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THE POSSIBILITY FOR THE USE OF ULTRAFILTRATION FOR THE TREATMENT OF POTATO PROCESSING WATER

Key words: ultrafiltration, fouling, wastewater, potato processing water, treatment.

Abstract: The legal regulations governing the water and wastewater management in enterprises and the announcements of their changes have been discussed. Potato processing water (PPW), resulted from potato chips processing line, was examined. The possibility of PPW treatment using ultrafiltration was investigated. A laboratory filtration plant and polysulfone flat sheet membranes (cut-off – 100 kDa; effective surface area – $1.4 \cdot 10^{-2} \text{ m}^2$) were used. Filtration was carried out in a cross flow system at 2 and 4 bar operating pressures. The formation of the fouling layer was minimized by applying centrifugation prior to the filtration process and washing the membrane with NaOH and H_2O_2 solutions. The use of ultrafiltration for the treatment of examined PPW decreased the turbidity and total suspended solids by 97–100% and other determined physico-chemical parameters by 12–96%.

Możliwość zastosowania ultrafiltracji do oczyszczania ścieków przemysłu ziemniaczanego

Słowa kluczowe: ultrafiltracja, fouling, ścieki, ścieki ziemniaczane, oczyszczanie.

Streszczenie: W pracy przedstawiono przepisy prawne dotyczące gospodarki wodno-ściekowej w przedsiębiorstwach przemysłowych oraz zapowiedzi ich zmian. Badano możliwość zastosowania do oczyszczania ścieków przemysłowych procesu ultrafiltracji. Oczyszczaniu poddano poużytkowe ścieki ziemniaczane emitowane w procesie produkcji chipsów ziemniaczanych. W badaniach stosowano laboratoryjną instalację filtracyjną i płaskie membrany polisulfonowe (punkt odcięcia – 100 kDa, efektywna powierzchnia filtracyjna – $1,4 \cdot 10^{-2} \text{ m}^2$). Filtrację prowadzono w układzie „krzyżowym” przy ciśnieniu roboczym 2 i 4 bary. Powstawanie warstwy *foulingowej* ograniczono poprzez zastosowanie wirowania przed procesem filtracji oraz mycie membrany z użyciem roztworów NaOH i H_2O_2 . Zastosowanie ultrafiltracji do oczyszczania badanych ścieków pozwoliło na zmniejszenie mętności i zawiesiny ogólnej o 97–100% oraz innych oznaczanych parametrów fizykochemicznych o 12–96%.

Introduction

Wastewater treatment has been a challenge for industrial enterprises for many years. In December 2015, there was a further increase in demand for technologies aimed not only at the purification of industrial wastewater but also the recovery of water and other raw materials. This was due to the adoption

of the Circular Economy Package by the European Commission [1], which aims to stimulate Europe's transition towards a circular economy, thus enhancing global competitiveness, generating new jobs, and providing sustainable economic growth. Key objectives of this package include, among others, a series of actions related to water reuse, including a legislative proposal on minimum requirements for the reuse of water.

Currently, industrial wastewater can be discharged into the sewerage system or into the environment. In certain cases, it can be recycled to the production process (“closed water circuit”). In each case, the wastewater must meet the criteria specified in the relevant legal acts, including the following in Poland: 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].

Wastewater emitted by the food industry is a particular problem, due to the presence of significant amounts of organic components, which results in a high chemical oxygen demand. Their chemical composition is a reflection of the chemical composition of the raw materials used in the production. Potato processing water, generated in the processes of slicing potatoes and washing them (Fig. 1 [5]), contains significant amounts

of carbohydrates, mainly starch (present in the form of free suspended solids), proteins, and other ingredients (e.g., mineral components), and it is a significant source in environmental pollution. PPW cannot be discharged directly into the municipal sewerage system or the environment, nor can it be reused in the production process, due to the high chemical and biological oxygen demands and suspended solids [6]. For its purification, complex, usually several stages, processes of treatment are used [6]. Potato processing water contains high concentrations of readily biodegradable compounds; therefore, it is usually purified using biotechnological methods [7–9]. Various combinations of aerobic and anaerobic biological processes are most commonly applied [7, 9]. The main disadvantage of the applied methods is the long process time. In consequence, large volume bioreactors allowing industrial effluent treatment is needed. Furthermore, microorganisms are very sensitive to such factors as temperature and pH.

