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# THE POSSIBILITIES OF USING MEMBRANE FILTRATION IN THE DAIRY INDUSTRY

**Key words:** membrane filtration, dairy industry, wastewater treatment, ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO).

**Abstract:** The possibilities of using membrane filtration in the dairy industry are presented in this paper. It was found that the pressure membrane processes are successfully used for the processing of raw milk into milk products. However, based on the analysis of the technology for producing chosen milk products, it was found that the dairies are wastewater in the form of white water, milk water, and spent cleaning baths. It was proposed to use two-step systems based on membrane filtration to treat these types of wastewater. Regeneration of white water and milk water is possible in RO/ROP system. In turn, UF/RO or NF/RO systems are necessary for the regenerations of spent cleaning baths. However, based on studies of the compositions of dairy wastewaters and preliminary filtration studies, it was claimed that, before the treatment of this type of wastewater by membrane modules, it is important to appropriately prepare wastewater with pre-treatment techniques.

### Możliwości wykorzystania filtracji membranowej w przemyśle mleczarskim

**Słowa kluczowe:** filtracja membranowa, przemysł mleczarski, oczyszczanie ścieków, ultrafiltracja (UF), nanofiltracja (NF), odwrócona osmoza (RO).

**Streszczenie:** W pracy przedstawiono możliwości wykorzystania technik filtracji membranowej w przemyśle mleczarskim. Stwierdzono, że ciśnieniowe procesy membranowe są z powodzeniem stosowane do przetwarzania mleka surowego na gotowe produkty mleczne. Jednak na podstawie przeprowadzonej analizy technologii wytwarzania wybranych produktów mlecznych stwierdzono, że w zakładach mleczarskich powstają ścieki w postaci białej wody, wody z mleka i zużytych kąpieli myjących. Do unieszkodliwiania tego typu ścieków zaproponowano wykorzystanie dwustopniowych układów opartych na technikach filtracji membranowej. Regeneracja białej wody i wody z mleka możliwa jest w układzie RO/ROP. Z kolei do regeneracji zużytych kąpieli myjących niezbędne jest zastosowanie układów UF/RO lub NF/RO. Jednak na podstawie przeprowadzonych badań fizykochemicznych ścieków mleczarskich oraz wstępnych badań filtracyjnych stwierdzono, że przed podaniem tego typu ścieków na moduł membranowy niezbędne jest odpowiednie ich przygotowanie.

## Introduction

The food industry is one of the most important and fastest growing sectors of the economy in Poland. The proportion of this sector in the total sales value of Polish industry is approx. 24%, and it is one of the highest in Europe. The total value of the market of meat and milk and their products is approx. 40% of the market of food products [1]. It is connected with a high demand on utilities, especially water. In the year of 2009, industrial processing plants in Poland used 585.6 $hm^3$ of water for production, and 12.3% was used by the food businesses. In the year of 2012, the contribution of the food industry in the total emission of wastewater from processing industry was 11.4% [1, 2].

The largest sectors of the food industry in Poland both in terms of the overall weight of the processed raw materials and the consumption of water is the dairy industry, which is also considered to be the largest source of wastewater from food processing [2]. This industry generates considerable organic waste streams, and the longer forecasts assume that global milk production will grow by 2.1% per year [3]. Due to the seasonal nature of food processing and a large range of products, it is difficult to predict the composition of wastewaters and wastes. The consumption of water is dependent on the type of production and varies in a wide range of 0.5–11 $dm^3$ water to 1 $dm^3$ of the milk [4]. It has been estimated that 1.44 $dm^3$ of water is consumed in the processing of 1 $dm^3$ of raw milk on the drinking milk. During the production of cheese, 1.6–2.0 $dm^3$ water to 1 $dm^3$ of processed milk is consumed. However, in the case of butter (from 1 $dm^3$ milk), it is approx. 3 $dm^3$ water, cottage cheese – approx. 4 $dm^3$ and milk powder – 15–20 $dm^3$. Moreover, about 80–90% of water used by dairies becomes wastewater [2].

The sustainable management of water resources requires the use of modern wastewater treatment technology in industrial plants, including the processing of milk, and building closed water systems, which now become the basis for sustainable development [5]. The modern techniques of wastewater treatment are membrane filtration processes, which also have many applications in the dairy industry [6]. The dynamic development of new membrane materials and design modules extends the practical use of filtration processes in extreme conditions, such as high temperature, acidity, or alkalinity, including a significant burden of organic substances. The advantage of membrane filtration also makes it possible to achieve high mass transfer surface area with a relatively small installation dimensions. This is especially important during the integration of membrane filtration installations with process lines. In addition, the possibility of modular membrane installation configuration, which can be compiled as needed, is making the membrane filtration technique particularly useful in dairies. Besides the economic and environmental benefits, they are also easily adapted, which enables the use of membrane filtration in integrated technologies of water regeneration, which contain physical, physicochemical and biotechnological operations of their pretreatment, purification, or final cleaning.

