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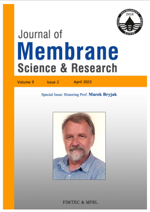

Research Paper

Membrane separation processes in the treatment of municipal wastewater

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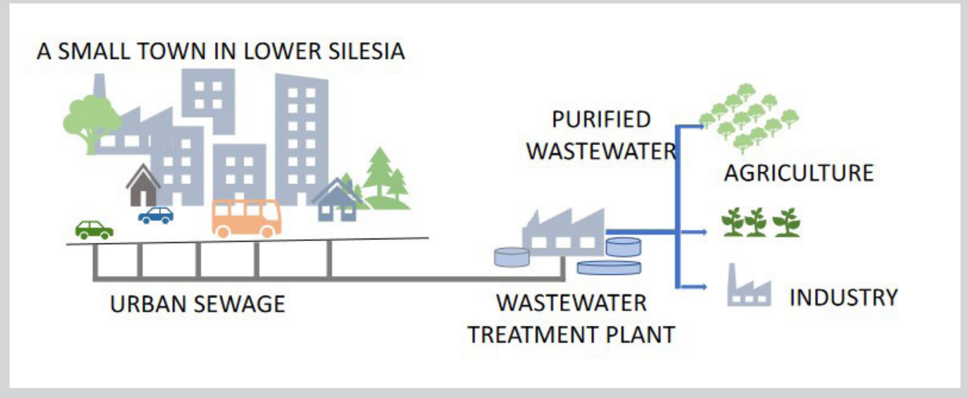
Highlights

- A real municipal wastewater from a medium-sized town in the Lower Silesia Province of Poland was treated
- Ceramic UF and polymeric UF and NF membranes were tested.
- The Best treatment results were achieved by the combination of a 50kDa ceramic UF and a 200Da polymeric NF membranes

Graphical abstract



Abstract

The municipal wastewater from a medium-sized town in the Lower Silesia Province of Poland was treated by several methods. They included filtration through ceramic, 300 kDa or 50 kDa, polymer membranes, 5 or 30 kDa, sedimentation, and nanofiltration membranes with a cut-off 200 Da (NF90), 400 Da (NF270), and 300-500 Da (NFW). The character of all streams at each stage of the treatment process was determined by detecting the chemical oxygen demand, total nitrogen, and phosphorus. Concentration of sodium, calcium, magnesium, and potassium ions was also detected after the purification process. The best treatment parameters were achieved in the case of using a combination of ultrafiltration on 50 kDa ceramic membrane and nanofiltration on NF 90 polymeric membrane. It was a determined reduction in total nitrogen by a factor of 3, phosphorus by a factor of 9, and a decrease in sodium from 101.47 mg/L to 21.58 mg/L in the final permeate.

1. Introduction

The 21st century, known as the age of water, puts the world in crisis: water quality is deteriorating due to continued population growth, rising living standards, and urbanization of ever larger areas with inadequate water and sewage management. Increasing the pollution of natural water resources is a global problem. Human economic activity results in an increase in the levels of pollution in natural waters, which in turn manifests itself in the difficulty of treating these waters for food and industrial purposes. According to the European Environment Agency [1], water scarcity affects up to 1/3 of the territory of the European Union today. Such a situation leads to the introduction of new regulations so that previously unusable water can be used for other purposes (e.g., boiler, cooling, industrial, agricultural, street cleaning, irrigation of green spaces and golf courses, public fountains, etc.). This is expected to lead to improved economic performance and environmental protection [2].

In highly developed countries, closed water loops are used. Wastewater treatment requires a series of complementary technologies that primarily recover valuable substances in wastewater or remove pollutants to such an extent that treated wastewater can be reused for industrial purposes or safely discharged into the environment [3-5].

Both conventional physical and chemical processes (sedimentation, sorption, flocculation, or chemical precipitation) and membrane processes are used to treat wastewater in a closed-loop system [6, 7]. The choice of a suitable separation method is mainly influenced by two factors - it must be technically feasible and economically attractive. No less important, and in

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many cases decisive for the use of wastewater treatment methods, is the ecological aspect, including legal considerations. Although the problem of wastewater treatment is not new, the search continues for new, more attractive, more efficient, and less environmentally damaging methods and solutions tailored to the specifics of the waste being treated [8-11].

