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## REGENERATION OF SPENT CAUSTIC AND ACIDIC CLEANING BATHS USING A PILOT MEMBRANE SYSTEM

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**Key words:** caustic and acidic cleaning baths, regeneration, microfiltration, ultrafiltration, nanofiltration.

**Abstract:** In this work, the concept based on using pressure driven membrane processes was developed to effectively recover caustic and acidic cleaning baths that wash out of technological lines in dairy plants. As a result, the influence of both microfiltration (MF) and the integrated system of micro- and ultrafiltration (MF/UF) process parameters were assessed on the efficiency of the regeneration of caustic cleaning baths. It was found that MF process allows reducing the level of contaminants in caustic cleaning baths, yet the integrated MF/UF system is more efficient in the removal of high molecular organic compounds. In turn, during the regeneration of acidic cleaning baths, the influence of process parameters of ultrafiltration (UF) and integrated ultra- and nanofiltration systems (UF/NF) on their effectiveness was examined. As a result, it was confirmed that integrated UF/NF system is crucial to regenerate acidic cleaning baths.

### Regenerowanie zasadowych i kwasowych zużytych kąpeli myjących przy użyciu pilotowych instalacji membranowych

**Słowa kluczowe:** alkaliczne i kwasowe kąpeli myjące, regeneracja, mikrofiltracja, ultrafiltracja, nanofiltracja.

**Streszczenie:** W ramach przeprowadzonych prac badawczych opracowano koncepcję efektywnego regenerowania alkalicznych i kwasowych kąpeli myjących powstających podczas mycia linii technologicznych zakładów mleczarskich. W wyniku przeprowadzonych eksperymentów dokonano oceny wpływu parametrów procesowych mikrofiltracji (MF) i zintegrowanego układu mikro- i ultrafiltracji (MF/UF) na efektywność regenerowania alkalicznych kąpeli myjących. Stwierdzono, że proces MF umożliwia zmniejszenie poziomu zanieczyszczenia alkalicznych kąpeli myjących, ale dopiero zastosowanie układu MF/UF pozwala na usunięcie wielkocząsteczkowych związków organicznych. Z kolei podczas regenerowania kwasowych kąpeli myjących zbadano wpływ parametrów procesowych ultrafiltracji (UF) i zintegrowanego układu ultra- i nanofiltracji (UF/NF) na ich efektywność. Na podstawie przeprowadzonych prac badawczych stwierdzono, że do regenerowania kwasowych kąpeli myjących niezbędne jest wykorzystanie układu procesów UF/NF.

## Introduction

The European Commission recently adopted an ambitious Circular Economy Package, which includes revised legislative proposals on waste to stimulate Europe's transition towards a circular economy which will boost global competitiveness, foster sustainable economic growth, and generate new jobs. Over the course of its mandate, the Commission intends to support a series of actions on water reuse, including a legislative proposal on minimum requirements for the reuse of wastewater. The proposed actions will

contribute to “closing the loop” of product life cycles, through greater recycling and reuse, and bring benefits for both the environment and the economy [1]. In Poland, the industrial wastewater discharged into the sewerage system or into environment or recycled to the production process must meet the criteria specified in the relevant legal acts, including the following: on the conditions for the wastewater introduction into sewerage equipment [2], on the wastewater discharged into water or soil [3], and on the quality of water intended for human consumption (in case of water recovered for its use in food production) [4].

The food industry consumes large amounts of water, which is involved in many processes and unit operations, including production and cleaning purposes [5]. In particular, the dairy industry is one of the most polluting food industries considering the generated effluent [6]. Around 12 billion tons of milk is produced annually in our country, which leads to the production of around 92 000 m<sup>3</sup> of wastewater per day. Depending on the technology used and the production of the assortment, the unit water consumption is from 3 L up to 20 L on 1 L processed milk [7, 8].

Proper reconditioning (treatment of water intended to be reused) and reuse of wastewater in the food industry is a promising alternative to current practices of discharging these streams in places where they can negatively affect the environment [9, 10].

