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Reclamation of water from dairy wastewater using polymeric nanofiltration membranes

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ABSTRACT

Water recovery from the dairy wastewater involves several integrated steps while the key process is nanofiltration (NF) that might reduce the water consumption in the dairy industry. However, NF membrane should be selected adequately enabling efficient filtration performance. This paper presents how the efficiency of the water recovery from dairy wastewater is affected by the type of NF membrane. In the study, two types of thin film composite membranes were investigated, such as DL and TS80. It was shown that TS80 membrane characterized with the properties similar to that of reverse osmosis (RO) membrane, which resulted in higher quality of the water produced. This membrane significantly contributed to recovery of water of high purity from dairy wastewater. Nevertheless, the diverse composition of dairy wastewater resulted in a rapid deterioration of filtration properties of polymeric NF membranes. Thus, it is necessary to select appropriate pretreatment steps prior to NF of dairy wastewater.

Keywords: Nanofiltration; Polymeric membrane; Dairy wastewater; Water regeneration

1. Introduction

The dairy industry is considered as one of the largest food industries, both in terms of the total weight of the processed raw material and water consumption. The amount of water consumed by dairy plant depends on the production profile and varies within a wide range of 0.5–11 dm^3 of water per 1 dm^3 of milk [1]. The dairy industry is prevalently considered as a largest source of wastewater from food processing, since approximately 80%–90% of water consumed by dairy plants becomes a wastewater [2]. Analysing the production technologies of individual products and the functioning of industrial dairy plants, it was found that the plants generated wastewaters such as milk water formed during concentration and demineralization of whey; white water produced during first washing of plant; and spent washing baths resulting from cleaning the production lines with acidic solutions, alkaline detergents or using automatically controlled CIP (clean-in-place) system [3,4]. In several dairy plants, wastewaters from different production processes are blended together and subjected to biological treatment. Sustainable water management provides the opportunities for technologies of modern wastewater treatment to close water cycles in dairy plants. These involve membrane separation technologies that have already found numerous applications in dairies [5–8]. Low-pressure driven membrane processes involve micro- (MF) and ultrafiltration (UF). MF is widely used to remove microbes, separate and fractionate milk fats as well as to break down proteins during cheese and milk production [3,9]. UF process has found the application in concentration of whey protein from milk and whey [10] and to normalize milk for the production of cheese, yoghurt and other dairy products [11], and recently, to produce cheese [12]. High-pressure driven membrane processes such as nanofiltration (NF) and reverse osmosis (RO) have been widely used to desalinate and dehydrate the whey, which is a by-product of cheese production [13–15]. Furthermore,

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an interesting area of using membrane technologies is the regeneration of spent washing baths produced while cleaning the dairy process lines [4,16,17]. The main contaminants of spent washing baths are organic (proteins, lactose and fats) and chemical cleaning substances (acids, bases, enzymes and detergents) [16–19]. NF membrane is considered a convenient way to treat spent alkaline and acidic cleaning solutions [20–23], whereas RO process is proposed to treat dairy wastewaters [1,20–22]. However, RO is restricted by the high costs of operating process pressure and low permeate flux. A significantly less energy-consuming and efficient membrane process includes NF. The aim of this study was to investigate the possibility of using NF membranes to reclaim the water from raw dairy wastewater.

2. Experimental

2.1. Experimental set-up

The experiments were performed on the raw dairy wastewater generated during the cleaning of the technological line in the plant that produced milk, cream, kefir, buttermilk and cottage cheese. The dairy plant is located in Mazovian Voivodeship in Poland and produces approximately 400 m^3 of wastewater per day. The pollutants content was analysed in the samples from raw and pretreated wastewaters, permeate and retentate from NF. In order to separate suspended solids, it was necessary to pretreat the dairy wastewater prior to membrane filtration with a polypropylene bag filter with a cut-off of 5 μm (Allfilter). The following MF on ceramic filter (Aqua Filter) characterized by pore size of 0.3 μm, was carried out using *'dead-end'* laboratory system, which consisted of a pump and a filter in the housing. The MF process was conducted at 0.5 bar and feed flow rate 0.02 m^3/h. The deionized water flux of MF ceramic filter was 222 $dm^3/(m^2\,h)$, whereas an average permeate flux during microfiltration of dairy wastewater was 127 $dm^3/(m^2\,h)$. NF was performed in a batch mode using laboratory scale set-up (Fig. 1) under the transmembrane pressure equal to 14 bar and maintaining a crossflow velocity at 0.35 m/s. The value of transmembrane pressure was selected based on the previous studies carried out on dairy products or dairy wastewater [13,24,25]. The permeate was collected in a separate tank and a retentate stream was recycled to the feed tank. For NF process, 5 dm^3 of the feed was used, which was pretreated beforehand with a bag and ceramic filters. The process of NF was performed until 2.5 dm^3 of the permeate was received. During the process, the temperature in the feed/retentate tank was constant and equal to 25°C ± 1°C.