## 1. The membrane filtration used to treat raw milk

Membrane processes used in the dairy industry includes mainly microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), and their detailed characteristics are presented in the works [7, 8].

The application of low-pressure membrane processes, i.e. micro- and ultrafiltration, is particularly widespread in the dairy industry. During microfiltration (MF), there are separated particles having a diameter of 0.1–10 µm under a maximum pressure of 0.2 MPa [7]. This process allows the retention of fat, bacteria, spores, and somatic cells from the milk and whey [8, 9–12]. Microfiltration is mainly used for the removal of bacteria during the production of extended shelf life (ESL) milk (Fig. 1). This process is also used for the manufacturing of products with specific nutritional purposes, i.e. whey protein isolate and whey protein concentrates (Fig. 2 and 3). In addition, microfiltration membranes allow the fractionation of milk proteins. The use of microfiltration membranes with a pore diameter of 0.1 to 0.2 µm enables the separation of casein micelles from serum proteins [9].

The pore diameter of the ultrafiltration membranes is 0.001–0.1 µm. This process is carried out at a pressure in the range of 0.1–1.0 MPa [7]. This type of membrane, depending on the size of pores, allows the penetration of the particles characterized by low molecular weight, such as water, minerals, lactose, organic acids, and peptides [9–12]. The high molecular weight components, such as microorganisms, fatty balls, milk proteins, micellar casein, whey protein, and minerals are associated with the accumulation in the retentate after ultrafiltration [13, 14].

In the dairy industry, ultrafiltration is primarily used in the production of lactose-free milk (Fig. 2) and white cheese (Fig. 4). Furthermore, this process is used to concentrate the whey proteins in milk (Fig. 2) and to normalize the milk for the production of cheese and products with specific nutritional purposes, i.e. whey protein concentrate and whey protein isolate (Fig. 3). The whey protein concentrate (WPC) is a concentrated protein solution (80%) received during ultrafiltration process. In turn, in order to produce a whey protein isolate (WPI), it is necessary to separate lactose and sugar from the whey protein concentrate in the microfiltration process (Fig. 3).

The high-pressure membrane processes, i.e. nanofiltration and reverse osmosis, are used to desalting and dewatering of whey, which is a by-product in the making of cheese [14–17].

Membranes used in nanofiltration have a pore diameter up to 2 nm, and this process is carried out at the pressure of 0.5–3.0 MPa [7]. In the dairy industry, nanofiltration is used to concentrate and desalinate the whey (Fig. 1–4). It is due to the specific properties of nanofiltration membranes [14, 15]. Moreover, the ions of mineral salts and low-molecular organic compounds (e.g., lactic acid) pass through the NF membranes. Therefore, nanofiltration is also used for concentrating the acid whey [17–19].

Reverse osmosis (RO) is carried out under a pressure in the range of 1–10 MPa. During this process, mainly water with small amounts of mineral salts and compounds of non-protein nitrogen pass through the membrane [18, 19]. The RO process is mainly used to concentrate both whey and milk (Fig. 1–4).

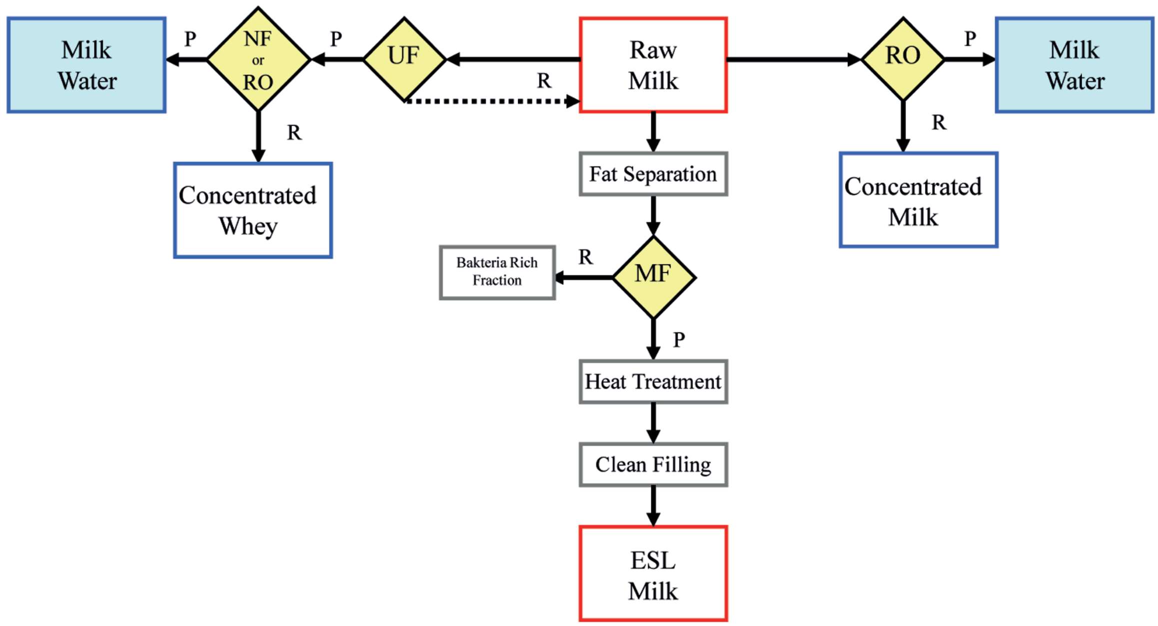


**Fig. 1. Technological scheme of extended shelf life (ESL) milk production: MF – microfiltration, UF – ultrafiltration, NF – nanofiltration, RO – reverse osmosis, P – permeate, R – retentate**

