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Regeneration methods of the ultrafiltration membranes applied in coke oven wastewater treatment

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ABSTRACT

The work aimed to determine the possibility of using an ultrafiltration membrane to pretreat the coke oven wastewater for the preparation of feed for the high-pressure process. The membrane selectivity is only one important parameter. The second is a possibility to carry out the process continuously that necessitates mechanical cleaning of the membrane during the process. Four different membranes with different pores size, material and producer were tested. The most effective rejection was observed for turbidity and iron compounds. For these compounds, the retention coefficient *R* for all tested membranes was in range 0.9–1.0. The minor rejection was observed for organic compounds (*R* was in the range 0.2–0.4), while the highest values were obtained for a PVDF membrane of Berghof with the biggest size of pores (0.03 μm). It suggests that pore size was not a dominating parameter during this separation. All tested membranes were prone to contamination. After about 30 min, the permeate flux was close to zero. Mechanical regeneration was necessary, and it was realized by backflushing, hydraulic flushing and sponge balls. Hydraulic flushing was the cheapest method, and an increase of linear velocity to 8 m s^{-1} for 30 s was enough for proper surface regeneration. Satisfactory results were obtained for CUT and Katmaj membranes. For Berghof membrane, backflushing allowed two times better renewal of its surface than hydraulic flushing. Sponge balls test with PCI membrane revealed to be the most effective (in 87%) method of regeneration. If a membrane diameter allows fitting sponge balls into it, this method is recommended at the coke oven wastewater treatment.

Keywords: Coke oven wastewater; Ultrafiltration; Backflushing; Sponge balls; Membrane regeneration

1. Introduction

Coke oven plants are a source of many hazardous substances generated during the coal coking process and processing of the final products. The size of the wastewater stream (30–120 m^3 h^{-1}), the diversity of components present and their instability over time are a problem for this wastewater treatment [1].

Before nitrification and denitrification processes, which are the main steps in the utilization of the organic compounds, coke oven wastewater should be pretreated. It can be done, for example, by coagulation with the natural or conventional coagulants [2] or by membrane processes. In the study by Wang et al. [3], coke oven wastewater was treated in two steps of membrane processes; by ultra- and nanofiltration. After nanofiltration, the chemical oxygen demand (COD) index, the concentrations of NH_4 and total hardness were reduced below 60, 2 and 30 mg L^{-1}, respectively. Among the organic components removed by

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nanofiltration, there are phenols [4], which can be biodegraded by adopted bacteria [5–7].

Depending on bioreactor efficiency, there may be a need for the output stream of coke oven wastewater to be treated post microbial treatment. It can be also done by membrane processes [8].

However, membrane techniques have not found a broader application in the treatment of coke oven wastewater because of their low efficiency caused by the decrease in permeate flux over time [9]. This decrease is caused by fouling, that is, compounds deposition on and into the membrane pores [10–13].

After some time, which can be determined only experimentally, it is necessary to regenerate the membrane surface [14,15]. Renewing the membrane surface brings the benefit of not only increasing the permeate flux but also extending the membrane's lifespan. At low-pressure filtrations (ΔP < 1.0 MPa), deposits on the surface are removed hydrodynamically and in special chemical baths dissolving deposits on the surface and in the pores of the membrane. The surface regeneration of membranes used in high-pressure filtration techniques is carried out only by chemical methods [16,17]. There is no data about fouling removal after coke oven wastewater filtration.

The membrane regeneration allows reducing the scale of the membrane installation. The tendency is to rescale the number of membrane modules due to maintaining the permeate intensity desired by the investor. The membrane regeneration is also connected with their lifespan directly related to the costs of technology [18].

1.1. Backflushing

Backwashing is the primary way to remove a blocking layer from porous membranes [19–22]. It is realized by a permeate pressure increasing above this in the retentate zone. In this way, the flow is achieved in the opposite direction. The permeate flowing through the pores detaches embedded particles and molecules and transfers them to the retentate. Forces that occur during backflushing can delaminate the active layer of the membrane. For this reason, only "open" membranes such as microfiltration membranes can be regenerated by this method. The type of material causing fouling also affects regeneration efficiency, for example, sticky layers that tend to gel will not be ripped off from the surface. Therefore, the membranes are still being modified by researchers to be less blocked and more accessible for regeneration by backflushing [23,24].

1.2. Sponge balls

Increasing hydrodynamics in membrane systems can be implemented by passing sponge balls of appropriately selected diameters through tubular membranes [25]. At a certain velocity, the balls cause vortex effects around the membrane. The problem with implementing this method is to create equipment that would regularly and continuously dispense balls into the system and capture them behind the modules so that they do not get into the pumps. The cost of the balls themselves is relatively small. This method is the least studied and for this reason, has few applications [26–28].