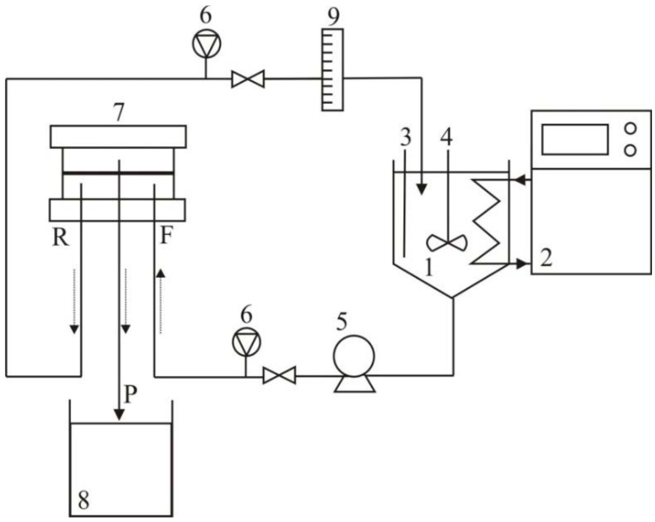

Fig. 1. The scheme of the laboratory scale membrane set-up: 1 – feed/retentate tank, 2 – thermostat, 3 – thermometer, 4 – mixer, 5 – pump, 6 – manometer, 7 – nanofiltration module, 8 – permeate tank, 9 – rotameter, P – permeate, F – feed and R – retentate.

2.2. Filtration materials

The ceramic MF filter and polymer membranes were used in experiments. The ceramic MF filter was characterized by an active area of 0.055 m^2. An active area of both NF membranes was 0.014 m^2. These two types of membranes are dedicated to protein-contaminated wastewaters based on the manufacturers' data sheets [26]. The NF membranes' surfaces are negatively charged with the low roughness (Table 1). Moreover, the membranes are characterized by a good chemical and thermal resistance. Each process of NF was performed with a new membrane.

Table 1
Characteristics of tested nanofiltration membranes

Membrane types	DL	TS80
Manufacturer	GE Osmonics	TriSep
Skin layer	Polypiperazine-amide	Polyamide
$MgSO_4$ retention, %	98	99
NaCl retention, %	–	80–90
Cut-off, Da	150–300	~150
Permeability coefficient, $dm^3/(m^2\,h\,bar)$	8.6	8.9
pH range	2–11	2–11
Maximum temperature, °C	50	45
Zeta potential, mV (pH ≈ 7, t = 25°C)	–22 [27]	–15 [28]
Surface roughness, nm (5 × 5 μm)	18 [27]	17 [28]

2.3. Materials and methods

The blended wastewaters were obtained from one dairy plant. These wastewaters were pretreated with polypropylene bag and ceramic filters and subjected to NF. The samples from raw, pretreated dairy wastewater, permeate and retentate were analysed in terms of total nitrogen, ammonium nitrogen, total phosphorous, chemical oxygen demand (COD), sulphates and volatile fatty acids (VFAs) using HACH cuvette tests for UV-vis DR6000 spectrophotometer; pH and conductivity were measured using Seven Multi-Mettler Toledo device. The concentrations of calcium, sodium, potassium, chloride and nitrate ions were determined using Mettler Toledo ion-selective electrodes. Turbidity was determined using the HACH measurement device. The dry residue was evaluated by the weight method. The samples of 5 cm^3 were placed on disposable aluminium plates in a Radwag MAC 50/1 moisture analyser. A standard drying profile was used (no mass change of 0.001 g within 60 s at 105°C). A standard counting-plate method was used to determine the number of microorganisms in the raw dairy wastewater and the permeate. Using a flame-sterilized bacteria spreader, the volume of 0.5 cm^3 of diluted samples was spread on the growth media containing agar. The plate was incubated at 37°C for 24 h. Then, the CFU was counted on the plates containing between 30 and 300 colonies.