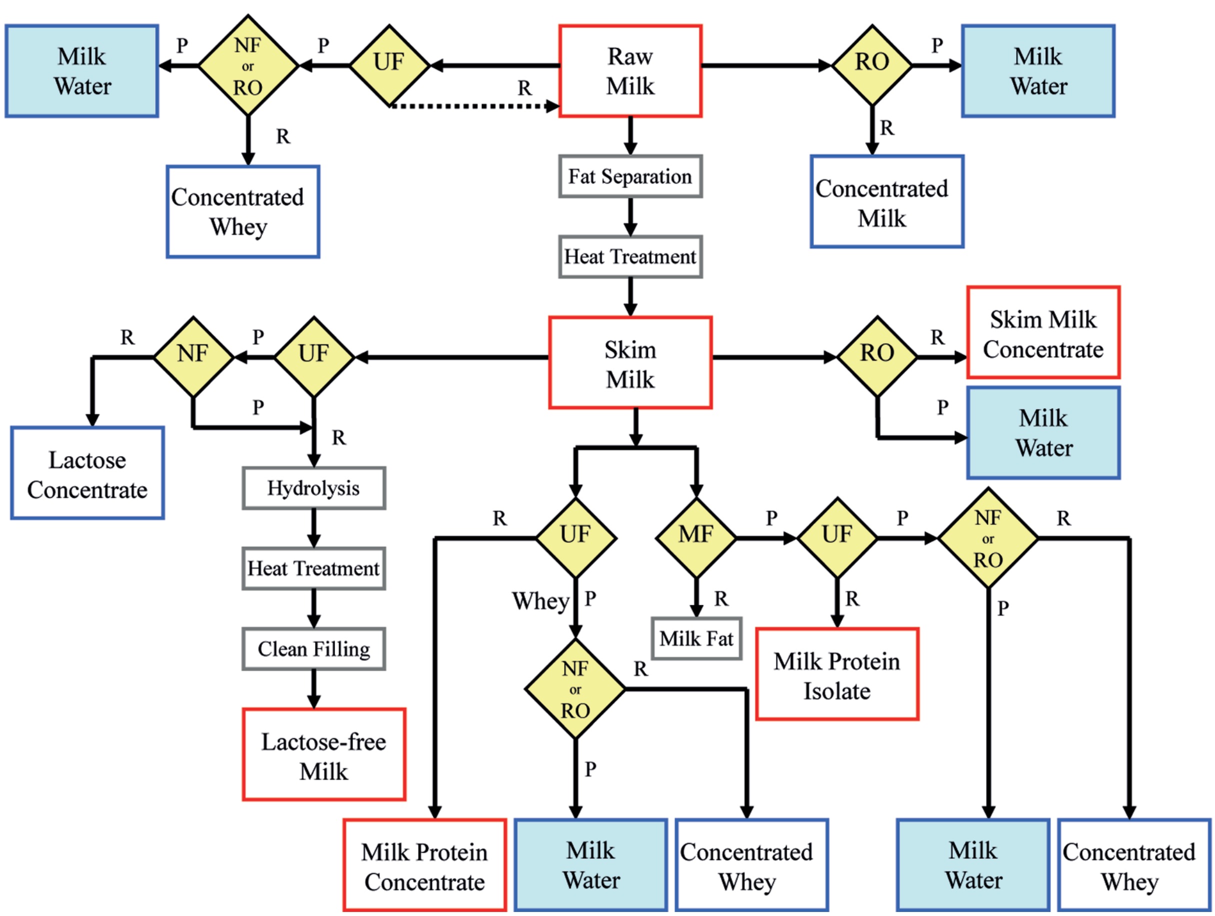


**Fig. 2. Technological scheme of skim milk treatment and the production of products with specific nutritional purposes: MF – microfiltration, UF – ultrafiltration, NF – nanofiltration, RO – reverse osmosis, P – permeate, R – retentate**