Membrane processes are frequently used in dairy product manufacturing [11]. However, membrane fouling is a serious problem in the application of these technology [12, 13]. To maintain the cleanliness and the hygiene of the membranes, pipes and dairy plants, require regular chemical cleaning. For ensuring hygienic safety of foods and for recovering plants performances, Cleaning-in-place (CIP) systems are commonly used in the food industry [14–19]. Depending on the processing practice and load of soiling on the process equipment, the cleaning solutions may be used in a single cycle or recycling and reused for multiuse. The effectiveness of cleaning is preconditioned by factors like chemical agents, mechanical power, temperature, and time of the procedure [15]. In the CIP system, cleaning solutions used include various types of detergents, sanitizers, or disinfectants. The detergents used in CIP system may be alkali or acid detergents. The commonly used alkali detergents are Sodium Hydroxide (NaOH), Potassium Hydroxide (KOH), and Sodium Carbonate (Na<sub>2</sub>CO<sub>3</sub>), and the acid detergents include Nitric Acid (HNO<sub>3</sub>), Hydrochloric Acid (HCl), Phosphoric Acid (H<sub>3</sub>PO<sub>4</sub>), and Citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>). Traditionally, the CIP process involves a series of washing steps in which caustic and acidic solutions are applied in concentrations of 0.5–2.0% w/w, followed by rinsing with water [16, 17]. The recycling of waste streams from CIP has the advantage that it reduces the need for expensive raw materials and fresh water, and thus the cleaning cost decreases. It is possible to use membrane filtration to recycle of the cleaning solutions. In the literature, there are only a limited number of studies on the recovery of detergents or detergent compounds for recycling in CIP processes [17–19]. Microfiltration (MF) can be used to remove solids in suspension as a pre-treatment method, while ultrafiltration (UF) can be proposed as a method for the removal of surfactants from aqueous solutions with critical micelle concentration (CMC). However, when the concentration is as low as the monomer concentration, nanofiltration (NF) is an effective removal process [18].

In order to recover acidic solutions from the dairy industry cleaning process, a number of experiments were carried out by Novalic et al. [19]. Two acidic solutions with different COD were passed through NF membranes. The highest permeate flux and the lowest COD (in the feed solution) were obtained for the solution that had been applied after the caustic step in the cleaning process. This was because the degree of contamination in the spent acidic solution was much lower. While, Merin et al. [20] shown that when MF and NF permeates of reused solutions were put to a cleaning test using UF membranes fouled with whey proteins, but they achieved better cleaning efficiency compared to newly prepared NaOH solutions. The life span of these solutions at the dairy plant is arbitrarily fixed with no scientific basis [20]. The recycling of caustic solutions has an enormous economic impact, since the consumption of caustic soda in the dairy industry is twice that of nitric acid. However, the recovery of acidic solutions is also considered minimization of the discharge of nitrates into the environment. This leads to the saving of water, chemicals, and energy, and avoids the disposal of cleaning agents into the environment.

The aim of the present work was to study the possibility of the application of membrane filtration processes for effective regeneration of caustic and acidic streams from CIP processes of technological lines in dairy plants.

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## 1. Material and methods

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The experiments were performed on the model wastewater generated during the cleaning of the technological lines in the dairy plants. They were prepared on the base of milk powder (1.0 g/L), and accordingly, sodium hydroxide (2.0 g/L) or nitric acid (1.4 mL/L) and the tap water. The pH values of caustic and acidic wastewaters were approx. 12 and approx. 2, respectively. The caustic and acidic wastewaters contaminated with macromolecular organic compounds (in the form of proteins, fats and lactose) were regenerated with the pilot membrane installation set, which is the part of the module system of water recovery from wastewaters and for closing water cycles which was projected and build in ŁUKASIEWICZ – ITeE in Radom.

### 1.1. Pilot membrane installation

In the first stage of the study, the caustic and acidic wastewaters contaminated with macromolecular organic compound were treated with one-stage membrane filtration. Each of the membrane filtration processes were performed in batch mode, which is a transient state process because of the change of volume in liquid phase (VRF – volume reduction factor) and the change in the

concentration of each of the components in feed solution. In the feed tank, 100L of model wastewater was placed, which was then pumped from the tank to the membrane module at a constant pressure that was adequate for the used membrane technique. After passing through the filtration module, the feed was separated into two different streams: stream of treated filtrate (permeate), and the stream of a concentrate (retentate). Filtrate

stream was collected in a separate tank and a retentate stream was recycled to the feed tank. As a result, the concentration of compounds which were retained by the membrane was gradually increasing. Microfiltration (MF) was performed using pilot membrane installation (Fig. 1) under the pressure between 0.5 and 2.0 bar, and the retentate flow was changed between 100 and 400 L/h.

