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Research Paper

# Influence of NF Membrane Properties on Water Recovery From the Dairy Industry Wastewater

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## Keywords

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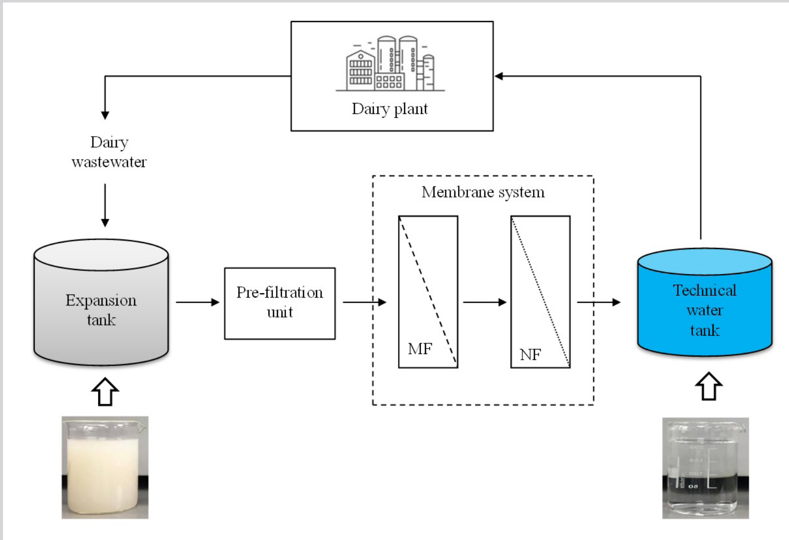
## Highlights

- NF membrane's type has an impact on the efficiency of water reclamation from the dairy wastewater.
- The PA layer of the NF membrane ensures high retention of organic compounds.
- Due to the PA layer of the NF membrane, the VFAs content decreased significantly.
- Due to the PA layer of the NF membrane, the susceptibility to fouling was lower.

## Graphical abstract




## Abstract

In the paper, the use of three types of polymer nanofiltration (NF) membranes, i.e. the TS80, DL, and NP010, to recover water from the dairy industry wastewater is described. The most desired results were obtained for the TS80 membrane with the skin layer made of polyamide. This membrane significantly contributed to the recovery of water to be reused for external cleaning of tank parts, road tankers, and floors. All tested NF membranes were characterized by a relatively low fouling index. This is due to the preliminary treatment of wastewater as part of an integrated system of bag filtration and microfiltration. However, the decrease in the permeate flux for all tested polymer membranes was observed during the NF process, which was mainly caused by an increase in the concentration factor of the dairy industry wastewater components. The presented results are part of the prospective trends in the development of the bioeconomy, especially in a closed circuit.



## 1. Introduction

The dairy industry is the main source of food processing waste and, at the same time, one of the largest raw material and water consumers in the food industry. It is estimated that 1 $dm^3$ of processed milk consumes 1.44 $dm^3$ of water, while the production of cheese, butter, or curd cheese is even more water-consuming (1.6-4.0 $dm^3$ of water per 1 $dm^3$ of milk), while the milk powder requires yet more water (15-20 $dm^3$ water per 1 $dm^3$ of milk) [1]. Approximately 80-90% of the used water becomes wastewater, which calls for economical water management [2]. The effective use of water resources in production processes requires water wastage prevention, i.e. the reduction of the industrial wastewater amount and the concurrent maximum possible recovery of reusable water.