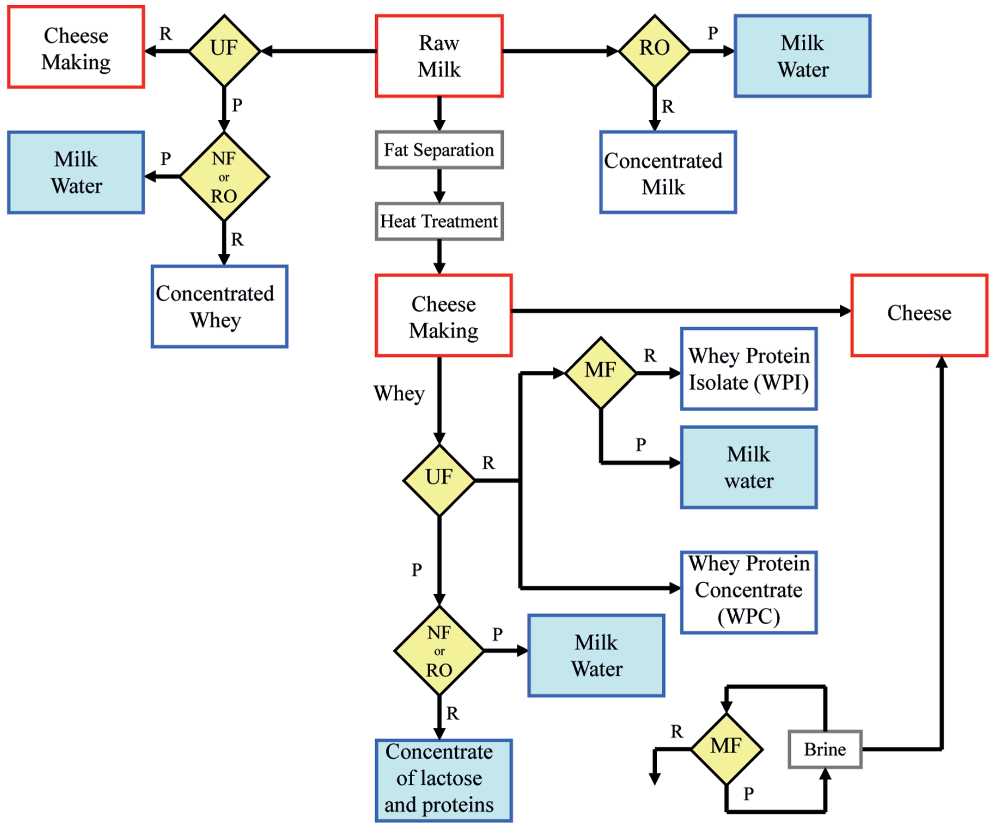


Fig. 3. **Technological scheme of cheese making and the production of products with specific nutritional purposes: MF – microfiltration, UF – ultrafiltration, NF – nanofiltration, RO – reverse osmosis, P – permeate, R – retentate**

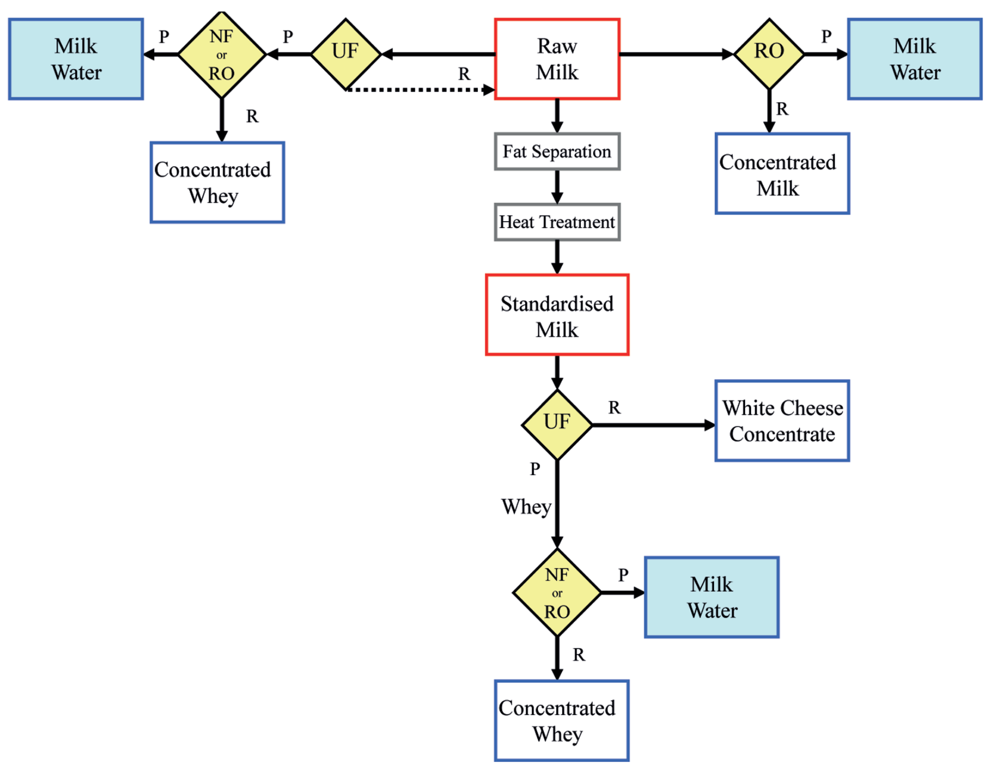


Fig. 4. **Technological scheme of white cheese production: UF – ultrafiltration, NF – nanofiltration, RO – reverse osmosis, P – permeate, R – retentate**

If the whey is to be used for further processing or as animal feed, then it should be concentrated by the process of reverse osmosis [20]. On the other hand, if the whey is concentrated in nanofiltration, the retentate is also demineralized and thus can be used for the production of bioenergy [8, 17, 20].