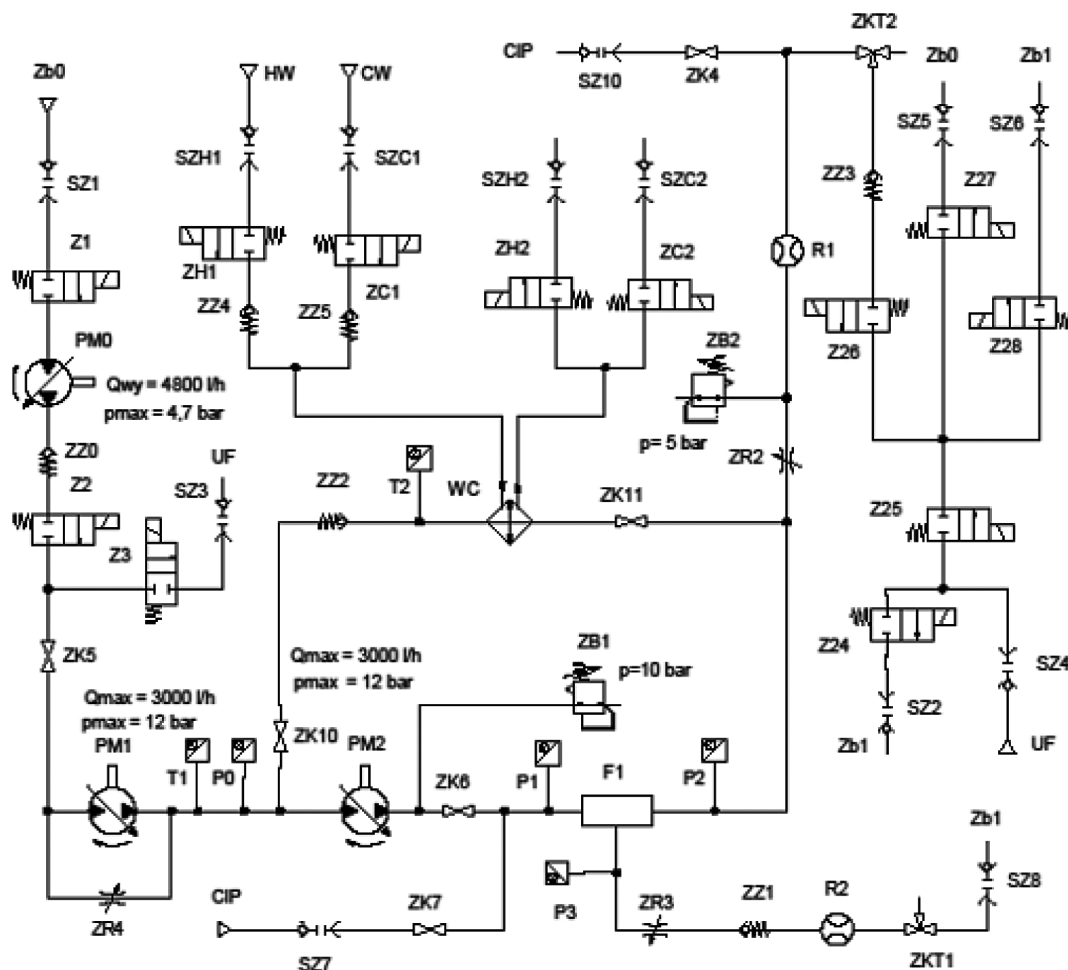


Fig. 1. The scheme of the pilot microfiltration installation: F1 – membrane module; P0, P1, P2, P3 – manometer; PM0, PM 1 – feed pump; PM2 – circulation pump; T1, T2 – electronic thermometer; R1, R2 – flowmeter; WC – heat exchanger; ZB1, ZB2 – emergency valve; ZH2, ZC2, ZC1, ZH1, Z1, Z2, Z3, Z27, Z25, Z26, Z28, Z24 – solenoid valve; ZH2, ZC2, ZC1, ZH1, Z1, Z2, Z3, Z27, Z25, Z26, Z28, Z24 ZK4 ZK5 ZK6, ZK7, ZK10, ZR2, ZK11 – ball valve; ZKT2, ZKT1 – 3-way ball valve; R4, ZR3 – needle valve; ZZ0, ZZ3, ZZ4, ZZ5 ZZ2, ZZ1 – return valve

In turn, ultrafiltration (UF) was performed using pilot membrane installation, which is shown on 3D visualization (Fig. 2) under the pressure between 4.0–8.0 bar, and the retentate flow changing between 100–250 L/h. The process of MF and UF was performed until 80 L of the permeate was received.