Modern technologies of wastewater treatment that permit closed water circuits involve processes of membrane filtration, which have already found numerous applications in the dairy industry [3–7]. Microfiltration (MF) is widely used to remove microbes, separate and fractionate milk fats, and break down proteins during cheese and milk production [8, 9]. On the other hand, ultrafiltration (UF) is most commonly applied to concentrate whey protein from milk and whey [10] and to normalize milk for the production of cheese, yogurt, and other dairy products [4], and recently – to produce cheese using a membrane method [5]. In turn, nanofiltration (NF) and reverse osmosis (RO) processes have been widely used to desalinate and dehydrate whey [11–13]. Furthermore, an interesting area of membrane technology application concerns the regeneration of wastewater from dairy plant technological line cleaning, contaminated both by organic (proteins, lactose, and fats) and

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chemical cleaning substances (acids, bases, enzymes, and detergents) [14–16]. NF membrane is considered a convenient way to treat used alkaline and acidic cleaning solutions [3, 17–19], while the RO process – the dairy industry wastewater [6, 18, 21]. However, RO is limited by the high costs of the operating process pressure and the low permeate flux. NF is a significantly less energy-consuming and more efficient membrane process. Its application for the separation of the selected components from the dairy industry wastewater depends primarily on the selection of a membrane with appropriate physicochemical properties, out of which the surface charge plays the major role as far as an NF membrane's separation properties are concerned [22–24]. Polymer NF membranes used in typical water or municipal wastewater treatment processes usually show a negative surface charge. On the other hand, in the case of acidified wastewater treatment, the membrane is characterised by a positive surface charge or it is not polarised.

The aim of this study was to investigate the influence of the NF membrane's properties on the efficiency of water recovery from dairy industry wastewater.

## 2. Materials and methods

### 2.1. Filtration installations

The experiments were performed on the raw dairy industry wastewater generated during the cleaning of the technological line in the plant that produces milk, cream, kefir, buttermilk, and cottage cheese. The dairy plant is located in Mazovian Voivodeship in Poland and it produces approx. 400 $m^3$ of wastewater per day. The pollutant content was analyzed in the samples from raw and pre-treated wastewaters, permeate, and retentate from NF. In order to separate suspended solids, it was necessary to pre-treat the dairy industry 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) characterised by the pore size of 0.3 µm was carried out using the 'dead-end' laboratory scale set-up. The MF process was conducted at 0.5 bar and the feed flow rate of 0.02 $m^3$/h. The pure water flux of the MF filter was 222 $dm^3/(m^2h)$, whereas the average permeate flux during microfiltration of dairy wastewater was 216 $dm^3/(m^2h)$. Nanofiltration was performed in a batch mode using the 'cross-flow' laboratory scale set-up (Figure 1) under the transmembrane pressure of 14 bar and the cross flow velocity of 0.35 m/s. The value of the transmembrane pressure was selected based on the previous dairy product or dairy wastewater-based studies [25–28]. The permeate was collected in a separate container and the retentate stream was recycled to the feed container. For the NF process, 5 $dm^3$ of the feed pretreated as part of the process employing a bag filter and an MF membrane were used. The NF process continued until the permeate amount of 2.5 $dm^3$ was received. During the process, the temperature in the feed/retentate container was kept at the level of 25±1°C.

### 2.2. Membranes

The experiments employed three previously not used NF polymer membranes dedicated to protein-contaminated wastewater treatment, as stated by manufacturers [29], with good chemical and thermal resistance, surfaces characterised by negative zeta potential and hydrophilic properties, and the active area of 0.014 $m^2$ (Table 1).

### 2.3. Physicochemical analysis

Mixed wastewaters were obtained from one dairy plant. These wastewaters were pre-treated with a polypropylene bag filter and an MF membrane and subjected to nanofiltration. The samples from raw, pre-treated dairy industry wastewater, permeate, and retentate were analysed in terms of the total nitrogen, ammonium nitrogen, and phosphorous concentration, chemical oxygen demand (COD), biochemical oxygen demand (BOD), as well as sulfate and volatile fatty acid (VFAs) content, using HACH cuvette tests for the UV-VIS DR6000 spectrophotometer. The concentrations of calcium, chloride, and nitrate ions were determined using Mettler Toledo ion-selective electrodes. The pH and conductivity were measured using the Seven Multi Mettler Toledo device. Turbidity was determined using the HACH measurement device. The dry residue was evaluated with the weight method. The samples of 5 $cm^3$ were placed on disposable aluminum plates in the Radwag MAC 50/1 moisture analyzer. A standard drying profile was used (no mass change of 0.001 g within 60 s at 105°C).