Membrane techniques are growing in interest and popularity. Their use is associated with many advantages, including the ability to remove a wide range of pollutants, low consumption of raw materials and energy, and no need to dose chemicals [12-15]. An unquestionable advantage of these methods is also the fact that after the treatment process, no intermediates of pollutant decomposition remain in the wastewater. The frequent use of membrane processes can be attributed to [16]:

- The ability to run the process continuously and at ambient temperature,
- The ease of combining with other treatment processes, including subsequent membrane processes,
- The lack of need for additional substances that constitute ballast or environmental hazards,
- The wide variety of membranes available on the market,
- The ease of selecting a membrane system to meet specific needs.

Despite the many advantages that membrane processes offer, their use is also associated with difficulties that can be reduced to varying degrees. Among the most common problems associated with membrane operation during wastewater treatment are concentration polarization, adsorption, formation of a biological layer on the membrane surface, scaling, and fouling. These phenomena have a decisive effect on the size of the permeate flux and its changes over time [17-20].

In the group of low-pressure membrane separation processes, ultra- and nanofiltration processes can give promising results for wastewater treatment [21,22]. Ultrafiltration involves the retention of fine suspended solids, colloids, bacteria, and viruses. The transport mechanism is sieve-like, which means that particles larger than the diameter of the pores do not pass through the membrane. Using transmembrane pressures in the range of 0.1 - 1 MPa, large permeate streams (up to several L/m^{2}h) can be obtained. The ultrafiltration process uses asymmetric porous membranes with a thickness of about 150 µm formed from various materials (from polymers to inorganic materials such as ceramics or metals). Due to its high selectivity, nanofiltration is also considered a suitable technology for wastewater treatment. In nanofiltration, the membrane retains substances with molecular weights in the range of 200-1000 Da. In this case, the separation mechanism is not only by sieving but also by dissolution and diffusion. In the literature, some papers have already described studies on the treatment of wastewater from other regions of the Lower Silesia Province. Data from a waste treatment plant, with about 260,000 inhabitants, were presented based mainly on biological treatment. The amount of nitrogen was 1.5% dry matter, phosphorus 0.55% dry matter, and potassium 1.0% dry matter, indicating a possible positive use in agriculture [23]. Also, the organic fraction of municipal waste located in Lower Silesia (Poland) was tested using the pressure membrane filtration process. In the experiments flat ceramic membranes for microfiltration and ultrafiltration from Tami Industries were used, and the average pore radius ranged from 0.035 to 0.29 µm. The best separation was observed for the 1 kDa membrane. The COD for the permeate after using this membrane decreased from over 6000 to almost 300 mg/L [24].

Given the above information, our goal was to determine the suitability of membrane separation processes (ultrafiltration and nanofiltration) for treating municipal wastewater from the middle-size region of 25000 PE (person equivalent) located in the Lower Silesia Province.

2. Methods and materials

2.1. Wastewater

Municipal wastewater was collected in the middle community sewage outlet. For this study, two types of samples were selected: low and highly polluted. As a metric of the pollution level, the COD parameter was taken.

2.2. Methods of wastewater treatment

Ultrafiltration spiral ceramic membranes in JAM INOX installation

The ultrafiltration process with the use of ceramic membranes was carried out with the use of a JAM INOX laboratory installation (Fig. 1) consisting of a membrane module, a 10 L feed tank, a flux temperature control unit, and a Grundfos pump. The membranes were hermetically sealed in a metal housing. The transmembrane pressure used in each experiment was set at 0.4 MPa. Each new membrane, before experiments, was conditioned by 15 minutes of filtration of tap water followed by 15 minutes of filtration of 0.1 M HCl solution, washing for 15 minutes of tap water, and filtering of distilled water until a constant permeate flux (J_0) was obtained. INSIDE CéRAM™ tubular ceramic membranes (TAMI Industries) were used in this study. The properties of the membrane can be found in Table 1.