In the next stage of the study, the possibilities of using integrated systems of MF/UF and UF/NF were considered to regenerate spent cleaning baths. MF and UF was performed in a serial way. First, the MF process

was conducted and permeate was collected into separate tank and the retentate stream was recycled to the feed tank. MF was performed until 80 L of the permeate was received. The MF permeate was the feed for UF and UF permeate was collected into another separate tank, while retentate stream was recycled into the feed tank. UF was performed until 60 L of the permeate was received. The UF and NF were performed in the same way. Nanofiltration (NF) was carried out using the pilot membrane installation (Fig. 3).

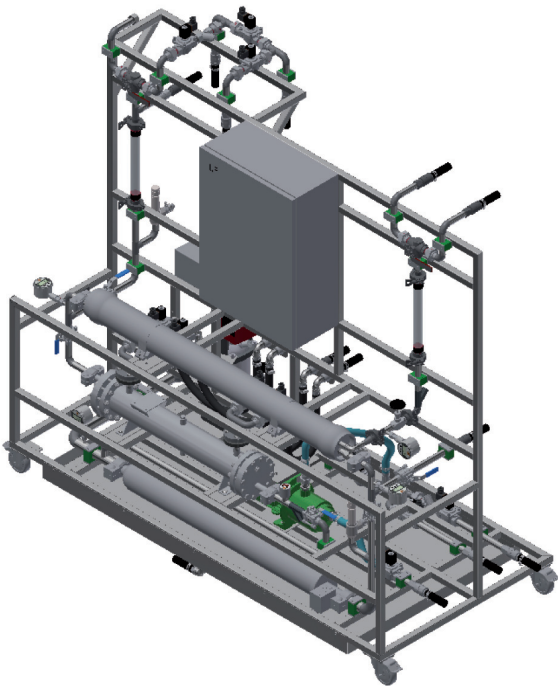


Fig. 2. 3D visualization of pilot ultrafiltration installation



Fig. 3. Photo of pilot nanofiltration installation

The spiral wound membrane modules were used in this study. The characterization of used membranes was shown in Table 1.

Table 1. Characteristics of tested membranes during MF, UF and NF processes

Parameters	MF membrane	UF membrane	NF membrane
Manufacturer	TriSep	Microdyn-Nadir	TriSep
Model	TM10-QXF	UP150	TS80
Polymer	PVDF	PES	PA
Active area, [m <sup>2</sup> ]	5.8	6.0	8.2
Pore size, [μm]	0.2	–	–
Cut-off, [kDa]	–	150	–
Max. pressure, [bar]	4	10	41
Max. temperature, [°C]	45	45	45
pH range	1–12	2–11	1–12

PVDF – polyvinylidene fluoride, PES – polyether sulfone, PA – polyamide

The basic physical and chemical properties each of streams (feed, permeate and retentate) was investigated after each membrane process was finished. The value of pH was measured using a Seven Multi Mettler Toledo device. Turbidity was determined using a HACH measurement device. Samples of feed, permeate, and retentate were analysed in terms of total bound nitrogen (TNb), total phosphorous (TP), chemical oxygen demand (COD), and total organic carbon (TOC), using HACH cuvette tests for a UV-VIS DR 6000 spectrophotometer. The obtained results of physical and chemical analyses were used to determine the retention of components that shows the efficiency of wastewater regeneration. During each of the processes, the permeate flux and volume reduction factor was analysed, which are the basic parameters that shows the efficiency of filtration processes. The equations used to determine parameters to control the processes are shown in Table 2.

Table 2. Parameters controlled during regeneration of wastewater by pilot membrane installation

Parameter	Equation
Retention ( $R_i$ )	$R_i = \left(1 - \frac{C_p}{C_{R/N}}\right) \cdot 100\%$
Permeate flux ( $J_p$ )	$J_p = \frac{V_p}{A_m \cdot t}$
Volume reduction factor (VRF)	$VRF = \frac{V_F}{V_R}$

$C_N$  – concentration of component in feed [mg/L],  $C_R$  – concentration of component in retentate [mg/L],  $C_p$  – concentration of component in permeate [mg/L],  $A_m$  – membrane area [m<sup>2</sup>],  $t$  – time needed to receive a defined volume of permeate [h],  $V_N$  – volume of feed [L],  $V_R$  – volume of retentate [L],  $V_p$  – volume of permeate [L].