2.4. Calculated parameters

The performance of NF membranes was evaluated based on permeate flux [Eq. (1)] as follows:

$$J_P = \frac{V_P}{A \times t} \tag{1}$$

where J_P – permeate flux, dm^3/(m^2 h); V_P – permeate volume, dm^3; A – membrane area, m^2 and t – time needed to receive a defined volume of permeate, h.

The assessment of NF efficiency of the dairy wastewater was based on the volume reduction factor [Eq. (2)] and retention coefficient [Eq. (3)] as follows:

$$\text{VRF} = \frac{V_F}{V_R} \tag{2}$$

where VRF – volume reduction factor, –; V_F – feed volume, dm^3 and V_R – retentate volume after defined permeate receiving time, dm^3.

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100\% \tag{3}$$

where R – retention of component, %; C_P – concentration of a component in permeate, mg/dm^3 and C_F – concentration of a component in feed, mg/dm^3.

The number of bacteria in 1 cm^3 of wastewater was counted according to the Eq. (4) as follows:

$$N = \frac{n \times \text{DF}}{V_C} \tag{4}$$

where N – number of bacteria, CFU/cm^3; n – number of colonies, CFU; DF – dilution factor and V_C – volume of bacteria suspension spread on agar culture plate, cm^3.

3. Results and discussion

3.1. Pretreatment of the dairy wastewater

The first step of the experiment involved examination of the composition of the blended dairy wastewaters (Table 2), which were found to have a diverse composition indicating on high levels of both mineral and organic substances. The wastewaters contained mono- and divalent ions, VFAs and microorganisms. The complexity of the composition was due to different types of production in the dairy plant, characteristics of cleaning agents and charges fluctuations during the day [23].

The particular stages of filtration system to regenerate water from the dairy wastewater are presented in Fig. 2. Owing to the complex composition of the dairy wastewater (Table 2), it was necessary to appropriately pretreat wastewater prior to NF.

The bag filter and ceramic filter with a pore diameter of 5 and 0.3 μm, respectively, were used to pretreat the dairy wastewaters. The results are presented in Fig. 3. Pretreatment of the dairy wastewaters using a bag filter was necessary so as to remove the larger solid particles. This resulted mainly in reducing the dry residue (Fig. 3), turbidity and pH of the dairy wastewater. Significantly, microfiltration reduced the viability of bacterial cells (CFU), organic matter (COD, VFAs) and nutrients such as nitrogen and phosphorus compounds, (Fig. 3). The retention of ammonium nitrogen of only 2%–10% was due to the salts of lactic acid and ammonia (ammonium lactate) in dairy wastewater that could easily pass through the MF system.

3.2. Nanofiltration of dairy wastewaters

The dairy wastewaters were pretreated in microfiltration on ceramic filters (0.3 μm) and subsequently via NF. It was found that the NF run on both types of membranes resulted in the partial retention of organic matter (Fig. 4). However, the process with TS80 was significantly more efficient. Apart from the decrease in the organic matter (Fig. 4), TS80 membrane also diminished the content of the VFAs by 87%. This shows that TS80 membrane has the separation properties similar to that of RO membrane. It has been found that NF

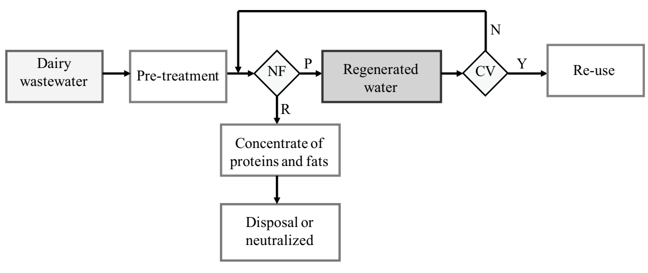

Fig. 2. The scheme involving nanofiltration (NF) process to regenerate water from the dairy wastewater: NF – nanofiltration, P – permeate, R – retentate, Y – yes, N – no and CV – convergence.