**Table 1**
Characteristics of polymer membranes used for the dairy industry wastewater.

| | DL | TS80 | NP010 |
|---|---|---|---|
| **Manufacturer** | **GE Osmonics** | **TriSep** | **Microdyn Nadir** |
| Material of skin layer | PPZ | PA | PES |
| $MgSO_4$ retention,% | 98 | 99 | - |
| Cut-off, Da | 150-300 | ~150 | ~1000 |
| pH range | 2-11 | 2-11 | 0-14 |
| Process temperature, °C | <50 | <45 | <95 |
| Zeta potential, mV (pH ≈ 7, t = 25°C) | -22 [30] | -15 [30] | -9 [31] |
| Contact angle,° | 43.8±2.5 [32] | 46.9±0.9 [33] | 65.9±3.5 [31] |

PPZ – Polypiperazine-amide, PA – Polyamide, PES – Polyethersulfone

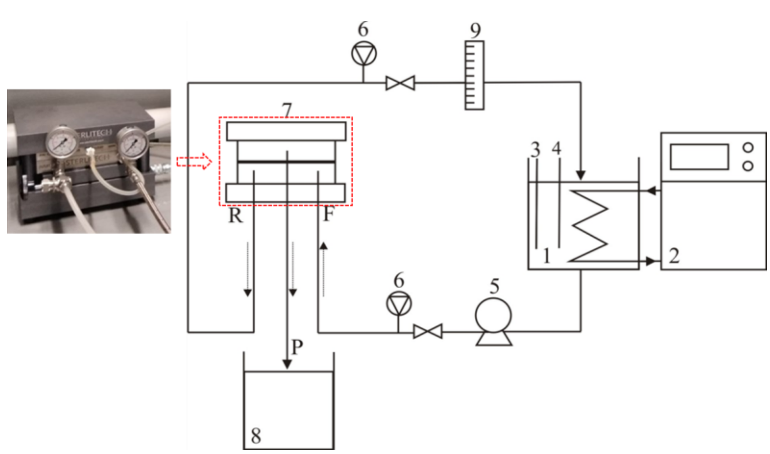


**Fig. 1.** Diagram of the installation used to treat the dairy industry wastewater: 1 – feed/retentate container, 2 – thermostat, 3 – thermometer, 4 – pH-meter or conductometer, 5 – high-pressure pump, 6 – manometer, 7 – flat-sheet membrane module, 8 – permeate container, 9 – flowmeter, P – permeate, F – feed, R – retentate.

### 2.4. Calculated parameters

The effectiveness of pollutant removal during the process of nanofiltration of the dairy industry wastewater was evaluated based on the percentage reduction of the pollutant content in a solution (Formula 1):

$$R = \left(1 - \frac{C_P}{C_F}\right) \cdot 100\% \quad (1)$$

where:

R – retention of component, %; $C_P$ – concentration of a component in the solution after the treatment, mg/dm$^3$; $C_F$ – concentration of a component in the solution before the treatment, mg/dm$^3$.

The efficiency of membrane processes of the dairy industry wastewater was determined with the permeate flux (Formula 2):

$$J_p = \frac{V_p}{A.t} \quad (2)$$

where:

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

The irreversible fouling index (IF) can be expressed as a percentage of the deionised water permeability decrease after the experiment (Formula 3):

$$IF = \left(\frac{L_P - L_F}{L_P}\right) \cdot 100\% \quad (3)$$

where:

$L_P$ - deionised water permeability of the new membrane, dm$^3$/(m$^2$·h·bar); $L_F$ - deionised water permeability of the membrane after NF of the dairy industry wastewater, dm$^3$/(m$^2$·h·bar).