1.3. Hydraulic flushing

The essence of this technique is rapid increase in the flow velocity along the membrane, causing vortices to entrain particles or molecules accumulated at the surface [29]. It can be released in several ways, such as closing and opening valves in front of modules, or controlling feed streams in the system. Care should be taken not to exceed the critical linear velocity, which with its shear stress may damage the active layer of the membrane. The advantage of this method is the low cost of expanding the installation and performing the operation itself and no permeate losses.

The paper describes using low-pressure membrane techniques as pretreatment methods before high-pressure membrane processes aimed at water recovery from coke oven wastewater. This cascade combination of membrane processes is a standard technological solution [30,31]. The second part of the paper describes the methods of applied membranes regeneration to increase their lifespan.

In the literature, there are not so many papers describing membrane processes application in coke oven wastewater treatment. Zou and Li [32] described the ultrafiltration process as the last stage of purification. The effluent from the bioreactor to which the feed came from a coal gasification plant was pretreated by activated coke adsorption and sand filtration and then used as the feed of UF membrane device. The UF membrane module, hollow fibre type, was made of polysulfone with the molecular weight cut-off of 20 kDa. Kwiecińska et al. [33] to this aim used the membrane with a cut-off of 5, 10 and 20 kDa and the effect of separation of COD and cyanides was similar. In our research, we try to compare the membranes with bigger pores since cut-off of 20 kDa (to compared with literature data) till 0.03 μm.

2. Materials and methods

Coke oven wastewater was collected from one of the most modern coking plants in Poland. All batches of wastewater were collected after biological treatment and settling tanks.

2.1. Membrane modules

Filtration modules were created in self-production way with proper number of commercial tubular membranes inside. The modules were fabricated with PVC-U tubes, sealed with epoxy resin on both ends. Their parameters are presented in Table 1. Le Carbone Lorraine (France) installation (Fig. 1) allowed for testing them in pilot scale.

2.2. Filtration process

The filtration of real coke oven wastewater was tested for several parameters with transmembrane pressure in the range 0.1–0.3 MPa and linear velocity in the range 2–4 m s^{-1}. It was noticed that the linear velocity was a

Table 1
Parameters of membrane modules used in pilot-scale filtration

Type	Producer	Material	Cut-off	Diameter (mm)	Length (mm)	No of tubes	Area (m^2)	Cross section area (m^2)
UF	Burkert CUT	PES	50 kDa	8.0	795	3	0.059	5.03E-5
UF	PCI	PVDF	20 kDa	12.5	795	2	0.062	1.23E-4
UF	Katmaj	PVDF	500 kDa	12.5	795	2	0.062	1.23E-4
MF/UF	Berghof	PVDF	0.03 μm	8.0	950	13	0.310	5.03E-5

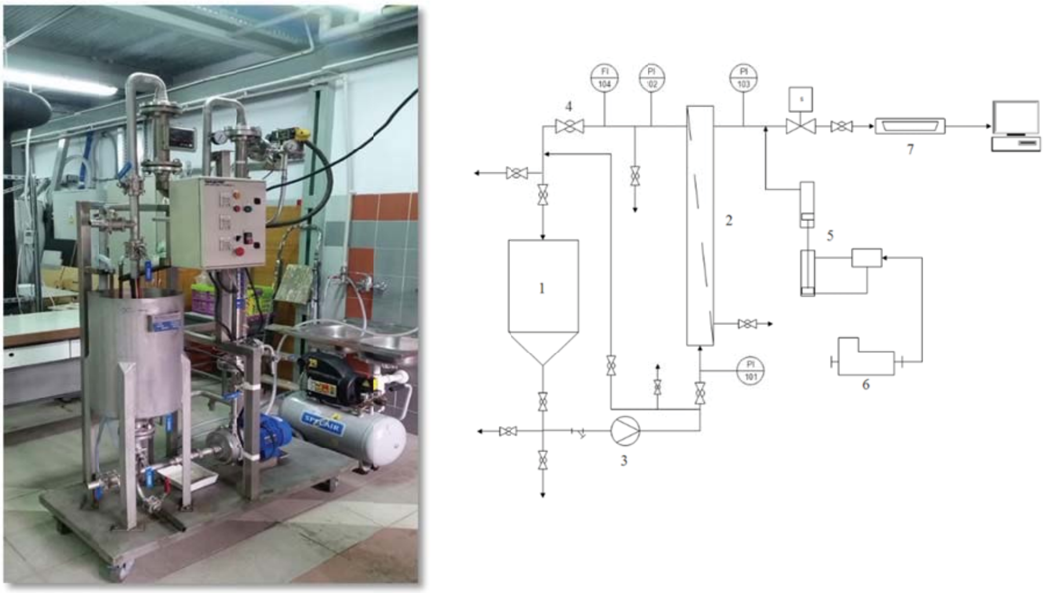

Fig. 1. Pilot-scale membrane installation used in the research: 1 – feed water tank, 2 – membrane module, 3 – recirculation pump, 4 – valve, 5 – backflushing system, 6 – compressor, 7 – weight.