Table 2
Compositions of the dairy wastewaters used in the experiments

Parameter	Dairy wastewater I	Dairy wastewater II
pH	8.5	7.4
Turbidity, FNU	2,144	1,788
Conductivity, µS/cm	1,410	1,651
Dry residue, g/dm³	4,840	4,600
Chemical oxygen demand (COD), mg/dm³	7,510	6,660
Total nitrogen, mg/dm³	141	107
Ammonium nitrogen, mg/dm³	1.3	17.4
Total phosphorous, mg/dm³	17.8	26.5
Calcium, mg/dm³	48.9	93.0
Sulphates, mg/dm³	419	577
Sodium, mg/dm³	105	380
Potassium, mg/dm³	92.4	55.0
Nitrates, mg/dm³	193.2	2.5
Chlorides, mg/dm³	72.4	59.0
Volatile fatty acids (VFAs), mg/dm³	486	633
Bacteria, CFU/cm³	13.0E+07	10.5E+11

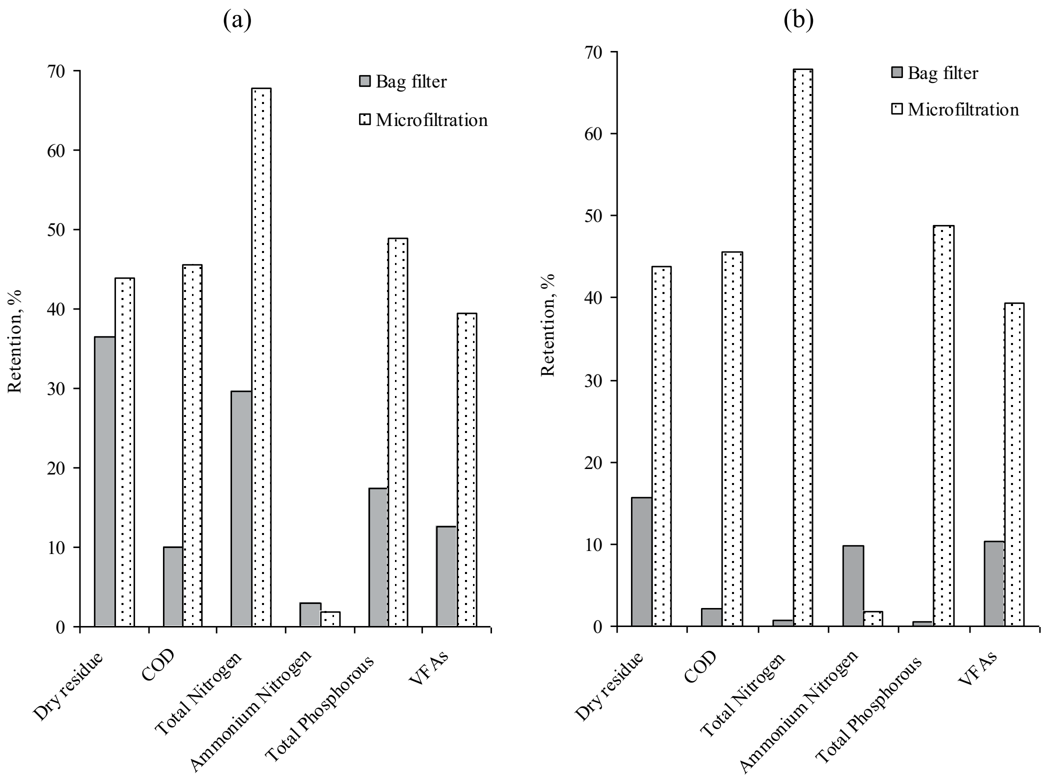

Fig. 3. The retention of components in dairy wastewaters I (a) and II (b) during pretreatment with a filtration system on a bag filter and microfiltration.

membranes might have even much better filtration properties than RO membranes [29]. RO process is generally characterized by high transmembrane pressure and inevitable high operating costs of the dairy wastewater treatment.