The deionised water permeability ($L_{P/F}$) was calculated as follows (Formula 4):

$$L_{P/F} = \frac{J_P}{TMP} \quad (4)$$

where:

$J_P$ – permeate flux of deionised water, dm$^3$/(m$^2$·h); TMP – transmembrane pressure, bar.

## 3. Results and discussion

### 3.1. Preliminary treatment of dairy industry wastewater

In the first stage of the research, the composition of the dairy industry wastewater was examined. The physicochemical analysis of mixed dairy industry wastewaters (Table 2) showed that they contained organic substances in particular, including volatile fatty acids, as well as mono- and divalent ions (given the nature of the dairy plant production process and the characteristics of the cleaning agents used for technological line cleaning).

From the analysis of the composition of the dairy industry wastewaters (Table 2), it followed that the water regeneration process from this type of wastewater should be carried out as part of nanofiltration after preliminary treatment. The dairy industry wastewater was treated using a bag filter and an MF ceramic membrane with a pore diameter of 5 µm and 0.3 µm, respectively. The dairy industry wastewater needed to be pretreated to remove solid contaminants in the form of a suspension. This resulted mainly in the reduction of the dry residue (Figure 2). MF significantly reduced the organic matter (COD, BOD, and VFAs) and biogens, such as nitrogen and phosphorus compounds (Figure 2). The low ammonium nitrogen retention (3% only) resulted from the fact that lactic acid and ammonia (ammonium lactate) salts in the dairy industry wastewater can easily pass through an MF system. In turn, the turbidity of the dairy industry wastewater was reduced to 532 FNU during the preliminary treatment.

### 3.2. Nanofiltration of the dairy industry wastewater

In the next stage of work, the dairy industry wastewater pre-treated in the MF process was subjected to the NF process. From the research results, it followed that nanofiltration of the dairy industry wastewater enabled the reduction in the organic substance concentration (Figure 3). This process turned out to be the most effective in the case of the TS80 membrane, the use of which resulted in a nearly 94% reduction in the content of substances responsible for COD and BOD, an 85% reduction in the total nitrogen concentration, and a 98% reduction in the total phosphorus concentration. Particularly noteworthy is the fact that the TS80 membrane retained 88% of VFAs, which may prove that this membrane has properties similar to membranes used in RO processes [34,35]. As regards the DL membrane-based nanofiltration process, the dry residue removal efficiency and the total concentration of nitrogen, phosphorus, and VFAs were lower by about 9% compared to the TS80 membrane (Figure 3). The retention of components responsible for COD and BOD, and ammonium nitrogen was comparable to that obtained during the nanofiltration process employing the TS80 membrane. The NP010 membrane turned out to be the least effective in the case of nanofiltration of the dairy industry wastewater – about 88% and 82% retention of components responsible for BOD and COD respectively, and less than 80% as regards the other parameters. Such low efficiency of the NP010 membrane is likely to result from its less dense structure (cut-off ~1000 Da), which also manifested itself in the high permeate flux obtained during the nanofiltration of the dairy industry wastewater (Figure 4). Nevertheless, NF carried out using all three aforementioned types of membranes made it possible to reduce the turbidity to a level of less than 0.5 FNU.

The characteristic feature of nanofiltration membranes is the ability to selectively separate inorganic salts ions [21, 22, 31]. The retention results for mono- and divalent ions in the dairy industry wastewaters within the NF process employing the three selected types of membranes are presented in Figure 4. The use of the DL membrane resulted in higher permeability of monovalent ions ($Cl^-$ and $NO_3^-$) than in the case of the TS80 and P010 membranes, as this membrane, which is characterized by low zeta potential, allows for more effective transport of chlorides and nitrates into the permeate. As regards the retention of divalent ions ($Ca^{2+}$, $SO_4^{2-}$), this parameter is high for each membrane type used.