## 2. Results

### 2.1. Regeneration of caustic cleaning baths using MF and MF/UF

The wastewaters generated during the caustic cleaning of the technological lines in the food industry using sodium hydroxide (NaOH) were treated in microfiltration. The influence of technological parameters (intensity of retentate flow –  $Q_R$  and transmembrane pressure – TMP) on the efficiency of caustic wastewaters regeneration was investigated (Fig. 4). MF was performed on three different retentate flow values (100 L/h; 250L/h; 400L/h) and at constant transmembrane pressure (2.0 bar) using two feed pumps and one circulation pump. The highest efficiency of the regeneration of caustic wastewaters generated during CIP cycles was observed at the intensity of retentate flow

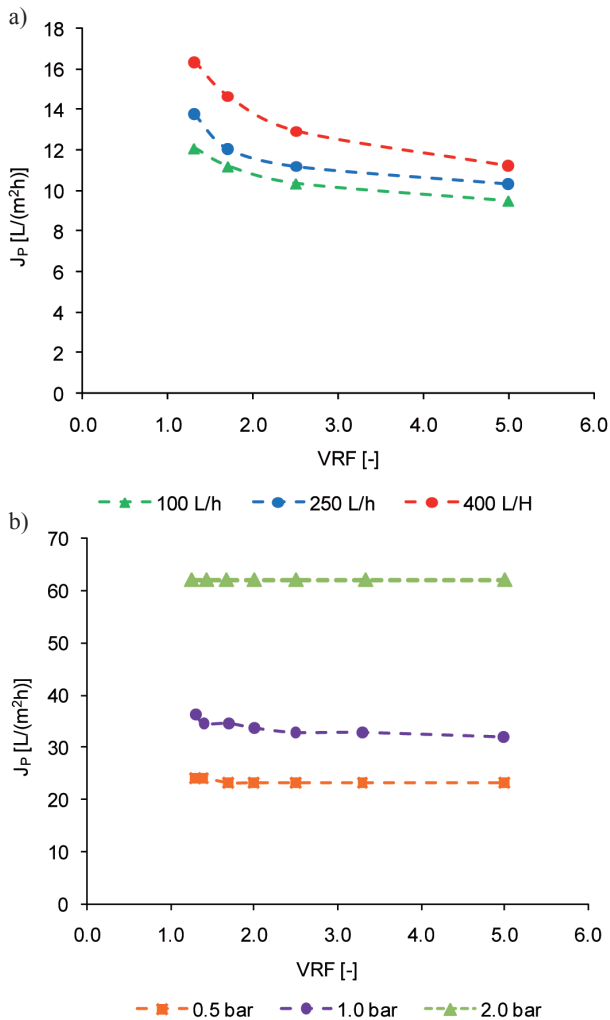


Fig. 4. Permeate flux ( $J_p$ ) vs. volume reduction factor (VRF) during MF of the caustic cleaning bath: (a) the process carried out at constant transmembrane pressure (TMP = 2.0 bar); (b) the process carried out at a constant retentate flow ( $Q_R = 400$  L/h)

of 400 L/h (Fig. 4a). In turn, when MF was performed at three different values of pressure (0.5 bar; 1.0 bar; 2.0 bar), which were kept using one feed pump and one circulation pump and a constant value of retentate flow (400 L/h), it was found that using higher transmembrane pressure resulted in the improvement of the efficiency of regeneration caustic wastewaters generated during CIP cycles (Fig. 4b). However, it was observed that the drop of permeate flux in relation to the time of volume reduction factor that depends on MF process parameters (Fig. 4). Considering the efficiency of the regeneration of caustic wastewaters, it is favourable to perform MF at a transmembrane pressure of 2.0 bar and an intensity of retentate flow at 400 L/h (Fig. 4b). Application of these parameters during MF reduces fouling (deposition of components of filtration medium at the surface and inner structure of polymeric membranes).

MF was able to remove turbidity completely, while fractioning and concentrating components of the filtration medium at the same time. That was shown by different levels of the retention of total bound nitrogen (TNb), total phosphorous (TP), and COD, which was independent of the used process parameters (Fig. 5).