The characteristic feature of NF membranes is the ability to selectively separate inorganic salts ions [30–32]. The retentions of mono- and divalent ions in the dairy wastewaters within the process of NF on two selected types of membranes are presented in Fig. 5. DL membrane resulted in a high permeability of monovalent ions (K^+, Na^+, Cl^- and NO_3^-) and retention of divalent ions (Ca^{2+} and SO_4^{2-}). TS80 membrane was characterized by a higher retention of both

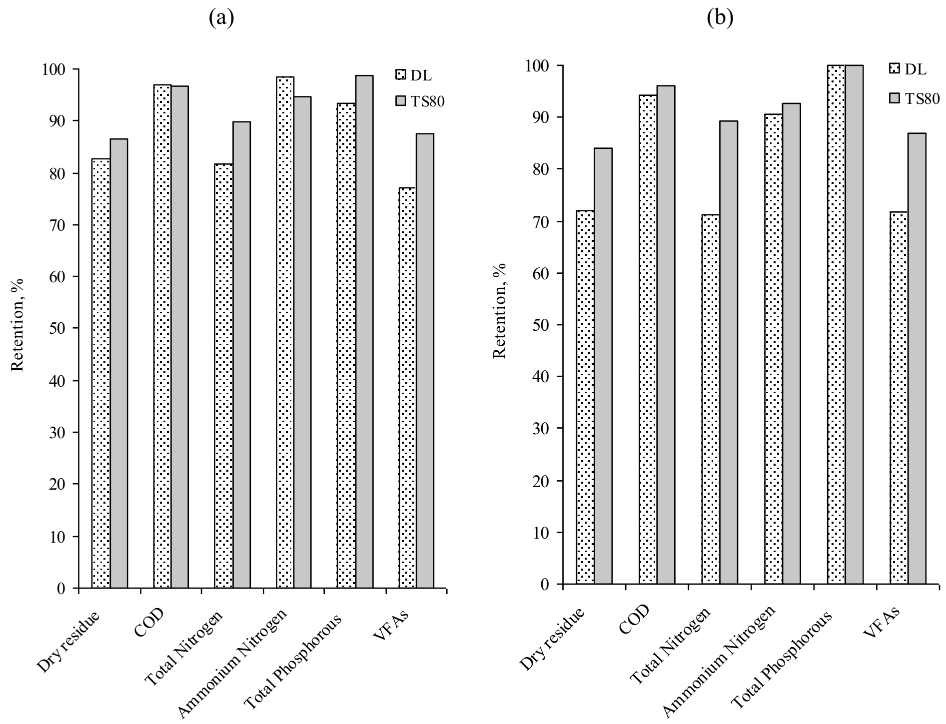

Fig. 4. The retention of components of dairy wastewaters I (a) and II (b) during nanofiltration carried out on DL and TS80 membranes.