The analysis of the efficiency of the dairy industry wastewater nanofiltration showed a significant decrease in the permeate flux at the time of the processes carried out (Figure 5). The treatment of the dairy industry wastewater during NF resulted in the permeate flux decrease by about 70% for each membrane tested. This is due to the fact that during the batch process, as the permeate flux drained over time, the concentration coefficient increased, causing the permeation rate to drop. Especially in the first phase of the process, the permeation rate decreased rapidly (Figure 5), as the membrane concentration polarisation layer was formed by the dairy industry wastewater components [26, 36–39]. According to Rice et al. [37], the deposition of proteins on the membrane surface and the formation of a gel/cake layer create high hydraulic resistance. In turn, according to Tang et al. [38], a gel/cake layer enhances concentration polarisation, which drastically increases the osmotic pressure on the membrane surface. Moreover, the adsorption of proteins on the surface of membranes strengthens the presence of polyvalent salts [39].

**Table 2**
Physicochemical properties of the dairy industry wastewater used in the experiments.

| Parameter | Unit | Value |
|---|---|---|
| pH | - | 7.9 |
| Turbidity | FNU | 1696 |
| Conductivity | µS/cm | 1553 |
| Dry residue | mg/dm$^3$ | 4720 |
| Chemical oxygen demand (COD) | mg/dm$^3$ | 7080 |
| Biochemical oxygen demand (BOD) | mg/dm$^3$ | 3500 |
| Total nitrogen | mg/dm$^3$ | 134 |
| Ammonium nitrogen | mg/dm$^3$ | 3.2 |
| Total phosphorous | mg/dm$^3$ | 22.5 |
| Volatile fatty acids (VFAs) | mg/dm$^3$ | 595 |
| Calcium | mg/dm$^3$ | 70.9 |
| Sulphates | mg/dm$^3$ | 264 |
| Nitrates | mg/dm$^3$ | 157 |
| Chlorides | mg/dm$^3$ | 65.7 |

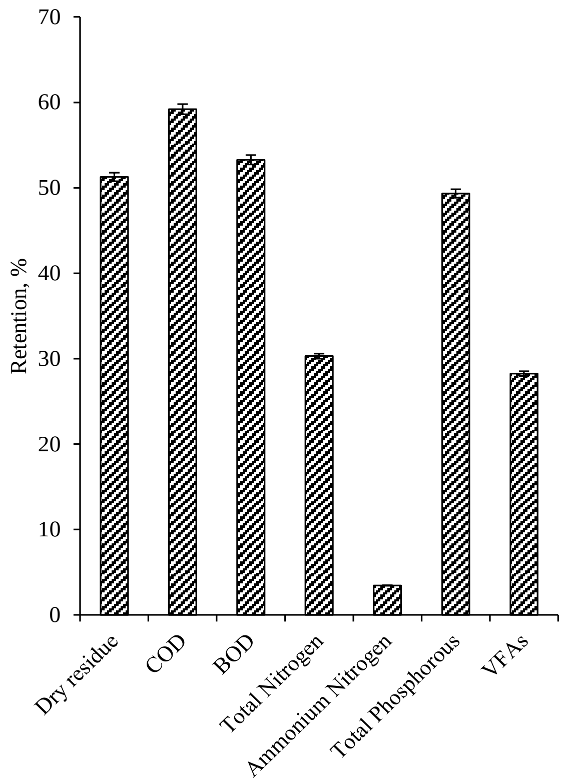


**Fig. 2.** Retention of the dairy industry wastewater components during pre-treatment using the bag filter and microfiltration system.