more critical parameter than the transmembrane pressure, and it mainly determines the filtration efficiency.

The process selectivity was estimated on the base of selected parameters such as COD index, turbidity, colour compounds, calcium and iron ions concentration. All analyses were carried out following applicable procedures of Polish Standardization Committee presented in Table 2.

The procedure for COD analysis was based on the oxidation of organic and some non-organic compounds with potassium dichromate in sulfuric acid. The method was described in the study by Kolb et al. [34].

Nephelometric turbidity is an optical index for the side scattering of light caused by fine particles suspended in water. The method described in the study by Bright et al. [35] allows checking turbidity measurements affected by the organic and inorganic materials present.

Colour measurements have been made using values of indexes of transparency parameter [36]. The method following EN ISO 7887 standards was obtained by taking absorbance at 436, 525 and 620 nm.

Atomic absorption spectrometry was applied to measure calcium and iron concentrations [37].

2.3. Hydraulic flushing

Hydraulic flushing was realized by a speedy (up to 8 m s^{-1}) increase in linear velocity along the surface of the membranes. Rapid, alternating closing and opening of the

Table 2
Methods for selected parameters determination

Parameter	Kind of method	Equipment
COD	Titration method	Titrator Compact G20S, Mettler Toledo (Switzerland)
Turbidity	Nephelometric method	Turbidity Meter TB1000, Thermo Scientific (USA)
Colour	Spectrophotometric method, 436/525/620 nm	Spectrophotometer UV-1800, Shimadzu (Japan)
Calcium	Atomic Absorption Spectrometry, 422.7 nm	ASA ICE3000, Thermo Scientific (USA)
Iron	Atomic Absorption Spectrometry, 248.3 nm	ASA ICE3000, Thermo Scientific (USA)

valve in front of the membrane module was the most efficient method for creating turbulence along the membrane.

2.4. Backflushing

The system in the original Le Carbone Lorraine installation used a hydraulic cylinder to stepwise overpressure on the permeate side. It was needed during a membrane regeneration by backflushing. However, this method of generating pressure affected the membranes, causing partial delamination of the active layer. Thus, the installation has been modified to avoid a sudden increase in pressure. In place of the actuator, a membrane pump was used, powered by compressed air, taking permeate from a 20 L tank (Fig. 2). This type of membrane regeneration was applied only to Berghof membrane due to its "open" structure.

2.5. Sponge balls

For the PCI 20kDa membrane, the sponge balls fitted to the diameter of the membrane tube used. The balls were purchased in PCI Filtration Group (UK). The system diagram and view of the balls used are presented in Figs. 3 and 4, respectively.

Before starting the process, the sponge balls, shown in Fig. 3, were placed in a mesh filter. After passing along the membrane, the balls were caught with a second mesh filter, installed after the membrane module. The mesh filters allowed placing up to six sponge balls at once. Due to pump limitations, the sponge balls could reach a maximum velocity of 6 m s^{-1}.

3. Results and discussion

3.1. Membrane characteristic

Initial tests of all membrane modules were carried out using demineralized water as a filtration medium. The dependence of permeate flux on transmembrane pressure was tested for all membrane modules at 25°C and presented in Fig. 5. In the given range of parameters, all modules exhibited stable work. The highest values of permeate flux presented the Berghof membrane what was expected because it had the biggest pores.

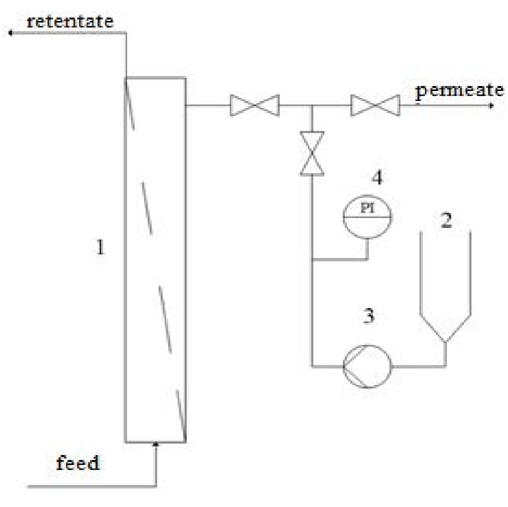

Fig. 2. Scheme of modified backflushing system, 1 – membrane module, 2 – permeate tank, 3 – membrane pump, 4 – gauge manometer.

