fluxes and quality without high energy consumption.

In addition, there should be a good quality permeate. For this, Fig. 4 shows the variation of retention parameters analyzed in terms of the TMP.

Fig. 4 shows that low TMP is sufficient to achieve retention rates exceeding 90% of TDS, COD, turbidity, BOD5, proteins, and fats and oils. In the case of the conductivity and dissolved salts, retention rates obtained are about 40 and 55%, respectively. The results shown in Table 3 show that permeate quality compliant to both standards NT106-02 and NT106-03 means that it will be possible to reject dairy effluent into the environment or to reuse it in the process.

Fig. 4 also shows that, between 1 and 2.5 bar, the retention rates of parameters analyzed remain constant or undergoes a slight increase. Beyond 2.5 bar, a

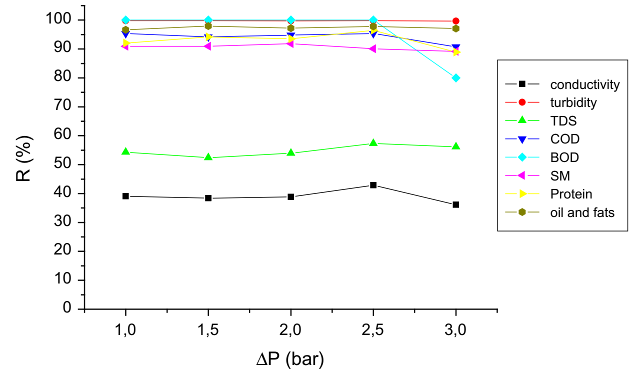

Fig. 4. Influence of TMP on the retention rates of analyzed parameters.

decrease in retention of the majority of the parameters analyzed was observed. This result can be explained by the occurrence of concentration polarization phenomenon, which limits the performance of the ultrafiltration process.

Taken into account these results and to have good performance and good permeate flux, we chose to operate at optimum TMP 2.5 bar for the rest of this work.

3.3. Effect of VRF

The evolution of permeate flux as a function of VRF was followed at constant TMP and temperature ($\Delta P_{\mathrm{optimum}}$= 2.5 bar and T = 25˚C) as shown in Fig. 5. In this study, 500 mL of dairy effluent is introduced into the feed cell and we followed the evolution of the permeate volume collected (V_p) over time (t) and we

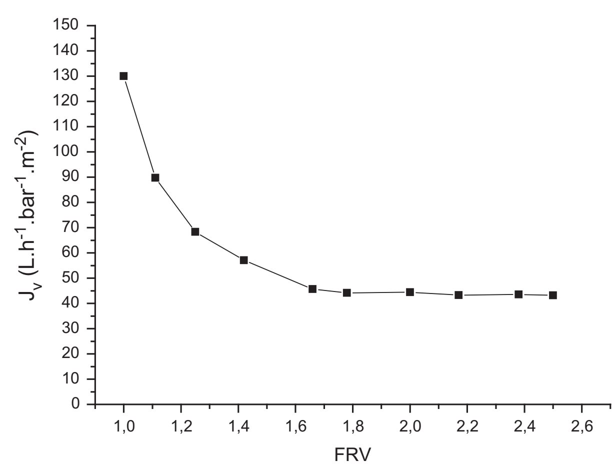

Fig. 5. Permeate flux variation under optimal conditions ($\Delta P_{\mathrm{optimum}}$= 2.5 bar and T = 25˚C).