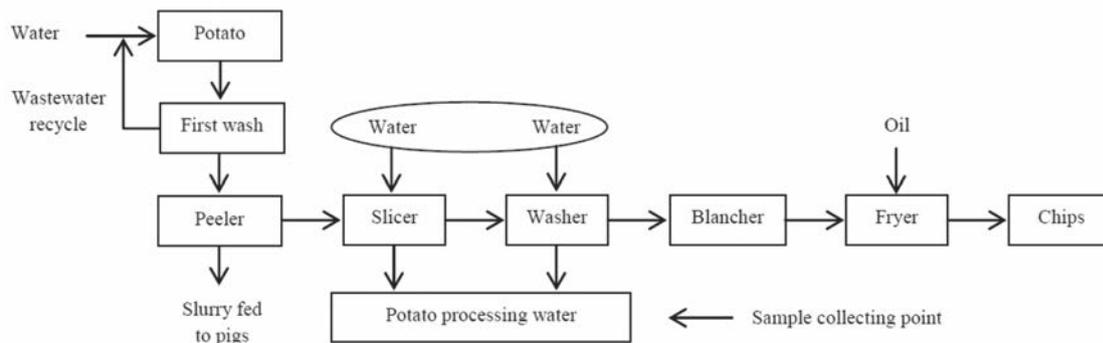


Fig. 1. Block diagram of the potato chips production and processing water

Source: Dabestani S., Arcot J., Chen V.: Protein recovery from potato processing water: Pre-treatment and membrane fouling minimization. *Journal of Food Engineering*. 2017, 195, 85–96.

Membrane technology has been successfully employed for wastewater treatment and the separation of ingredients in the food industry, especially in the dairy industry [10, 11]. Membrane filtration can be used to recover and recirculate the raw materials and water used in the production processes, reducing the need for them. Membrane technology offers the advantages of higher effluent water quality, a more compact footprint, and often simpler handling as compared to conventional processes. In addition, operating costs of membrane processes are considerably lower. Membrane filtration is classified in BAT (Best Available Technology) studies as clean (non-waste) technology. However, membrane filtration is not commonly used for the purification of PPW and the recovery of raw materials and water, mainly due to the occurrence of the fouling phenomenon.

Membrane fouling has always been a challenge for the practical application of membrane filtration due to economic and environmental factors. It is caused by the accumulation of the feed components on the membrane

surface and in the pores of the membrane. In addition, there is a chemical interaction between solutes and membrane material (concentration polarization and gel formation), and there is the growth of microorganisms on the membranes [12, 13]. A reduction in the permeate flux over time caused by fouling can be minimized by using various pretreatment methods and regular chemical cleaning, called cleaning-in-place (CIP). The most commonly used pretreatments are centrifugation, sedimentation, prefiltration, and microfiltration, and the most common cleaning agents are acids, bases, surfactants, and enzymes [14–16]. In the potato industry, fouling is mainly associated with the deposition of starch and proteins on the surface of the membrane. Better conditions for removing the fouling layer in the PPW filtration process were achieved using an alkaline cleaner (e.g., NaOH) as compared to surfactant (e.g., SDS) and acid (e.g., HCl) agents [5]. Efficiencies of these processes were 97%, 83% and 75%, respectively.

With a growing interest in the circular economy, there is potential in viewing food industry wastewater, indeed not as a waste, but as a valuable co-product suitable for further processing [17, 18]. PPW can be a source of useful products, such as starch and proteins [5, 19]. Common practices for the separation of useful product fractions from potato processing water include the use of sedimentation, hydro-cyclones, centrifuges, and in some cases membrane technologies (among others increasingly popular membrane bioreactors) [20, 21].