Currently, besides ceramic membranes from titanium dioxides ($TiO_2$) or zirconium dioxides ($ZrO_2$) for the processing of raw milk into milk products, polymer membranes made of polyethersulphone (PES), polyamide (PA), polyvinylidene fluoride (PVDF), poly(piperazine-amide) (PPZ), and polysulphone (PSU) are used (Table 1).

**Table 1. Polymer membranes used in the dairy industry [21, 22]**

| Manufacturer | Symbol | Process | Polymer | MWCO/Pore size | pH | Retention, % | Temperature,°C |
|---|---|---|---|---|---|---|---|
| GE Osmonics | DL | NF | PPZ | 150-300 Da | 3-9[1], 2-10[2] | 98[a] | 50 |
| TriSep | TM10 | MF | PVDF | 0.2 µm | 1-12 | - | 45 |
| | UF5 | UF | PES | 5 000 Da | 1-12 | - | 50 |
| | TS40 | NF | PPZ | ~200 Da | 2-11 | 90[a], 40-60[b] | 45 |
| | TS80 | NF | PA | ~150 Da | 2-11 | 99[a], 80-90[b] | 45 |
| | XN45 | NF | PPZ | ~500 Da | 2-11 | 95[a], 10-30[b] | 45 |
| Synder | FR | MF | PVDF | 800 000 Da | 3-9[1], 2-11[2] | - | 55[1], 50[2] |
| | V0.1 | MF | PVDF | 0.1 µm | 3-9[1], 2-11[2] | - | 55[1], 50[2] |
| | V0.2 | MF | PVDF | 0.2 µm | 3-9[1], 2-11[2] | - | 55[1], 50[2] |
| | BN | UF | PVDF | 50 000 Da | 3-10[1], 2-11[2] | - | 60[1], 85[2] |
| | MK | UF | PES | 30 000 Da | 3-9[1], 2-11[2] | - | 55[1], 50[2] |
| | ST | UF | PES | 10 000 Da | 3-9[1], 2-11[2] | - | 55[1], 50[2] |
| | NFX | NF | PA | 150-300 Da | 3-10[1], 2-11[2] | 99[a], 40[b], 99[d] | 50[1,2] |
| | NDX | NF | PA | ~800-1 000 Da | 3-10.5 | 90[a], 30[b] | 50[1], 40[2] |
| | NFG | NF | PA | ~600-800 Da | 4-10 | 50[a], 10[b] | 50[1,2] |
| | NFW | NF | PA | 300-500 Da | 4-9[1], 3-10[2] | 97[a], 20[b], 98[d] | 50[1], 40[2] |
| Nanostone™ | PV650 | MF | PVDF | 0.31 µm | 2-10[1], 2-11.5[2] | - | 60[1], 50[2] |
| | PE5 | UF | PES | 6 000 Da | 2-10 | - | - |
| | PE10HR | UF | PES | 10 000 Da | 2-10 | - | - |
| Microdyn Nadir™ | P010 | NF | PES | - | 0-14 | 35-75[c] | 95 |
| | P030 | NF | PES | - | 0-14 | 80-95[c] | 95 |
| Dow Filmtec | NF | NF | PA | ~200-400 Da | 2-11 | 99[a] | 45[1,2] |
| Koch Membrane System | Dairy-Pro™MF-0.1 | MF | PES | 0.1 µm | 2-10[1], 2-11[2] | - | 50 |
| | Dairy-Pro™UF-5K | UF | PES | 5 000 Da | 2-10[1], 2-11[2] | - | 55[1], 50[2] |
| | Dairy-Pro™UF-10K | UF | PES | 10 000 Da | 2-10[1], 2-11[2] | - | 55[1], 50[2] |
| | Dairy-Pro™MPS-34 | NF | PSU | ~200 Da | 0-14[1,2] | 95[b] | 50 |
| | Dairy-Pro™MPS-36 | NF | - | 1 000 Da | 1-13[1,2] | 10[a] | 50 |
| | Dairy-Pro™NF-200 | NF | PA | ~200 Da | 4-10[1], 2-11[2] | - | 50[1], 60[2] |
| | Dairy-Pro™RO | RO | PA | - | 4-10[1], 2-11[2] | - | 50[1], 60[2] |

[1]process, [2]CIP (Clean In Place), [a]$MgSO_4$, [b]NaCl, [c]$Na_2SO_4$, [d]lactose

The advantage of polymer membranes are lower cost and lower energy requirements compared to ceramic membranes. On the other hand, ceramic membranes are more resistant to extreme chemical and physical conditions than polymer membranes. The ceramic membranes may work in the pH range from 0 to 14 and at temperatures to 150°C. However, in the case of ceramic membranes, it is very important to make the temperature change in a stepwise manner (<10°C per minute) to avoid fracturing the membranes.