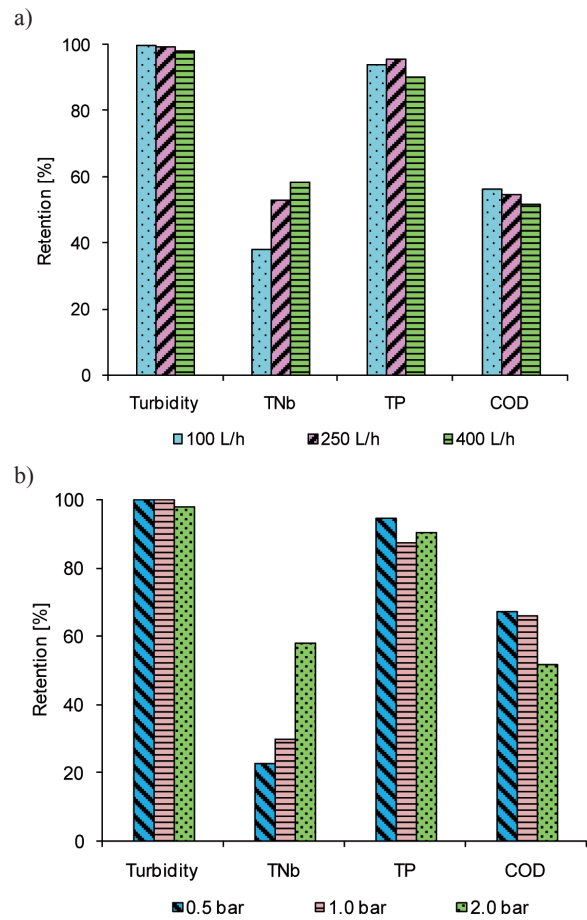


Fig. 5. Retention of caustic cleaning baths parameters after MF: (a) process performed at constant transmembrane pressure (TMP = 2.0 bar); (b) process performed at constant retentate flow ( $Q_R = 400$  L/h)

A comparison of basic psychical and chemical parameters for each of the streams (feed, permeate, retentate) of regenerated wastewater was shown in Table 3. Treating caustic wastewater that was contaminated by macromolecular organic compounds using MF caused a reduce level of contamination (Table 3). This also concerned both turbidity (from

1331 NTU to 0.7 NTU) and level of TP (from 14 mg/L to below 1 mg/L) and COD (from 1225 mg/L to 640 mg/L). In turn, the concentration of TNb was decreased slightly (from 41 mg/L to 27 mg/L). Application of the integrated MF/UF system allowed decrease in the concentration of TNb and COD to 9.0 mg/L and 303 mg/L, respectively.

**Table 3. Physical and chemical parameters values of caustic cleaning solution regenerated by MF\* and integrated MF/UF\*\* system**

Parameters	Feed	MF permeate	MF/UF retentate
Turbidity [NTU]	1331	0.7	0.8
TNb [mg/L]	40.9	26.9	9.0
TP [mg/L]	13.8	0.9	0.5
COD [mg/L]	1225	640	303

\*TMP<sub>MF</sub> = 2.0 bar, Q<sub>R</sub> = 250 L/h; \*\*TMP<sub>UF</sub> = 4.0 bar, Q<sub>R</sub> = 250 L/h

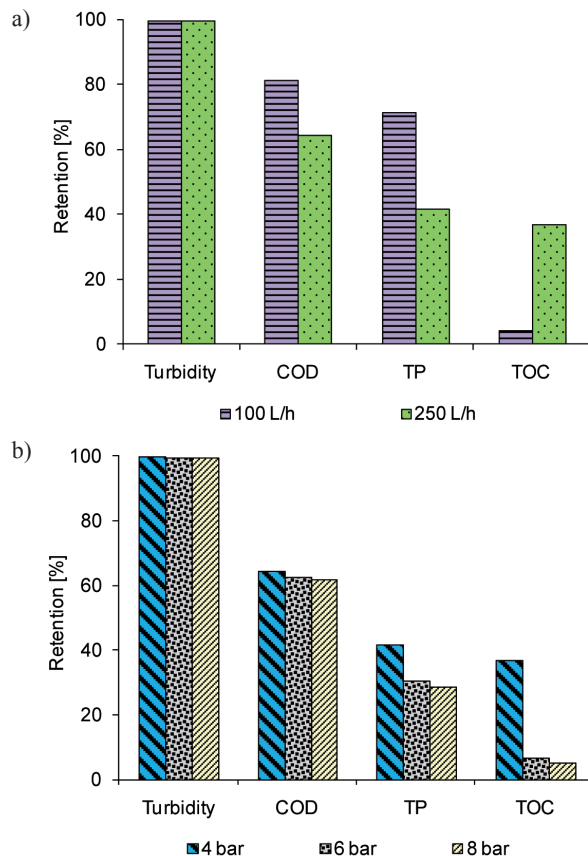
## 2.2. Regeneration of acidic cleaning baths using UF and UF/NF