3.2. Filtration efficiency

Coke oven wastewater characterized by a high content of components responsible for turbidity, salinity and COD

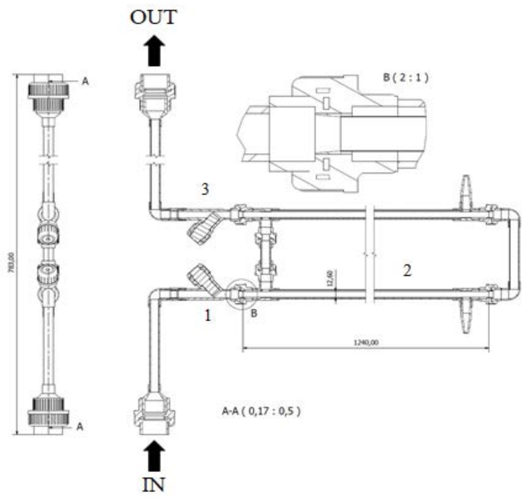

Fig. 3. Scheme of the sponge balls system, 1 – mesh filter, 2 – membrane module, 3 – second mesh filter.

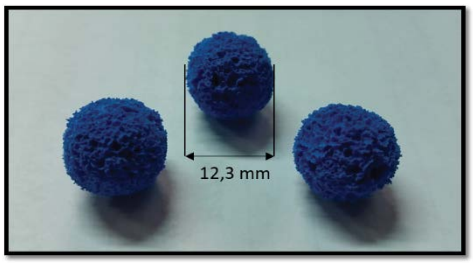

Fig. 4. View of the sponge balls.

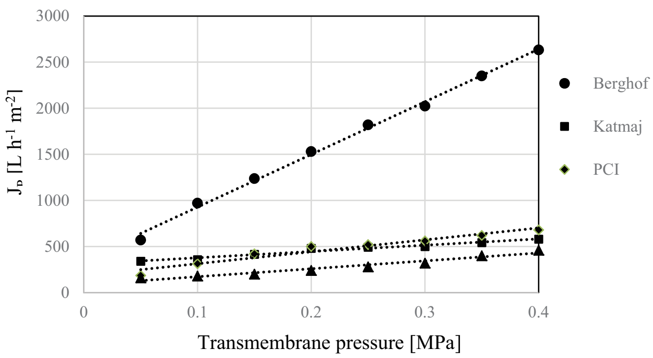

Fig. 5. Dependence of volumetric permeate flux on transmembrane pressure of used membranes.

index. The composition with the range and mean values are presented in Table 3.

The removal efficiency of selected compounds in the initial wastewater and filtrates collected in the pseudo-stationary state are presented in Fig. 6.

The highest membrane retention was obtained for colour, turbidity and iron ions. A high value for iron ions suggests that it is associated with turbidity. COD was retained to a relatively small extent. All of the tested membranes behaved similarly, but the highest values were obtained for the Berghof 0.03 μm membrane. Obtained permeate can be directed to high-pressure membrane installation [30].

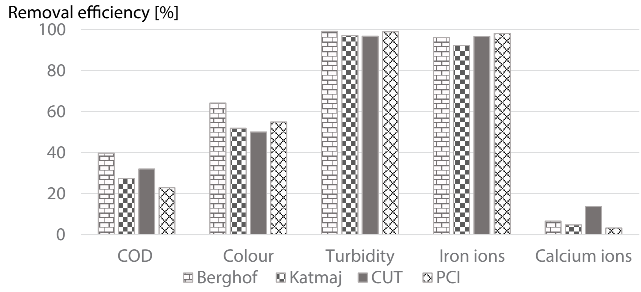

Fig. 6. Removal efficiency of selected compounds using four different membranes (T = 25°C, ΔP = 0.2 MPa).