## 2. Wastewater generated in dairies

The analysing of individual products technologies (Fig. 1–4) and the functioning of the dairies found that these type of plants generate wastewater in the following forms:

- Milk water, which is formed during the concentration and demineralization of whey in nanofiltration or reverse osmosis;
- White water, which is formed during the first cleaning of line production; and,
- Spent cleaning baths, which are produced during the cleaning of technological lines by acidic, alkaline, or detergents solutions using CIP (Clean In Place) station [23–27].

The main components of white water and milk water are organic substances composed of lactose, fat, and protein (Fig. 5).

In turn, spent cleaning baths contain organic matter washed out from the technological lines and chemical substances (acids, alkalis, and detergents). According to the reference document of the European Commission on the best available techniques in the food industry [24], sodium hydroxide (NaOH) and nitric acid ($HNO_3$) are mainly used in dairies. The consumption of these chemicals are dependent on the technology used (Table 2).

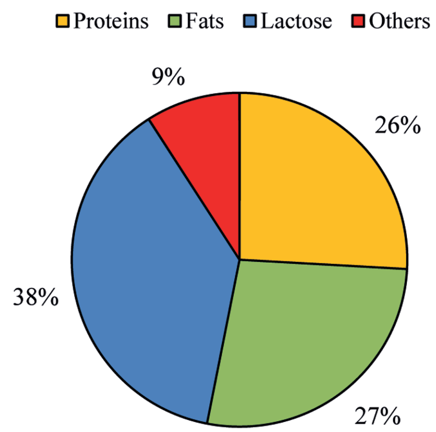


**Fig. 5. The percentage of the individual components of white water [27]**

**Table 2. Consumption of chemicals during the cleaning of technological lines after treatment or production of 1 m³ product [24]**

| Type technology | The consumption of the chemicals, kg | |
|---|---|---|
| | NaOH | $HNO_3$ |
| Treatment of milk | 0.2–0.9 | 0.1–1.0 |
| Production of milk and milk drinks | 0.4–5.4 | 0.6–3.8 |

On the other hand, phosphoric acid ($H_3PO_4$), sulphuric acid ($H_2SO_4$) and hydrochloric acid (HCl), as well as potassium hydroxide (KOH), and sodium hypochlorite (NaClO) are used to clean the process lines used for the treatment of whey. Moreover, the chelating agents, e.g., ethylenediaminetetraacetic acid (EDTA) are added to clean baths and water to prevent precipitation and the deposition of calcium and magnesium, and they also support the effect of bactericides [24]. The modern baths used for cleaning and disinfecting after neutralization are readily biodegradable, and wastewater from the cleaning of equipment and technological lines are connected to one another and biologically utilized [23, 26, 28]. However, due to the policy of the European Union, action is needed to help businesses transition to a more competitive and stronger economy that is based on a closed circuit in which water is used in a sustainable way. Therefore, the development of technological solutions of water recovery from the dairy industry is one of the key tasks of engineers. It enables the use of a membrane filtration for these purposes. The undoubted advantage of membrane filtration is the ability to achieve a high-mass transfer surface area with a relatively small size of installations [29]. This advantage and the possibilities of the construction of the membrane installations in the form of modules make membrane filtration attractive for the recovery and re-use of water.

## 3. Regeneration of dairy wastewater by membrane filtration

The state of the art [11, 13–18, 30–37] allowed the presentation of membrane filtration systems (Fig. 6 and 7) to recover of water from white and milk water and the regeneration of spent cleaning baths generated in dairies.

The recovery of water from the white and milk water is possible with the use of a RO/ROP system (Fig. 6). Regenerated water (permeate after the membrane filtration) will be able to be used for cleaning installations, tanks, and cisterns. If permeate after membrane filtration is disinfected by UV lamps or sodium hypochlorite (NaClO) in order to eliminate potential biological contamination, it might be possibly used as a process water [12].

The regeneration of acid or alkaline cleaning baths can be potentially carried out during ultrafiltration or nanofiltration (Fig. 7). The choice of the process depends on the composition of the spent cleaning baths. If the cleaning baths predominantly contains proteins and fats, it is possible to regenerate them by ultrafiltration. However, if the cleaning baths contain lactose, it is necessary to use nanofiltration processes [17, 30, 36–38]. The use of ultrafiltration or nanofiltration enables the regeneration of cleaning baths (permeate after the membrane filtration), which can be re-used for the cleaning of membrane plants and production lines. Furthermore, the permeate after ultrafiltration or nanofiltration can be subjected to reverse osmosis in order to obtain water which, after elimination of possible biological contamination by UV lamps or sodium hypochlorite (NaClO), will be re-used as a process water [12].