Since the announcement of more restrictive environmental regulations and with an increase in wastewater treatment and landfill costs, the food industry has seen a growing need to recover water and other useful raw materials from by-products. The aim of the study was to investigate whether the use of ultrafiltration, which is more energy effective than nanofiltration and reverse osmosis, for the potato processing water treatment is sufficient to discharge them into the

environment or to reuse the recovered water in the chips production process, and to propose a method of fouling layer removal from PES membranes.

1. Materials and Methods

1.1. Wastewater characteristics

Industrial wastewater was obtained from a potato chips processing factory located in Radom (Poland), which processes approximately 100 tons of potatoes and produces 40 m³ of wastewater per day. The starch-rich wastewater samples, resulted from the process line where potatoes are sliced and washed, were taken from a drain pipe located in plant area. Samples were collected five times within 3 months (from October to December) and immediately filtrated.

The characteristics of examined wastewaters are given in Table 1. The pH of the examined wastewater was 7.2 ± 0.3 and no pH adjustment was performed.

Table 1. Physico-chemical parameters of examined wastewater collected within 3 months

| Parameter/Analyte | Unit | Range of determined value/ concentration |
|--|-----------------------|---|
| Dry residue | % | 0.8–1.5 |
| Conductivity | μS·cm ⁻¹ | 1187–1451 |
| Turbidity | NTU | 496–2599 |
| Total suspended solids | mg·L ⁻¹ | 1027–1584.7 |
| COD | mg·L ⁻¹ | 3864–9275 |
| TOC | mg·L ⁻¹ | 846–2243 |
| ΣP (as PO ₄ ³⁻) | mg·L ⁻¹ | 4.0–9.17 |
| ΣN | mg·L ⁻¹ | 160.7–263.2 |
| Microbiology | Total including G- | 0.54–1.25*10 ⁶ 0.08–0.5*10 ⁶ |

1.2. Wastewater pretreatment

In order to remove the suspended and settled solids from the tested wastewaters, sedimentation for 1 hour followed by centrifugation for 5 minutes at a speed of 5000 rpm was used. This reduced dry residue, turbidity, total suspended solids, COD and TOC by 21.7%, 13.4%, 24.6%, 33.2%, and 53.3% on average, respectively.

1.3. Ultrafiltration experiment

Membrane experiments were conducted on a laboratory scale. The plant (Sterlitech, USA) was fitted with a membrane module placed in the hydraulic press, a conical fuel tank (19 L), a thermostatic system,

and a high pressure pump. The polysulfone flat sheet membranes (cut-off – 100 kDa) were used. The effective surface area of membrane was 1.4 · 10⁻² m². Filtration was carried out in a cross flow system (“batch” structure). Initial membrane resistance was tested prior to UF by using deionized water as a feed. The processes were conducted at an operating pressure of 2 and 4 bar, maintaining a constant feed temperature of 25 ± 1°C. The flow rate of the concentrate was in the range of 0.22 to 0.24 m³ h⁻¹. Permeate was collected in a graduated cylinder for permeate flux measurements, and the averaging samples of collected filtrate and concentrate for physico-chemical tests were taken after the process was terminated.

The permeate flux (at constant temperature and pressure) was calculated by the following equation:

$$J_A = \frac{V}{A \cdot t} \quad (1)$$

where J_A is the permeate flux ($\text{mL} \cdot (\text{min} \cdot \text{cm}^2 \cdot \text{bar})^{-1}$), V is the volume of filtrate (mL), A is the effective area of flat sheet membrane (cm^2) and t is the sampling time (min).

The efficiency of PPW purification was determined by the rejection coefficient calculated according to the following equation:

$$R = \left(1 - \frac{x_p}{x_f} \right) \times 100\% \quad (2)$$

where R is the rejection coefficient (%), x_p and x_f are the values of the parameter tested or the concentrations of the component in the permeate and in the feed, respectively.

The rejection coefficients were calculated for all examined physico-chemical parameters. The confidence intervals ($\pm \Delta R$) for the received results were calculated by the equation obtained from the differentiation of Equation 2. The final form of the equation is as follows:

$$\frac{\Delta R}{R} = \left| \frac{\Delta x_f + \Delta x_p}{x_f - x_p} \right| + \left| \frac{\Delta x_f}{x_f} \right| \quad (3)$$

where Δx_f and Δx_p are the uncertainties of the determined physico-chemical parameters in the feed and permeate, respectively.