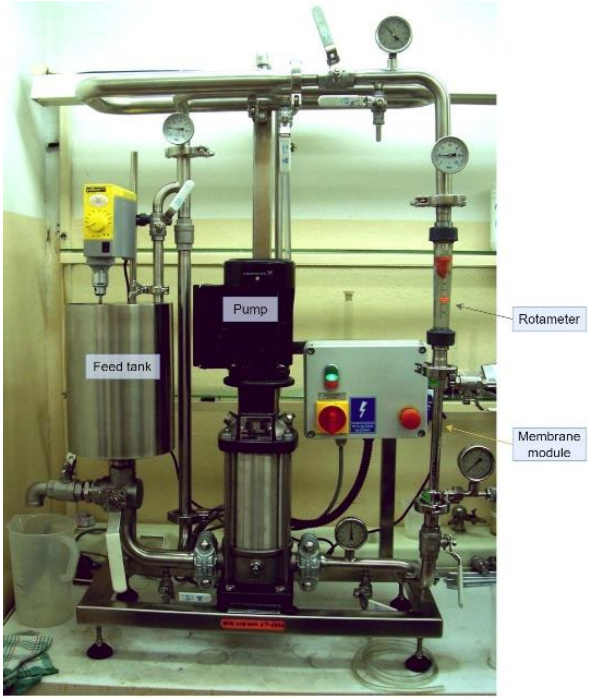

Fig. 1. JAM INOX cross-flux membrane system

Table 1
Membranes used in experiments in JAM INOX installation

Membrane type		Material	Cut-off, (kDa)	Max. pressure, (MPa)	Max temp., (°C)	pH range	Effective filtration area, (cm^2)
Tubular ceramic, 1-channel							
Ceram INSIDE 50 kDa	UF	$Al_2O_3 \cdot TiO_2$	50	< 9	150	0-14	40
Tubular ceramic, 7-channel							
Ceram INSIDE 300 kDa	UF	$Al_2O_3 \cdot TiO_2$	300	< 9	150	0-14	125
Polymeric membranes							
Biomax UF 5	UF	Polyethersulfone	5	na	95	1 - 14	28.9
Biomax UF 30	UF	Polyethersulfone	30	na	95	1 - 14	28.9
Dow Chem NF90	NF	Polyamide	0.2	4.1	45	2 – 11	28.9 and 266
Dow Chem NF270	NF	Polyamide	0.4	4.1	45	2 – 11	28.9
NFW-TFC	NF	Polyamide	0.3-0.5	4.1	50	4 - 10	266

SEPA CF II membrane cell system

The Crossflux membrane Sepa* CF II Membrane Cell system was used for the nanofiltration process. In the cell, a flat sheet nanofiltration membrane was placed and pressurized to 0.16 MPa. The following membranes, 14x19 cm, were tested: NF90 (cut-off 200 Da) Dow Chemical Company, Synder Filtration NFW (TFC cut-off 300-500 Da).

Amicon 8200 cell system

Two samples of processed wastewater were treated in the Amicon Stirred Cell Mode 8200 at 0.42 MPa. In the case of highly polluted wastewater, two types of Dow Chem polyamide membranes were tested, NF90 (cut-off 200 Da) and NF270 (cut-off 400 Da). Low polluted wastewater was filtered through Biomax polyethersulfone membranes PBCC06210, B5K - UF 5 (cut-off 5 kDa) and PBTK06210, B30K - UF30 (cut-off 30 kDa) delivered by Merck.

2.3. Characterization of samples

2.3.1. Chemical analysis of wastewater components

The COD, N_{total}, P_{total}, $N\text{-}NO_2$, $N\text{-}NO_3$, $P\text{-}PO_4$, SO_4^{2-} and N-NH components were determined using HACH's Cuvette spectrophotometry tests (Hach Lange, USA). The $Ca^{2+,}$ Mg^{2+}, K^+, and Na^+ concentration was monitored by atomic absorption spectroscopy (GBC Avanta). The treated wastewater parameters according to the regulations of the Minister of Environment of 29 November 2002 are presented in Table 2 [25].

Table 2
The highest value allowed for the indicator in Poland [25]

Indicator name	Unit of measure	The highest allowed value
COD	mg O_2/L	150
Total nitrogen	mg N/L	30
Ammonium nitrogen	mg $N\text{-}NH_4$/L	10
Nitrate nitrogen	mg $N\text{-}NO_3$/L	30
Nitrite nitrogen	mg $N\text{-}NO_2$/L	1
Total phosphorus	mg P/L	5
Sulfur	mg SO_4/L	500
Sodium	mg Na/L	800
Potassium	mg K/L	80
Magnesium	mg Mg/L	na
Calcium	mg Ca/L	na

2.3.2 Permeate flux

During all experiments, the permeate flux (J, L/m^{2}h), through the given membrane was calculated according to equation 1:

$$J = \frac{v}{t.s} \tag{1}$$

where, v is the permeate volume (L), t is the time of permeate collection (h) and s is the active membrane surface area (m^2).