Table 4
Physico-chemical analysis of permeates-based VRF ($\Delta P_{optimum}$= 2.5 bar and T = 25˚C)

VRF	1.11	1.25	1.42	1.66	1.78	2	2.17	2.38	2.5	Average value
Conductivity (μs/cm)	2,500	2,520	2,560	2,590	2,585	2,572	2,590	2,589	2,710	2,579
Turbidity (NTU)	0.15	0.15	0.16	0.20	0.20	0.21	0.21	0.22	0. 25	0.19
TDS (mg/L)	1,140	1,150	1,170	1,270	1,200	1,232	1,250	1,245	1,700	1,262
COD (mg O_2/L)	17	18	19	24	20	21	22	25	40	23
BOD_5 (mg O_2/L)	0	0	0	0	0	0	0	0	0	0
SM (mg/L)	12.5	12	11	18	21	22	20	21.5	23	17.9
Protein (mg/L)	0.35	0.36	0.39	0.41	0.40	0.42	0.43	0.45	0.52	0.41
Oils and fats (mg/L)	1.67	1.70	1.96	0.66	1.72	1.80	1.74	1.71	1.61	1.61

Table 5
Study of the retention rates of analyzed parameters in terms of VRF

VRF	R (Conductivity) (%)	R (TDS) (%)	R (SM) (%)	R (COD) (%)	R (Protein) (%)	R (Fat/oil) (%)	R (Turbidity) (%)	R (BOD) (%)
1.11	43.56	57.30	88.70	95.08	96.23	97.68	99.78	100
1.25	43.11	56.92	89.15	94.79	96.12	97.63	99.78	100
1.42	42.21	56.17	90.05	94.50	95.80	97.27	99.76	100
1.66	41.53	52.43	83.73	93.06	95.59	99.08	99.70	100
1.78	41.64	55.05	81.01	94.21	95.69	97.61	99.70	100
2	41.94	53.85	80.11	93.93	95.48	97.50	99.67	100
2.17	41.53	53.18	81.92	93.64	95.37	97.58	99.69	100
2.38	41.55	53.37	80.56	92.77	95.17	97.62	99.67	100
2.5	38.82	36.32	79.21	88.43	94.40	97.76	99.63	100

calculated the permeate flux (J_v) and retention rate (R) corresponding to each value of VRF to evaluate the effectiveness of the membrane treatment.

After each experiment, the PES-5 membrane was washed using pure water and the measurement of water permeability is checked to ascertain the absence of clogging and regain reference flux (J_0).

Fig. 5 shows that J_v decreases gradually when the permeate volume increases and then reaches a constant value of about 44.15 L/h m^2, which represents 32% of the initial flux. This was attributed to concentration polarization phenomenon.

We also notice that the initial permeate flux (J_v = 89.77 L/h m^2) represents 65.19% of the reference flux obtained by pure water (137.7 L/h m^2). This result confirms the predominance of the concentration polarization phenomenon that affects in a very short time the PES-5 membrane performances.

Tables 4 and 5 illustrate the results obtained for the various parameters analyzed after treating the dairy wastewater by UF under optimal conditions previously presented. These results are shown as a function of VRF.

For VRF between 1.1 and 2.38, retention rates remain almost invariable and show that the characteristics of permeate recovered after ultrafiltration treatment undergoes a significant improvement. However, a slight decrease in retention related to conductivity, TDS, SM, COD, and proteins was observed for VRF equal to 2.5.

According to Eq. (3), $V_i = \text{VRF} \times V_R = \text{VRF} \times (V_i - V_p)$, which means that $V_p = 0{,}58 \times V_i$ at VRF = 2.38. This result shows that a recovery of 58% of dairy effluent after treatment using PES-5 ultrafiltration membrane becomes possible.

In addition, the comparison of the physico-chemical characteristics of permeate treated by ultrafiltration using PES-5 membrane to those imposed by the Tunisian standard NT106-02 shows that the quality of the permeate present levels do not exceed the limits of this standard for the various parameters studied.

4. Conclusion

This work demonstrated that single UF treatment of Tunisian dairy wastewater was a viable and promising method to recycle and reuse water. The PES-5 membrane was suitable to improve the wastewater quality under optimal conditions of TMP and VRF, which makes its discharge possible without risk of contamination (according to NT106-02 Tunisian standard) and its reuse feasible (according to NT106-03 Tunisian standard). A recovery of 58% dairy effluent after UF process was achieved. This means less consumption of water and energy.

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