In some dairies, white water, milk water, and spent cleaning baths are mixed together and these averaged wastewaters are treatment biologically. However, the sustainable management of water resources requires the use of modern wastewater treatment technology for the creation of closed water circuits in dairies. It is necessary to assign the characteristics of averaged dairy wastewater to the development of water regeneration system. The results of physicochemical analyses of a few samples of averaged wastewater generated by a dairy plant are presented in Table 3. It was found that this type of wastewaters have different compositions indicating a heavy load of organic substances. Therefore, it is impossible to give general characteristics of dairy wastewater.

The differences in the values of the determined parameters in wastewater collected at different times from single dairy plant are several orders of magnitude. It is evident, especially in the case of chemical oxygen demand (COD), ammonia nitrogen and turbidity (Table 3). Therefore, it is essential to adequately prepare

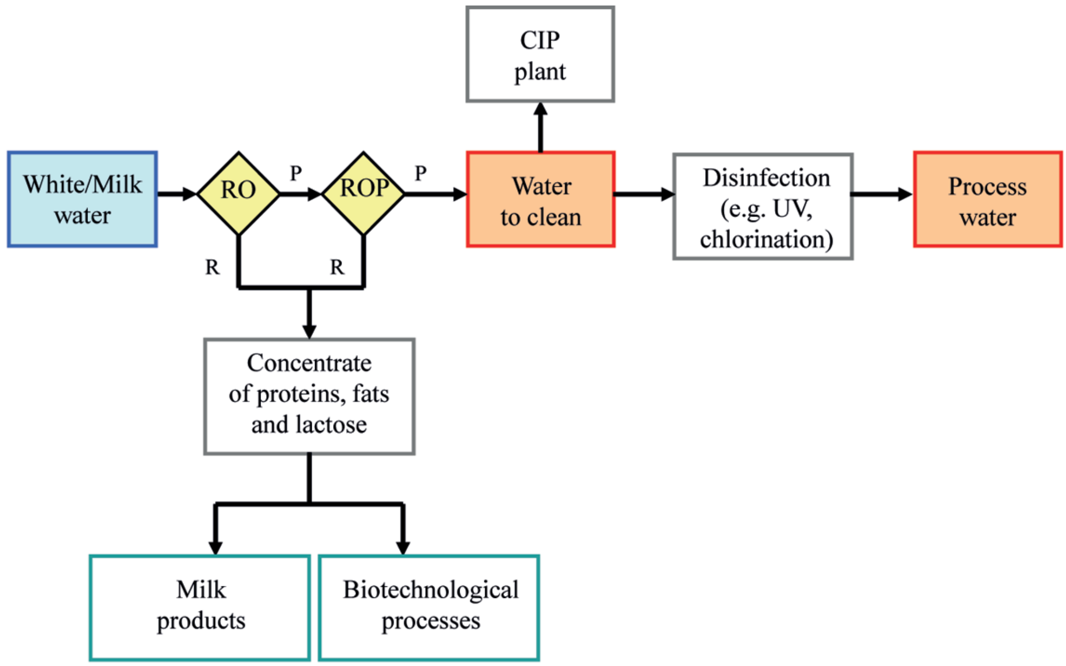


**Fig. 6. The concept of using the RO/ROP system for the recovery of water from white water and milk water: RO – reverse osmosis, ROP – polisher reverse osmosis, P – permeate R – retentate**

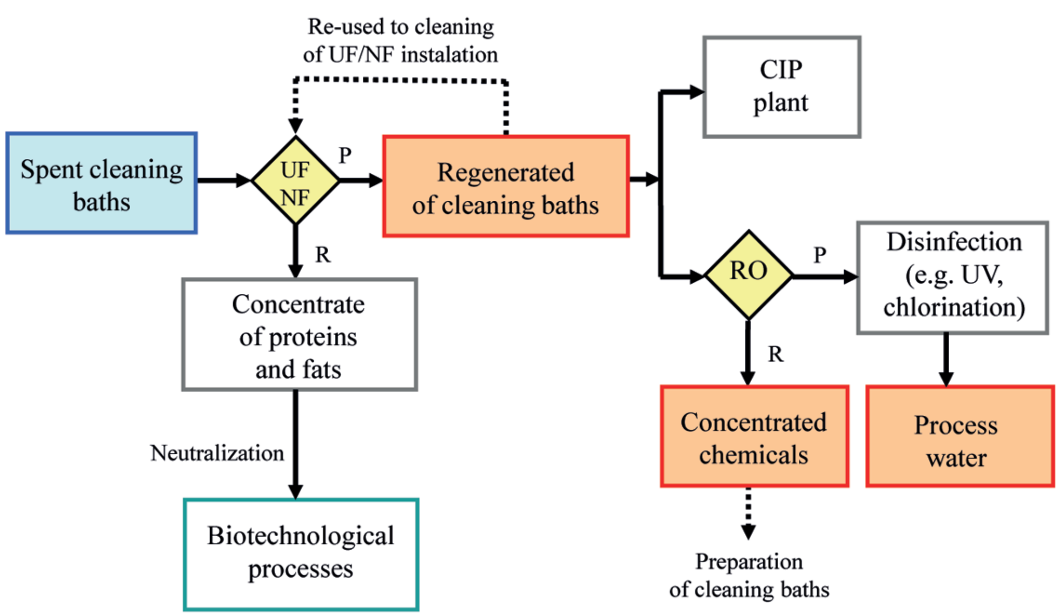


**Fig. 7. The concept of using the UF/RO or NF/RO systems for the regeneration of spent cleaning baths: UF – ultrafiltration, NF – nanofiltration, RO – reverse osmosis, P – permeate, R – retentate**