2.3.3 SAR index

Sodium adsorption ratio (SAR) was calculated by using equation 2 [26]:

$$SAR = \frac{\left[Na^{+}\right]}{\sqrt{\frac{\left[Ca^{2+}\right]\left[Mg^{2+}\right]}{2}}} \tag{2}$$

2.4. Membrane regeneration

After mechanical cleaning of the wastewater tank, the membranes were regenerated according to the procedure suggested by the membrane manufacturer. In the case of the ceramic membrane, the CIP protocol was applied with the following four steps:

1. Washing the system three times with tap water, 30 minutes each time,
2. Filtrating 0.1m aq. Hydrochloric acid, 30 min,
3. Washing with tap water, 25 min,
4. Filtrating with distilled water, for 20 min.

Polymeric membranes were regenerated by washing the membrane according to the following procedure:

1. Filtrating the NaOH solution, pH 11, 15 min,
2. Washing with tap water, 15 min,
3. Filtrating citric acid solution pH 3, 15 min,
4. Wash twice with tap water, 15 minutes each time.

3. Results and discussion

A membrane separation system was used for municipal wastewater of a medium community to verify the thesis on the usability of such resources for small agglomerations. The resulting water should show the parameters that meet the regulations of the Ministry of Environment of November 29, 2002 [25]. Some of them are shown in Table 2 at point 2.3.1.

Due to the large difference in wastewater quality, the low-polluted water was treated as the first. Therefore, in this approach, ultrafiltration polymer membranes with various cut-offs 30 and 5 kDa, were tested (Fig. 2).

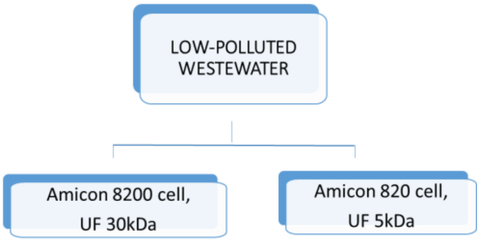

Fig. 2. Scheme of low polluted wastewater treatment

The wastewater was characterized before and after treatment (see Table 3). The wastewater studied had quite low COD compared to other sewages in Poland [27, 28]. Using ultrafiltration membranes, it was possible to reduce the amount of COD and ammonium and nitrate to the allowed values for wastewater discharged into water reservoirs [25]. However, the level of nitrogen and phosphorus in the treated water exceeded the acceptable level. For this reason, it was decided to continue the studies with the use of other membrane processes, such as filtration on ceramic membranes and nanofiltration of the permeate obtained. For these studies, highly polluted wastewater was taken. Therefore, having roughly acceptable results from low-polluted water, the processes for high-polluted water were searched.

Table 3
Chemical properties of low-polluted wastewater

	COD, mg/mL	N_{total} mg/L	P_{total} mg/L	$N\text{-}NO_2$ mg/L	$N\text{-}NO_3$ mg/L	$P\text{-}PO_4$ mg/L	SO_4^{2-} mg/L	N-NH mg/L
Raw wastewater	700	79.0	117	0.11	1.62	80.9	109.7	6.27
UF 30kDa	115	69.0	70	0.05	1.315	77.5	94.5	4.695
UF 5 kDa	102	53.0	59.4	0.04	1.32	23.65	78.0	0.545

The highly polluted wastewater was treated with the variants depicted in the 3 modes scheme (Fig. 3). Each mode of treatment was divided into two steps. In the first, for *Mode I* and *II*, ceramic ultrafiltration membranes, 50 kDa and 300 kDa, were used. In the case of *Mode III*, the sedimentation process was selected at ambient temperature (25°C) for 7 days. Since the highly polluted water was collected in a few days, the values of the feed parameters differ from one to another.