The possibility of membrane regeneration using hydrogen peroxide and sodium hydroxide was investigated. Three membrane filtration processes were performed at 4 bar working pressure:

1st process – using a new membrane;

2nd process – with the filtration of the new wastewater batch with a membrane already used in the first process, after cleaning its surface with a stream of deionized water;

3rd process – with the filtration of the next wastewater batch with a membrane already used in the first and the second processes, after cleaning its surface with a stream of deionized water followed by an off-site sequential cleaning of the membrane by immersion in the following:

- a) deionized water (24 h),
- b) 0.5% sodium hydroxide solution (6.5 h),
- c) deionized water (16 h),
- d) 0.03% hydrogen peroxide solution (6.5 h),
- e) deionized water (24 h).

These processes are marked throughout the paper as “1st process,” “2nd process,” and “3rd process”, respectively.

1.4. Reagents

All reagents used in the work were of analytical grade. Water purified in a Hydrolab HPL system (a specific conductance $< 0.06 \mu\text{S cm}^{-1}$) was used in all experiments.

1.5. Analytical procedures

Raw wastewaters, feeds, and permeates taken during each process were analysed for dry residue, conductivity (κ), turbidity, total suspended solids (TSS), chemical oxygen demand (COD), and total organic carbon (TOC), and phosphorus and nitrogen contents. The dry residue was determined at 105°C using the laboratory moisture analyser (MAC 50/1, Radwag, Poland), and turbidity was measured with a 2100Q IS Portable Turbiditymeter (Hach Lange, Germany). SevenMulti conductometer and InLab 731 conductivity probe, both from Mettler Toledo (USA), were used to examine conductivity changes of the samples collected after the filtration process. Total suspended solids, chemical oxygen demand (COD), and total organic carbon (TOC), phosphorus, and nitrogen contents were determined by spectrophotometry. A DR 6000 spectrophotometer (Hach Lange, Germany) was used. The wastewater chemical analyses were carried out using cuvette tests (Hach Lange, Germany; Table 2). The TSS was determined using 25 mL cuvette and measured at the 810 nm wavelength.

Table 2. Characteristics of methods used for the determination of chemical oxygen demand, total organic carbon, total phosphorous, and total nitrogen

| Parameter/ Analyte | Number of cuvette test | Applied method | |
|--|---------------------------|-------------------|---|
| | | Number of norm | Name of norm/method |
| COD | LCK 514 | ISO 6060 | Determination of the chemical oxygen demand |
| TOC | LCK 387 | EN 1484 | Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC) |
| ΣP (as PO_4^{3-}) | LCK 350 | EN ISO 6878 | Determination of phosphorus. Ammonium molybdate spectrometric method |
| ΣN | LCK 338 | EN-ISO 11905-1 | Determination of nitrogen. Method using oxidative digestion with peroxodisulphate |

2. Results and discussion

The permeate flux for the examined potato processing water was approximately 1.7 times higher for 4 bar operating pressure compared to 2 bar (Fig. 2). Carrying out the process under pressure elevated to 4 bar did not significantly affect the amounts of retained organic carbon compounds by the membrane (Fig. 3). During the filtration process carried out under 2 bar of transmembrane pressure, only higher amounts of

nitrogen and phosphorus compounds were stopped. Other determined physico-chemical parameters (dry matter, turbidity, total suspended solids, and chemical oxygen demand) of purified wastewaters were decreased to a comparable extent. In both cases, a significant (77–99%) purification degree of examined PPW from organic substances (total organic carbon content) and the reduction of turbidity and total suspended solids were achieved (Fig. 3).