The wastewaters generated during acidic cleaning of technological lines in food industry by using nitric acid (HNO<sub>3</sub>) were treated in ultrafiltration (UF). The influence of process parameters (TMP – transmembrane pressure and intensity of retentate flow – Q<sub>R</sub>) on regeneration efficiency was investigated. UF was performed on two different values of retentate flow (100 L/h and 250 L/h) and three different values of transmembrane pressure (4.0 bar, 6.0 bar, and 8.0 bar). It was found that the efficiency of acidic wastewater regeneration depends on the value of retentate flow – Q<sub>R</sub> (Table 4). The permeate flux (J<sub>p</sub>) was at a constant level during the volume reduction factor (VRF) of feed during the UF of acidic cleaning baths. Concerning the efficiency of the process, it is necessary to perform UF at the value of retentate flux (250 L/h) to obtain the best results. This value of retentate flow barely effects on the efficiency of UF and removal of compounds from filtration medium (Fig. 6a).

**Table 4. The effect of process parameters on an efficiency of regeneration of acidic cleaning baths during UF**

Transmembrane pressure (TMP) [bar]	Retentate flow (Q <sub>R</sub> ) [L/h]	Permeate flux (J <sub>p</sub> ) [L/(m <sup>2</sup> h)]
4.0	100	16.7
4.0	250	41.7
6.0	100	16.7
6.0	250	41.7
8.0	100	16.7
8.0	250	41.7

In turn, analyses of the level of the retention of compounds in the filtration medium show that using high transmembrane pressure resulted in a drop in the efficiency of wastewater regeneration (Fig. 6b).



**Fig. 6. Retention of acidic cleaning baths parameters after UF: (a) process performed at constant transmembrane pressure (TMP = 4.0 bar); (b) process performed at constant retentate flow (Q<sub>R</sub> = 250 L/h)**

Physical and chemical properties of regenerated wastewater contaminated with macromolecular organic compounds were shown in Table 5. Treatment of acidic wastewater with UF process was able to decrease turbidity (from 899 NTU to 2.6 NTU) and limited the reduction of TP (from 8 mg/L to 5 mg/L), COD (from

1186 mg/L to 429 mg/L), and TOC (from 482 mg/L to 138 mg/L). Integration of UF and NF provides more satisfying results (Table 5). Acidic wastewater treated with integrated UF/NF was able to remove of TP to 0.2 mg/L, COD to 3.5 mg/L, and TOC to 51 g/L.

**Table 5. Physical and chemical parameters values of acidic cleaning solution regenerated by UF\* and integrated UF/NF\*\* system**

Parameters	Feed	UF permeate	UF/NF permeate
Turbidity [NTU]	899	2.6	0.3
TP [mg/L]	8.0	5.3	0.2
COD [mg/L]	1186	429	3.5
TOC [mg/L]	482	138	51

\*  $TMP_{UF} = 4.0$  bar,  $Q_R = 250$  L/h; \*\* $TMP_{NF} = 8.0$  bar,  $Q_R = 250$  L/h

## Conclusions

The aim of the study was to evaluate the abilities of using membrane processes for effective regeneration of acidic and caustic wastewaters generated during washing technological lines in the dairy industry. Results of research based on model solutions showed that caustic cleaning solution can be treated using integration of MF/UF system, which decreased the concentration of TNb to 9.0 mg/L and COD concentration to 303 mg/L. In turn, to regenerate acidic cleaning solutions, it is necessary to use an integrated UF/NF system, which was able to reduce the concentration of TP to 0.2 mg/L, COD to 3.5 mg/L, and TOC to 51 g/L. The results obtained during the tests on model solutions are promising. Therefore, it is necessary to continue experiments on the possibilities of regeneration of real dairy wastewater resulting from the chemical cleaning of production lines. It will enable the development of an effective regeneration procedure for spent cleaning baths and contribute to reduction of chemicals and water consumption in dairy plants.

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