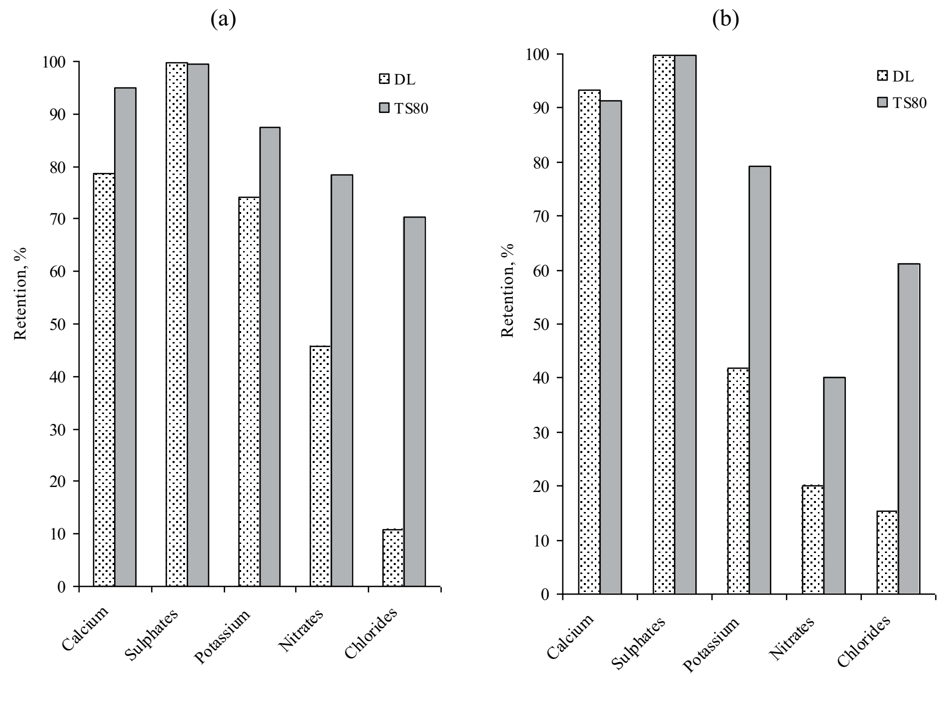

Fig. 5. The retention of ions of dairy wastewaters I (a) and II (b) during nanofiltration carried out on DL and TS80 membranes.

mono- and divalent ions than DL membrane. As a result, the permeate from NF using TS80 membrane, was less than twice as conductive as permeate, which passed through DL membrane (Table 3).

The physicochemical parameters of the water received in NF of the dairy wastewater II using two different types of membranes (Table 2) were compared with the limits forced by Regulation of the Minister of Environment from 18 November 2014 on the discharge of effluents into environment (Table 3).

As it turned out, neither the DL permeate nor TS80 did not meet the COD safe values for effluent discharged into environment. According to Cuartas-Uribe et al. [13], organic compounds such as lactose molecules, which determine the COD value is able to permeate through NF membranes. The permeate resulting from NF also contained VFAs, which caused unpleasant odour of the water (Table 3). Hence, such contaminated water cannot be recycled to the dairy plants processes. Based on the pollutants parameters obtained for reclaimed water (Table 3), it was found that such water could be used for technological purposes to wash tanks, cars and floors. The similar results were obtained during NF of dairy wastewater I on DL and TS80 membranes.

The state of the art [33–36] has shown that there is a possibility of integrating membrane filtration techniques with other methods such as advanced oxidation and activated carbon adsorption. Such an integration aims at achieving a synergistic effect in the elimination of VFAs and COD.

Table 3
Quality of permeates obtained in NF on DL and TS80 membranes referred to Regulation of the Minister of Environment on the discharge of effluents into environment (18 November 2014, Warsaw, Poland)

Parameter	Permeate after DL membrane	Permeate after TS80 membrane	Environmental discharge limits
pH	8.0	7.9	6.5–9.0
Turbidity, FNU	0.38	0.52	–
Conductivity, μS/cm	837	275	–
Dry residue, mg/dm^3	740	420	–
Chemical oxygen demand (COD), mg/dm^3	184	130	125
Total nitrogen, mg/dm^3	6.99	2.59	30
Ammonium nitrogen, mg/dm^3	1.58	1.25	10
Total phosphorous, mg/dm^3	0.022	0.008	2
Calcium, mg/dm^3	6.2	8.0	–
Sulphates, mg/dm^3	1.27	1.58	500
Sodium, mg/dm^3	178.0	14.8	800
Potassium, mg/dm^3	32.0	11.4	80
Nitrates, mg/dm^3	2.0	1.5	50
Chlorides, mg/dm^3	50	23	1,000
Volatile fatty acids (VFAs), mg/dm^3	94.5	58.7	–

(a) (b)