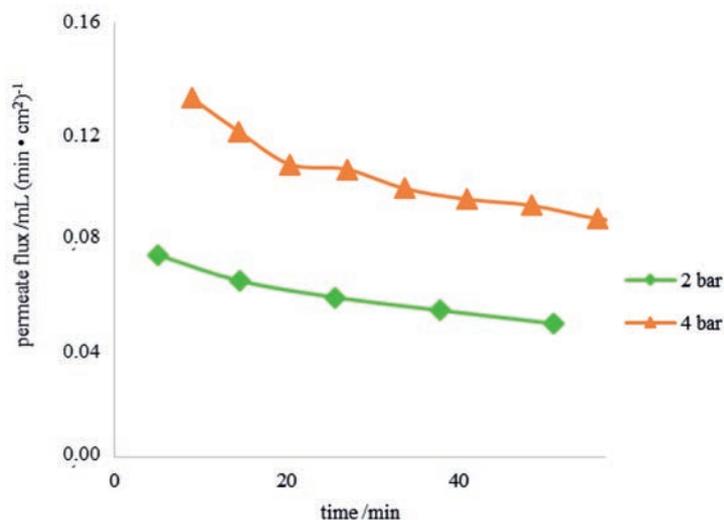


Fig. 2. Changes in PPW permeate flux for processes conducted under 2 and 4 bar of transmembrane pressure
Source: Authors.

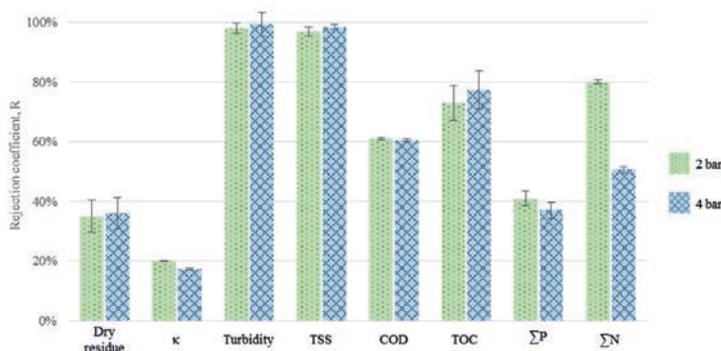


Fig. 3. A comparison of rejection coefficients for determined physico-chemical parameters (i.e. dry residue, conductivity (κ), turbidity, total suspended solids (TSS), chemical oxygen demand (COD), total contents of organic carbon, phosphorus, and nitrogen) of industrial potato wastewater purified under operating pressures of 2 and 4 bar
Source: Authors.

As a result of the ultrafiltration processes (on membranes not subjected to periodic cleaning), 36–38% (v/v) of water was recovered from examined industrial wastewater. The limited efficiency of these processes is due to the phenomenon of fouling, i.e. continuous blocking of the membrane pores by components

(suspended or dissolved) of purified liquid, increasing the hydraulic resistance of the flow through the membrane and consequently decreasing the filtration rate (Fig. 2). Increased filtration efficiency and the possibility of continuous operation of the membrane plant are ensured through periodic regeneration of

membranes. The physical cleaning of the membrane with a stream of deionized water is insufficient to restore the initial filtration efficiency (Figs. 4a, and b, 5b and d). The decrease in this productivity, which is probably only due to the partial release of the membrane pores, simultaneously has a beneficial effect on the filtration efficiency (Fig. 6). The filtrates obtained after these processes were markedly reduced in chemical oxygen demand, dry residue, as well as phosphorus and nitrogen contents. In the case of polymer membranes,

the flow direction of water cannot be reversed to remove the fouling layer, due to the limited strength of the membrane material, which is generally insufficient for the physical cleaning of the membrane surface. In such configurations, the filter membranes are regenerated by using appropriately selected chemical reagents and/or enzyme preparations. In this paper, the efficiency of removing the fouling layer from PES UF membrane with sodium hydroxide and hydrogen peroxide solutions was investigated.