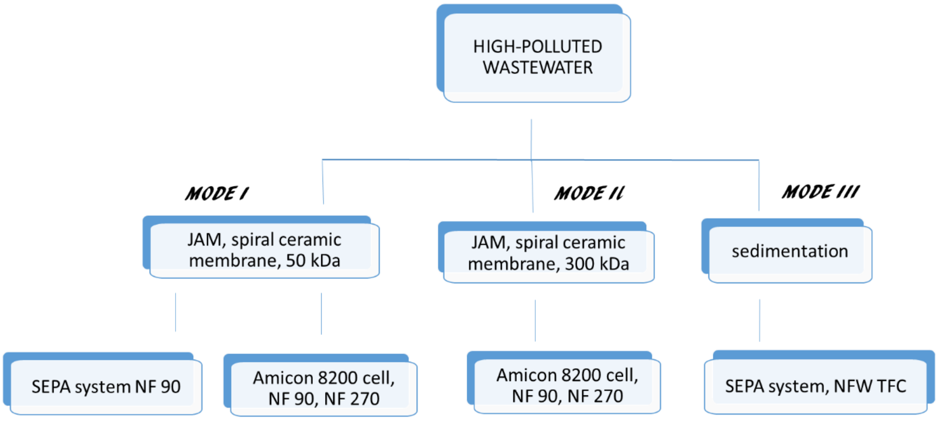

Fig. 3. Scheme of highly polluted wastewater treatment

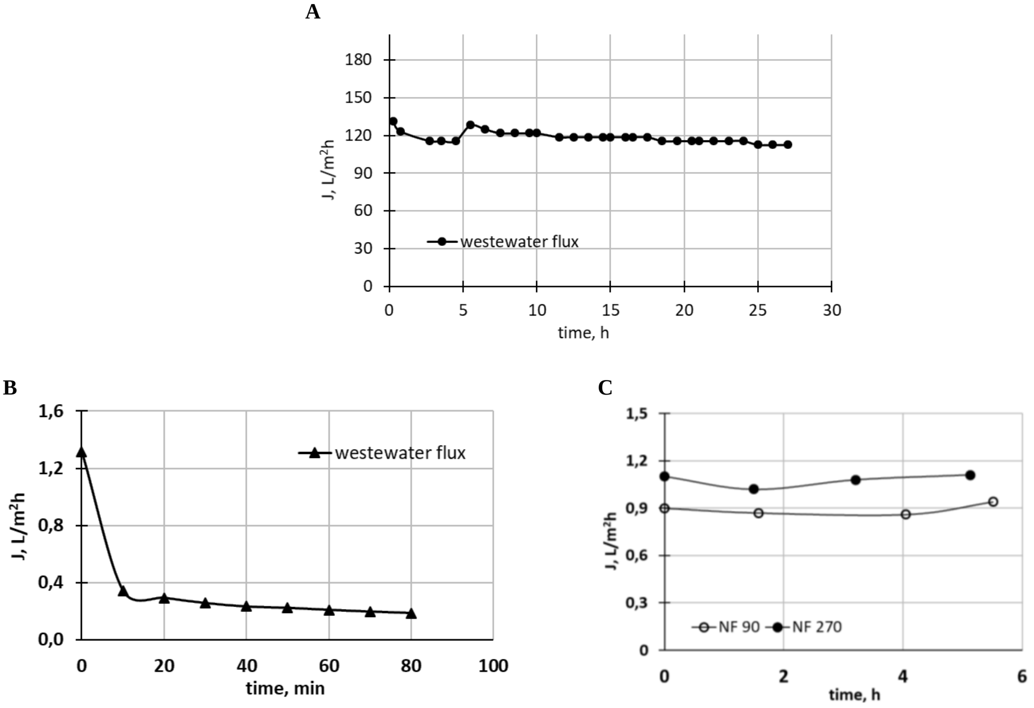

Fig. 4. The flux of highly polluted wastewater A:50 kDa, B: SEPA system NF90, C: Amicon cells NF 90 and NF270.

Mode I

In the first stage, a membrane with a cut-off of 50 kDa was used for filtration. This process was carried out for 27 h. The pure water flux before filtration was 130 L/m^{2}h while after filtration it decreased to 112 L/m^{2}h (Fig. 4A). Thus, a slight decrease in flux was noted, which after regeneration returned to its initial value. It is worth highlighting that no significant reduction in the stream was observed in the run of the process. In the next step, the UF permeate was nanofiltered in two ways. The first portion was loaded into the SEPA module with an NF90 membrane. In this case, the permeate flux decreased rapidly within the first 10 minutes from 1.31 L/m^{2}h to 0.34 L/m^{2}h (Fig. 4B). The second portion was filtered on the Amicon stirred cell. In this case, two types of membranes NF90 and NF 270 were tested. In both cases, the filtration was carried out for more than 5 hours and no changes in permeate flux were observed (Fig. 4C).