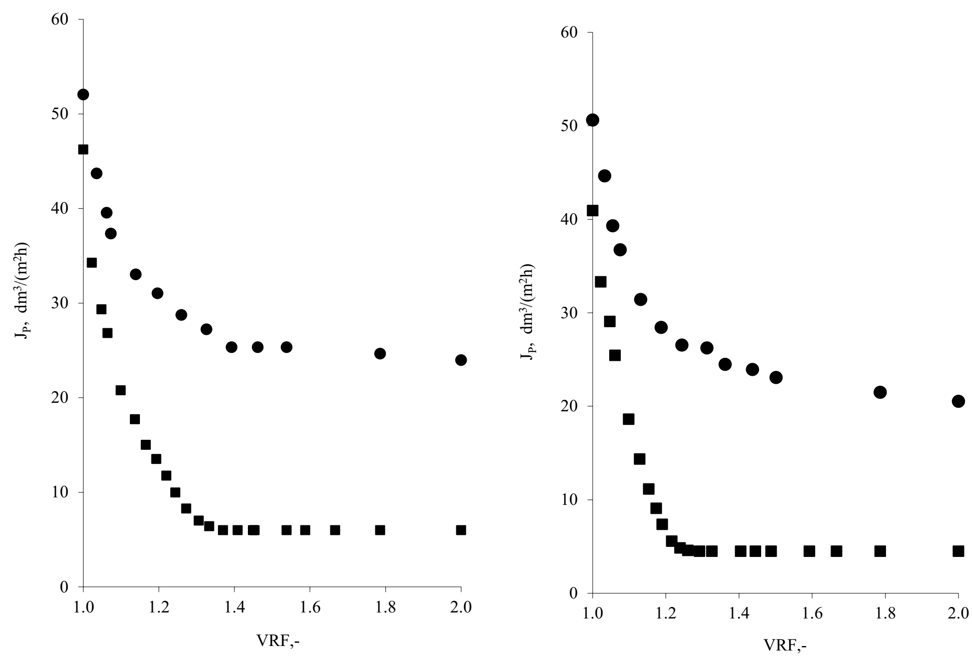

Fig. 6. Permeate flux (J_p) versus Volume Retention Factor (VRF) of nanofiltration of dairy wastewaters I (■) and II (●) on DL (a) and TS80 (b) membranes.

Adsorption on activated carbon and advanced oxidation processes can be used as a pre- or posttreatment of industrial wastewaters in a combination with NF. This might result in obtaining water, parameters of which will meet Drinking Water Directive.

3.3. Fouling of NF membranes used to treat dairy wastewater

During NF of both dairy wastewaters pretreated beforehand, the decrease in permeate flux (J_p) was observed (Fig. 6). The treatment of both dairy wastewater I and II on DL membrane (Fig. 6(a)) resulted in the permeate flux decrease by 86% and 54%, respectively. However, the permeate flux for TS80 membrane (Fig. 6(b)) decreased by 89% and 59%, respectively. This is due to the fact that during the batch-mode process, an increase in the concentration caused a decrease in permeation. The reduction in permeate flux overtime was due to the adsorption of the components of the dairy wastewaters on the membrane surfaces. Current state of the knowledge [12,37–41] shows the influence of the composition of the feed and the properties of the membranes on the fouling phenomenon.

Hausmann et al. [37] have found that protein adsorption on the surface of membranes strengthens the presence of polyvalent salts. However, Nanda et al. [40] and Vrijenhoek et al. [41] have indicated that the less rigid membranes exhibit less tendency to retain both organic and inorganic molecules. Proteins and lactose have been identified as the dominant components of fouling polymeric membranes used in the whey and milk filtration [37]. Presumably, such composition of fouling is commonly observed on polymer membranes during NF of dairy wastewaters. Thus, in order to prevent this effect, it is necessary to appropriately select the pretreatment steps.

4. Conclusions

The results have shown that the TS80 membrane in the dairy wastewater treatment is more efficient compared with DL membrane. This membrane provides a high retention of both organic compounds and divalent ions. This also contributes to a significant decrease in monovalent and VFAs contents. The outcomes of this study allow to propose the reclaimed water to be reused for external washing of tanks, car tanks and floors. Moreover, the results have shown that the compositions of the dairy wastewaters adversely affect filtration properties of both NF membranes tested. The decrease in the permeate flux during NF of dairy wastewaters was due to the deposition of the components of wastewaters on the surfaces of the tested membranes. Therefore, it is necessary to continue experiments on the possibilities of minimizing the fouling effects associated with the regeneration of water from dairy wastewater by NF and to develop an effective chemical membrane regeneration procedure.

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