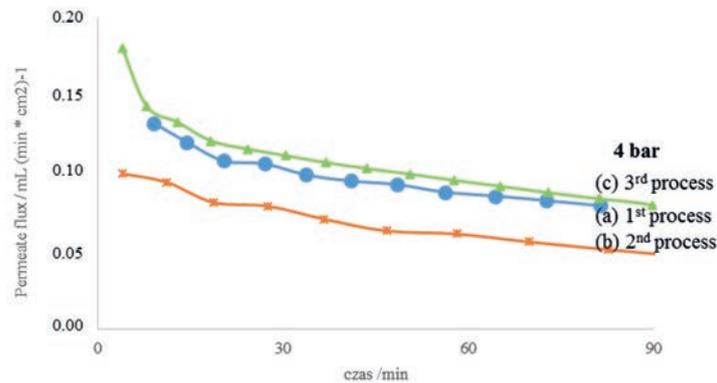


Fig. 4. Changes in the PPW permeate flux obtained on the new ((a) 1st process) and regenerated ((b) 2nd and (c) 3rd processes) membrane (4 bar)

Source: Authors.

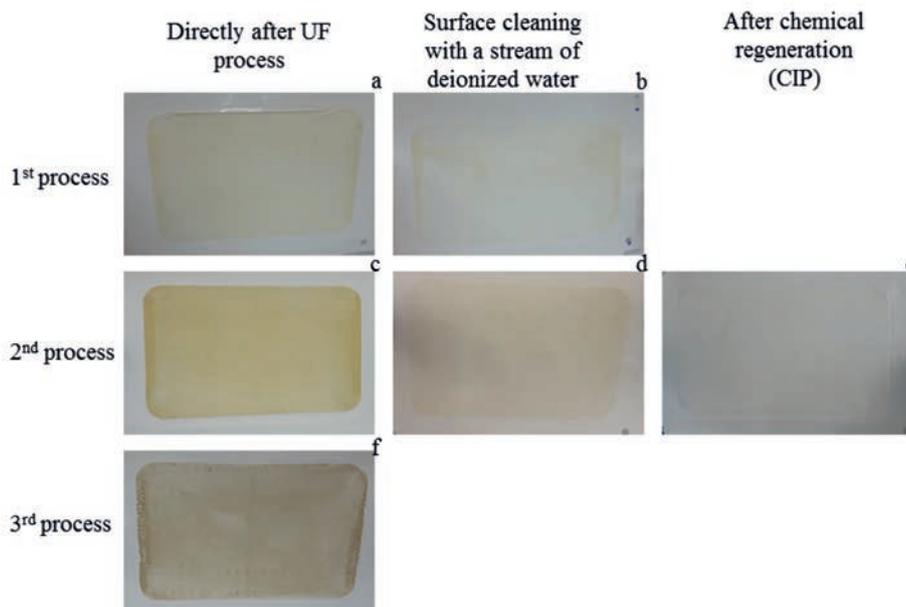


Fig. 5. Membrane photographs taken directly after ultrafiltration processes (a,c,f), after surface cleaning with a stream of deionized water (b,d), and chemical regeneration using NaOH and H₂O₂ solutions (e)

Source: Authors.

The filtration efficiency of the potato industrial wastewater carried out on the regenerated (using proposed method) membrane was comparable to that on the membrane used for the first time (Fig. 4a and c). Analysis of determined rejection coefficients (Fig. 6) has shown that the use of NaOH and H₂O₂ regenerated

membrane in the filtration process results in a higher degree of purification compared to filtration carried out on the new membrane, and it is comparable to that for re-used membrane cleaned only by flushing with a stream of deionized water.

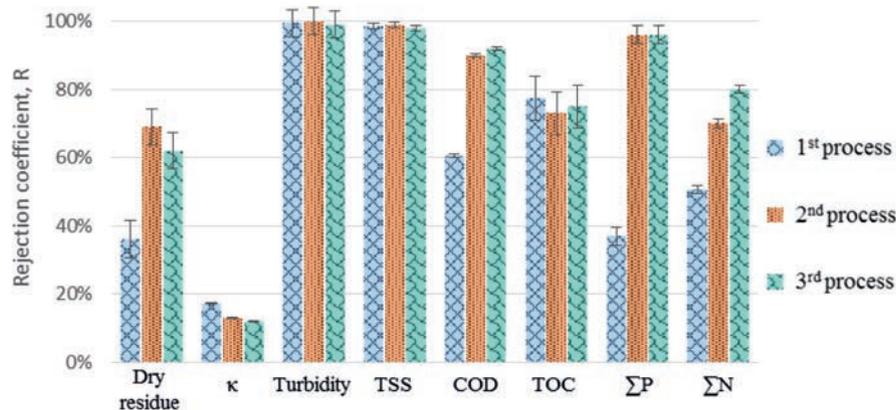


Fig. 6. A comparison of rejection coefficients for the determined physico-chemical parameters of PPW treated by ultrafiltration using new (1st process) or regenerated (2nd and 3rd processes) membrane

Source: Authors.