Before and after each stage of filtration, the chemical parameters of the wastewater were verified. The results were summarized in Table 4. All parameters of high-pollutant raw sewage were higher than those of low-pollutant sewage. It was probably related to a much higher ambient temperature and, therefore, a greater intensity of processes occurring in wastewater. The best parameters were achieved in the case of using this version of the filtration, in which the nanofiltration was carried out on the Amicon with the NF90 membrane. The total nitrogen value decreased by about 17 times, phosphorus by about 9 times, and nitrogen from the NO$_2$ groups was reduced from 0.31 mg/L to 0.06 mg/L. However, this mode did not reduce the amount of total phosphorus and ammonium nitrogen to acceptable values.

Mode II

In the second mode, a spiral ceramic module with a cut-off of 300 kDa was used for ultrafiltration. At the beginning of the process, a very significant reduction in flux was observed (from 200 L/m^{2}h to 62 L/m^{2}h). Over the next 3 hours, the flux slowly decreased and finally reached a level of 31 L/m^{2}h, finally (Fig. 5A). Such a significant decrease in flux was caused by the lack of mechanical pre-cleaning and the presence of large particles in the highly polluted wastewater. The permeate obtained was nanofiltered in the Amicon cell through the membrane of NF90 or NF270 (Fig. 5B-C). The tests were carried out for two consecutive days. A similar trend was observed for both membranes. The permeate flux varied between 1.5 L/m^{2}h and 1.0 L/m^{2}h on the first day and between 0.9 and 1.2 L/m^{2}h on the second day. A slightly smaller flux change on the second day could be caused by the fouling layer on the membrane surface. In *Mode II*, better results were obtained with NF90. In the case of NF270, the COD, and the total nitrogen values were 235.4 mg/L and 26.7 mg/L respectively, while NF90 offered 175.5 mg/L and 19.6 mg/L. However, the amounts of sulfates and ammonia were slightly lower when using the NF270 membrane than when using NF90. These differences were not significant, so it could be concluded that they were comparable (Table 4).

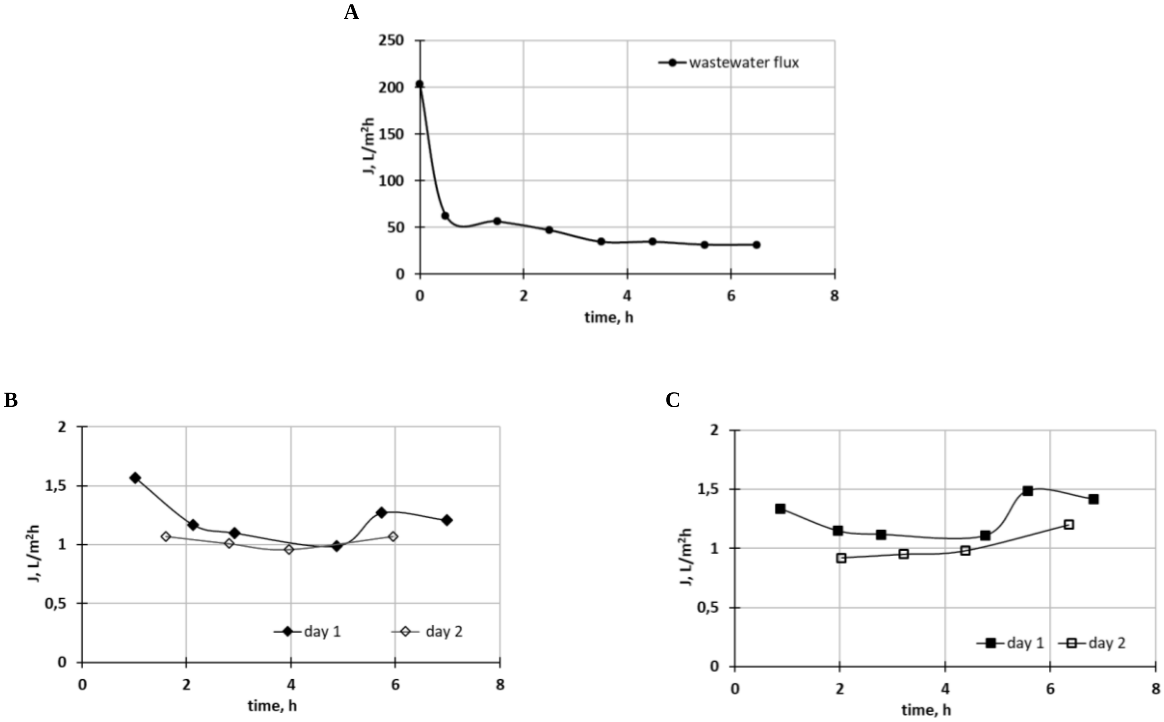

Fig. 5. Permeate flux during filtration, A**:** ceramic membrane 300 kDa, B: membrane NF270, and C: membrane NF90.