the dairy wastewater before treatment with membrane modules. Otherwise, a significant reduction in the efficiency of the process in time has been observed (Fig. 8). The initial high flux decreases rapidly while the cake layer accumulate, becoming more and more compacted starting from the bottom (stage I). As filtration proceeds, cake layer growth causes higher filtration resistance, which in turn causes lower flux (stage II). When the cake layer grows enough the membrane permeability maintains at the constant level (stage III). Thus, the wastewater should be pre-filtered [39–42]. However, due to the differences in the values of designated physical and chemical parameters, it is extremely important to study the properties of multiple dairy wastewaters before deciding on their pre-treatment technology.

**Table 3. The composition of averaged wastewater generated during the cleaning of a process line in a dairy plant – author's research results**

| Parameter | Unit | Content |
|---|---|---|
| pH | – | 7.0–8.5 |
| Turbidity | NTU | 900–2144 |
| Conductivity | µS/cm | 1410–1880 |
| COD | mg/dm³ | 1903–7510 |
| Total nitrogen | mg/dm³ | 80.6–141.0 |
| Ammonium nitrogen | mg/dm³ | 1.1–6.8 |
| Total phosphorus | mg/dm³ | 19.0–23.1 |
| Total solids | mg/dm³ | 2580–4840 |

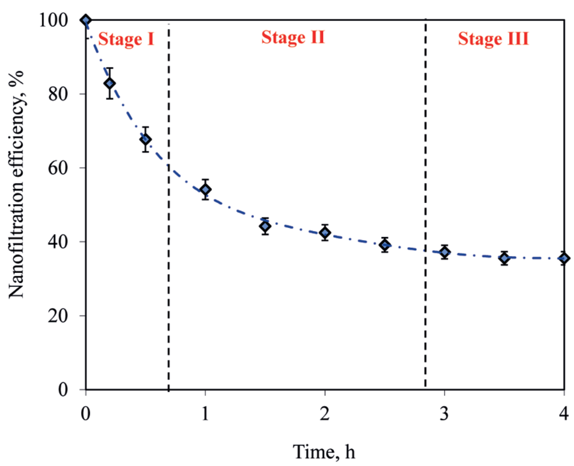


**Fig. 8. The reduction of efficiency during the nanofiltration of averaged wastewater generated during the cleaning of the process line in a dairy plant (composition of dairy wastewater: pH 7.0; turbidity 1554 FNU; conductivity 1668 µS/cm; COD 3750 mg/dm³; total nitrogen 98.9 mg/dm³; ammonium nitrogen 7.9 mg/dm³; total phosphorus 15.5 mg/dm³; total solids 3500 mg/dm³; polymer membrane; ΔP = 14 bar; t = 25±1°C) – author's research results**

The problem with treatment of this type of wastewater can also be the presence of volatile organic compounds [23], which penetrate even through a reverse osmosis membrane [8, 36]. Then, it is necessary to apply the filtration through the active carbon, pervaporation, or advanced oxidation. The correct choice of treatment technology for the final regenerated water or cleaning baths requires laboratory tests. The use of pressure membrane processes for the treatment of dairy wastewater (Fig. 6 and 7) causes a concentration of impurities retained by the membrane. Therefore, it is necessary to develop a disposal method for these types of wastes.

The state of the art [37–39] allows one to propose that the concentrate from the membrane system (Fig. 6 and 7), characterized by high values of chemical oxygen demand (COD), biochemical oxygen demand (BOD), protein, fat, lactose, and nutrient, are used for the production of milk used as animal feed or neutralized by means of biotechnological processes. The biotechnological processes such as cultures of algae [43–48] and microorganisms [28, 49, 50] can be used to dispose of the concentrates from the membrane filtration process (Fig. 6 and 7). The particular advantage of using these types of biotechnological processes for the disposal of the contaminants retained by the membrane may be that the selection of appropriate process parameters helps enable the manufacture of bio-oils, bio-fuels, or biogas.

## Conclusions

The wastewaters produced by dairies are milk water, white water, and spent cleaning baths. Neutralization of these types of baths might be performed through the membrane filtration processes. The RO/ROP system is proposed to recover the water from the milk and white water. However, the use of UF/RO or NF/RO systems is proposed for the regeneration of spent cleaning baths. Prior to membrane filtration and the removal of volatile organic substances, it is necessary to appropriately prepare the process wastewater. The special advantages of the systems based on membrane processes are the possibilities of water recovery, the regeneration of spent cleaning baths, and the extraction of ingredients for the production of dairy products used as animal feeds or used as sources of bioenergy.

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