A comparison of the results of water recovered in the membrane filtration process with currently applicable legal regulations for water intended for human consumption, wastewater discharged into water or soil, and wastewater introduced into sewerage equipment is presented in Table 3. Ultrafiltration treatment of PPW is insufficient neither for reuse of recovered water in the production process (e.g., for potato cleaning) nor for its direct discharge into the environment (water or soil). In the first case, when the water has to meet the quality criteria for water intended for human consumption, the

turbidity of the filtrates is greater than the permissible value of this parameter. Discharge of permeate into the environment requires the reduction of the concentration of the compounds that can be oxidized by dichromate ions (COD_{Cr}), as well as total organic carbon, phosphorus, and nitrogen contents. In order to obtain more purified water, nanofiltration is proposed. The results obtained in this work will be verified in a larger technological scale, i.e. in quarter-technical conditions using the prototype membrane installation (Fig. 7) designed and built in the Institute for Sustainable Technologies – NRI.

Table 3. The physico-chemical parameters and concentrations of analytes determined in the obtained filtrates and the maximum allowable values of these parameters for water intended for human consumption (Dz.U. 2015 poz. 1989), wastewater discharged into water or soil (Dz.U. 2017 poz. 328), and wastewater introduced into sewerage equipment (Dz.U. 2006 nr 136 poz. 964)

| Parameter/Analyte | UF permeate | Legal standards | | |
|---------------------------------|-------------|--------------------------------------|--|---|
| | | water intended for human consumption | wastewater discharged into water or soil | wastewater introduced into sewerage equipment |
| Dry residue /% | 0.11–0.16 | – | – | – |
| pH | 6.9–7.5 | 6.5–9.5 | 6.5–9.0 | 6.5–9.5 |
| Conductivity / $\mu S\ cm^{-1}$ | 1008–1075 | 2500 | – | – |
| Turbidity /NTU | 2.7–5.3 | 1 | – | – |
| TSS /mg L ⁻¹ | 10.5–11.5 | – | 35 | * |
| COD_{Cr} /mg L ⁻¹ | 790–955 | – | 125 | * |
| TOC /mg L ⁻¹ | 188–192 | without abnormal change | 30 | * |
| ΣP /mg L ⁻¹ | 5.7–7.8 | – | 3 | * |
| ΣN /mg L ⁻¹ | 71.6–94.2 | – | 30 | – |

* The value of the parameter should be determined on the basis of the permissible pollution load at the wastewater treatment plant.



Fig. 7. Prototype membrane ultrafiltration plant

Source: Authors.

Summary

The EU's policy on water and wastewater management in companies envisages the reduction of water consumption through wastewater treatment and the reuse of recovered water. The use of closed water circuits aims to protect the environment, save money, and increase the competitiveness of companies on the international market. Achieving this goal in the food industry requires the purification of post-consumer water to the level required for the water intended for human consumption. Membrane techniques enable the purification of industrial wastewater to the required level. The use of ultrafiltration for the treatment of potato processing water has reduced the conductivity by 12–20% and other determined parameters by 35–100%. However, reclaimed water can be neither reused in the chips production process, because it does not meet the requirements (i.e. turbidity) for water intended for human consumption, nor can it be directly discharged into the environment, due to the high COD_{Cr} , TOC, and total phosphorus and nitrogen contents. The ultrafiltered PPW can be discharged to the sewerage equipment. The further purification of examined wastewater to a level that allows them to be reused in the production process or discharged into the environment requires nanofiltration and reverse osmosis if necessary.

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