Mode III

In the last mode, sedimentation was used instead of ultrafiltration, and nanofiltration was carried out with a flat polyamide membrane (NFW TFC cut-off 300-500 Da). A fairly significant decrease in flux was observed during the first 10 minutes of the process from 15 L/m^{2}h to 12 L/m^{2}h (Fig. 6). However, it was not as rapid as in the case of *Mode I* with a 50 kDa ultrafiltration membrane and NF90 nanofilter in the SEPA cell. Therefore, it seems that in the case of this type of wastewater treatment, sedimentation turned out to be a better method of separation than UF followed by the use of an NF90 membrane. During the next 90 minutes of the process, the flux gradually decreased to a value of approximately 10 L/m^{2}h (Fig. 6). In this mode, a significant reduction in COD was also observed, but it was about 2 times greater than for *Mode I*. The amount of total phosphorus also decreased significantly (Table 4).

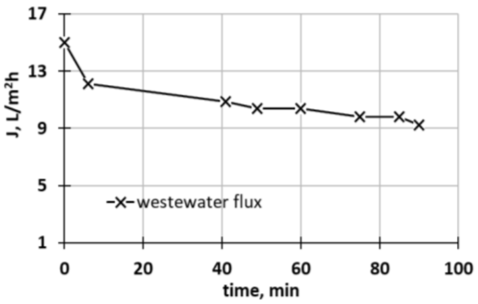

Fig. 6. Change in permeate flux during filtration in the SEPA system with the NFW TFC membrane.

The amount of sodium, potassium, magnesium, and calcium in the treated wastewater was also determined. This analysis was performed to investigate the potential suitability of the permeate for use as irrigation water. High sodium ions in water are unfavorable for plant growth. This is related to the fact that this element can change the permeability of the soil and cause infiltration problems. The suitability of water obtained after the treatment of various types of wastewater for use in agriculture is determined, among others, by the Sodium Adsorption Ratio (SAR) index. The SAR determines the relative ratio of sodium ions to calcium and magnesium. Depending on the SAR value, the waters were classified in terms of the possibility of their further use in agriculture. Thus, for a SAR of less than 10, it is considered that water can be used on sodium-sensitive crops. Water with a SAR range of 10 - 18 should be additional amendments (such as gypsum) and leaching is needed. The SAR index above 18 classifies water as generally unsuitable for use [29-31]. The amount of sodium has been reduced much more than required by the standards and, in some cases, more so than in other papers using the same membranes. For example, these types of membranes, DowChem NF90 and NF270, were used, among others, to treat wastewater from the Izmir City Treatment Plant, Turkey. A comparative ion analysis is shown in Table 5.

In the case of potassium, the permissible values have only been reached for *Mode I* with the option UF50/NF90 and for the option *Mode II*, UF300/NF90 (Table 6). The concentration of magnesium and calcium was also significantly reduced. The ratio of sodium to the sum of calcium and magnesium obtained in this way did not meet the standards for water approved for use in agriculture [31,34]. Perhaps, to make treated wastewater useful for agriculture, calcium should be added.

After the filtration process, the tubular ceramic and flat polymeric membranes used in the SEPA system were regenerated. The procedures used to clean the membranes used in the various stages of wastewater treatment worked very well. A 96-97% degree of regeneration was obtained for all materials.

Table 4
Chemical properties of highly polluted wastewater

	COD, mg/L	N_{total}, mg/L	P_{total}, mg/L	N-NO_2, mg/L	N-NO_3, mg/L	P-PO_4, mg/L	SO_4^{2-}, mg/L	N-NH, mg/L
				Mode I				
Raw wastewater	1855.0	61.5	94.5	0.315	2.943	89.5	106.6	52.8
UF 50kDa	1273.0	39.7	66.5	0.23	2.94	20	63.5	47.5
NF 270	190.8	12.9	32.9	0.12	2.28	17.25	69	26.5
NF 90	**109.0**	**19.4**	**10.6**	**0.06**	**1.45**	**7.45**	**54**	**24.6**
TFC	240.0	34.0	6.1	0.02	1.42	37.2	21.4	39.4
				Mode II				
Raw wastewater	1608.0	64.5	93.0	0.231	1.96	90.5	107.5	60.5
UF 300kDa	1768.0	45.0	79.5	0.203	1.48	73.3	97.5	49.0
NF270	235.4	26.7	28.0	0.188	1.24	22.9	63.0	26.0
NF90	**175.5**	**19.6**	**26.5**	**0.043**	**1.15**	**10.15**	**66.0**	**30.6**
				Mode III				
Raw wastewater	1753.0	56.0	107	0.174	1.91	101	76.8	59.1
TFC	189.5	37.7	8.65	0.100	1.74	8.86	66.7	32.4