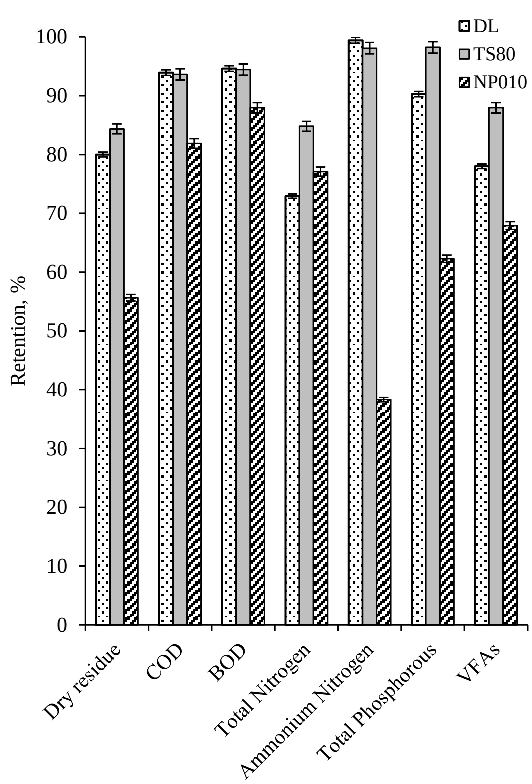


**Fig. 3.** Retention of the dairy industry wastewater components during nanofiltration using DL, TS80, and NP010 membranes.

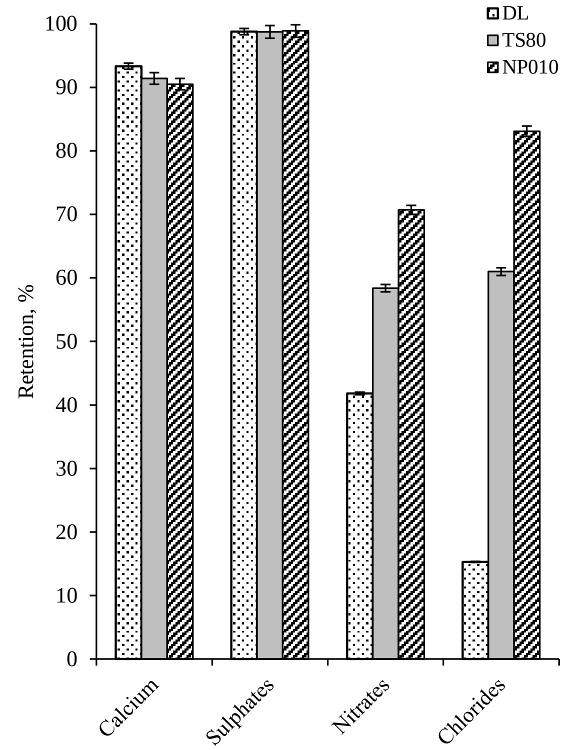


**Fig. 4.** Retention of the dairy industry wastewater ions during nanofiltration using DL, TS80, and NP010 membranes.

### *3.3. Fouling of NF membranes by dairy microfiltration permeates*

In the next stage of work, the susceptibility of nanofiltration membranes to blockage caused by the deposition of the dairy industry wastewater components was analyzed based on the results of the tests carried out (Figure 6). It was found that all the NF membranes used were characterised by a relatively low fouling index (FI) as a result of the preliminary preparation of the dairy industry wastewater in a system involving bag filtration and microfiltration processes. The TS80 membrane was characterised by the lowest FI – 6% only. On the other hand, as regards the DL and NP010 membranes, the fouling rates were two and three times higher than in the case of the TS80 membrane.