Table 5
Ions analysis after wastewater treatment – comparison

	Presented filtration results NF 90	**NF 90** [26]	**NF 270** [26]	**NF 90** [32]	**NF 270** [32]	**NF 90** [33]	**Irrigation water standards** [26]
COD	175.5	5.90	4.49	<5	6.87	na	na
Na^+	21.58	584	134	29	363	26.5	0-920
K^+	45.12	47.2	14.5	1.5	24	2.93	0.200
Ca^{2+}	0.07	57.5	218	6.7	41	4.73	0-400
Mg^{2+}	0.24	22.3	0.41	0.03	4.0	0.03	0-60.0

Table 6
Ions analysis

	Ca^{2+} mg/L	**Mg^{2+} mg/L**	**K^+ mg/L**	**Na^+ mg/L**	**SAR**
High-polluted wastewater	1.21	15.40	383.53	101.47	35.20
		Mode I			
UF50kDa	0.46	5.77	259.45	88.06	49.90
NF 90	**0.07**	**0.24**	**45.12**	**21.58**	54.63
NF 270	0.30	2.77	146.64	58.32	47.08
		Mode II			
UF 300kDa	1.13	6.47	248.17	79.17	40.59
NF 90	**0.31**	**1.48**	**80.24**	**26.39**	27.89
NF 270	0.69	2.45	155.10	58.90	47.00
		Mode III			
Low-polluted wastewater	1.00	1.23	383.53	122.47	116.16
UF 30 kDa	2.31	0.89	349.69	104.97	82.97
UF 5 kDa	0.31	0.41	338.41	94.18	157.31

4. Conclusions

The treatment of municipal wastewater from the middle agglomeration should be flexible as the composition of the water changes from time to time. It seems the membrane system with ultrafiltration on 5 kDa membranes can be applied for streams with low COD values. The processed water meets the regulation of the Ministry of Environment of Poland and can be discharged to the surface aquafers. However, this system is not effective enough to reduce the amount of total nitrogen and total phosphorus to the desired values. In the case of highly polluted wastewater, a combination of more membrane processes is needed. For such a medium, the use of ceramic ultrafilters followed by polymer nanofilters allows the reduction of sulfates, nitrate, and nitrite nitrogen to the permissible level. However, COD was sufficiently reduced for ceramic ultrafiltration membranes with a cut-off of 50 kDa. Unfortunately, none of the tested systems reduced phosphorus and ammonia to the recommended level. Hence, considering the composition changes of the wastewater, it is recommended to use a two-stage filtration system for the treatment of sewage from the middle-size region.

Abbreviations

CIP	Cleaning in place
COD	Chemical oxygen demand
N_{total}	The total amount of nitrogen
N-NO_2	Nitrogen nitrite amount
N-NO_3	Nitrogen nitrate amount
N-NH	Nitrogen ammonium amount
NF 90	Nanofiltration membrane, cut-off 200 Da
NF 270	Nanofiltration membrane, cut-off 400 Da
NFW	Nanofiltration membrane (NFW TFC), cut-off 300-500 Da
P-PO_4	Phosphorus contained in groups -PO_4
P_{total}	Total amount of phosphorus
SAR	Sodium adsorption ratio
SO_4^2	Sulfur amount
UF 30	Ultrafiltration membrane, cut-off 30 kda
UF 5	Ultrafiltration membrane, cut-off 5 kda

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K. Smolinska-Kempisty: Data curation; Roles/Writing - original draft; Formal analysis. Writing – review & editing. Methodology, Investigation

J. Wolska: Roles/Writing - original draft; Writing – review & editing, Investigation; Formal analysis; Methodology,

A. Urbanowska: Investigation, Methodology, Formal analysis; Roles/Writing - original draft, Writing – review & editing

B. Dach: Investigation, Methodology

D. Podstawczyk: Investigation, Methodology

A. Bastrzyk: Investigation

K. Czuba: Investigation

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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