Differences in susceptibility of the applied NF membranes to the deposition of the dairy industry wastewater components as a result of the 4-hour treatment process (Figure 6) may stem from the different types of their active layer polymers. The NP010 membrane is made of polyethersulfone and it is characterized by a higher contact angle (65.9±3.5°) than the DL and TS80 membranes, the skin layers of which are made of polyamide (43.8 ± 2.5°) and polypiperazine amide (46.9±0.9°). According to Mantarri et al. [40], it is due to the fact that the dissociation of carboxyl and amino groups on the surface of membranes increases hydrophilicity. In turn, according to Bildyukevich et al. [24], there is no direct relationship between the wettability of the surface of polymer UF membranes and the tendency to fouling caused by the adsorption of milk proteins. Nevertheless, this work proved that membranes with a higher contact angle are more prone to fouling. The results obtained for the NF membranes tested overlap with the data reported by Chen et al. [23]. Moreover, the NP010 membrane has a higher separation limit (~1000 Da) than the DL (150-300 Da) and TS80 (~150 Da) membranes. This can result in the deposition of components of the dairy effluent in the internal structure of the membrane, which further reduces the permeate flux and is often difficult to remove.

## 4. Conclusions

The results of the research allowed for the comparison of the efficiency of water recovery from the dairy industry wastewater in nanofiltration processes employing three different membrane types. The best results were obtained for the TS80 membrane, the application of which enabled the removal of organic substances from the dairy industry wastewater, and also contributed to a significant reduction in the concentration of volatile fatty acids and monovalent ions. Based on the analysis of the obtained results, it was proposed that the water reclaimed from the dairy industry wastewater in the above-mentioned manner should be reused for external cleaning of tanks, road tankers, and floors. For all the examined membranes, a decrease in the permeate flux was observed during the process, which was mainly caused by an increase in the concentration factor of the dairy industry wastewater components. As a result of the preliminary wastewater treatment, the decrease in the permeate flux during the nanofiltration process, as caused by the fouling effect, was insignificant.

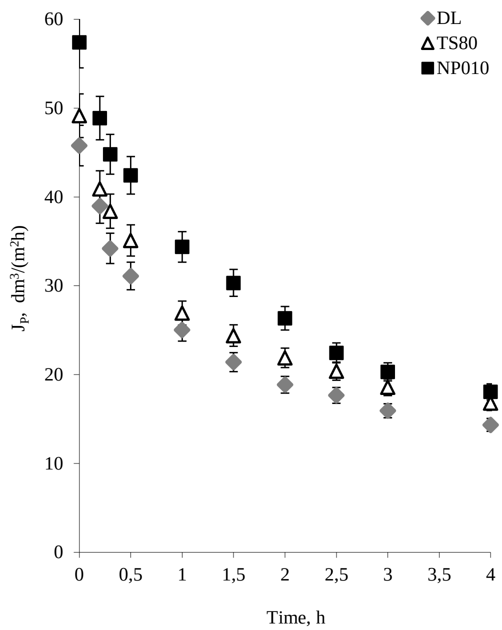


**Fig. 5.** Declining the permeate flux ($J_P$) during nanofiltration of the dairy industry wastewater using DL, TS80 and NP010 membranes.

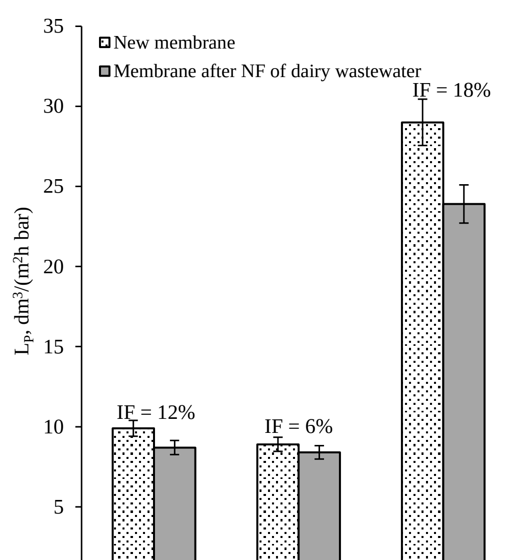


**Fig. 6.** Demineralised water permeability coefficients through DL, TS80, and P010 membranes determined before and after nanofiltration of the dairy industry wastewater.

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