Table 3
Average composition of the coke oven wastewater after biological treatment and sedimentation process (data obtained from the company)

Parameter	Range	Mean value
pH	8.2–8.5	8.4
Conductivity, mS cm^{-1}	11.3–12.1	11.7
Total nitrogen, mg N dm^{-3}	50.5	50.5
Kjedahl nitrogen, mg N dm^{-3}	39.1	39.1
Organic nitrogen, mg N_{org} dm^{-3}	6.4	6.4
Ammonia, mg N_{NH_4} dm^{-3}	0.7–32.7	16.7
Nitrate, mg N_{NO_3} dm^{-3}	10.6–25.4	18.0
Nitrite, mg N_{NO_2} dm^{-3}	0.8	0.8
Phosphates, mg P dm^{-3}	4.1	4.1
Total P, mg P dm^{-3}	4.2	4.2
Oxygen dissolved, mg O_2 dm^{-3}	8.9	8.9
BOD, mg O_2 dm^{-3}	8.3	8.3
COD (Mn), mg O_2 dm^{-3}	165.8	165.8
COD (Cr), mg O_2 dm^{-3}	398–795	617
Total hardness, mg $CaCO_3$ dm^{-3}	339.3–857	598.1
Alkalinity, mg $CaCO_3$ dm^{-3}	550–700	625
Turbidity, NTU	4.7–34.5	17.3
Sulphates, mg SO_4 dm^{-3}	1,663.0–1,780.5	1,721.8
Chlorides, mg Cl dm^{-3}	3,463–5,150	4,306.5
Sodium, mg Na dm^{-3}	200.4–233.7	217.0
Potassium, mg K dm^{-3}	8.9–11.6	10.3
Calcium, mg Ca dm^{-3}	28.0–78.7	53.4
Magnesium, mg Mg dm^{-3}	5.6–17.2	11.4
Manganese, mg Mn dm^{-3}	0.1	0.1
Iron, mg Fe dm^{-3}	1.9–5.1	3.5
Colour, mg Pt dm^{-3}	300–1,560	930
Total dry matter, mg dm^{-3}	7,730	7,730
Mineral dry matter, mg dm^{-3}	480	480
Organic dry matter, mg dm^{-3}	7,250	7,250
TDS, mg dm^{-3}	7,540	7,540
Mineral TDS, mg dm^{-3}	365	365
Organic TDS, mg dm^{-3}	7,175	7,175
Total suspension, mg dm^{-3}	190	190
Mineral suspension, mg dm^{-3}	115	115
Organic suspension, mg dm^{-3}	75	75

BOD: biological oxygen demand; COD: chemical oxygen demand; TDS: total dissolved solids.

3.3. Regeneration by hydraulic flushing

Fig. 7 shows the change in permeate flux obtained by hydraulic flushing realized every 1 h (the first after 2–3 h) of the process for 10 s. Linear velocity along the surface of the membranes was increased up to 8 m s^{-1}.

Membrane surface regeneration using hydraulic flushing was easy in realization. It was also the cheapest method. Nevertheless, the results in Fig. 7 showed that the membrane surface is only partially regenerated, that is, the initial values of permeate flux were not obtained. Extending the time of the increased linear velocity (till 2 min) did not bring additional benefits. Particularly satisfactory results were not obtained for Berghof and PCI membranes, that is why for them, other methods of surface regeneration were tested.

3.4. Regeneration of Berghof membrane by backflushing

Fig. 8 shows the change in permeate flux obtained for the Berghof PVDF microfiltration membrane. During the process, the membrane surface was regenerated with a backflushing technique every 1 h (the first time after 110 min) for 3 min.

The effect of each backflushing is visible as a jump in the permeate stream value. Nevertheless, the stream quickly fell to the pseudo-stationary state. The higher the pressure on the permeate side and the larger volume of liquid passed through the membrane, the better the degree of regeneration was obtained. The downside of this method is a loss of permeate of up to 30%.

3.5. Regeneration of PCI membrane with sponge balls

Fig. 9 shows the results of the regeneration of PCI membrane surface using sponge balls. From 1 to 6 balls were used at a linear velocity of 4 and 6 m s^{-1}. The duration of surface regeneration was 1 min.

It looks that the velocity of balls is more important than the number of balls. The manufacturer (PCI Filtration Group) recommends using a velocity of at least 3.5 m s^{-1} along

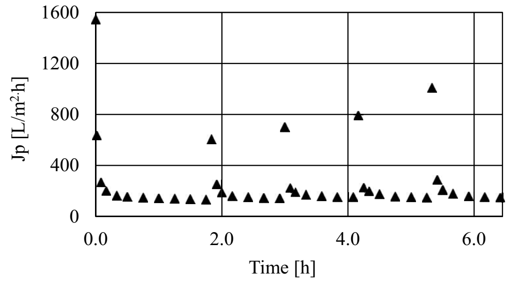

Fig. 8. Effects of Berghof membrane regeneration with a backflushing technique applied every 1 h for 3 min (ΔP = 0.2 MPa, w = 2 m s^{-1}, T = 25°C).

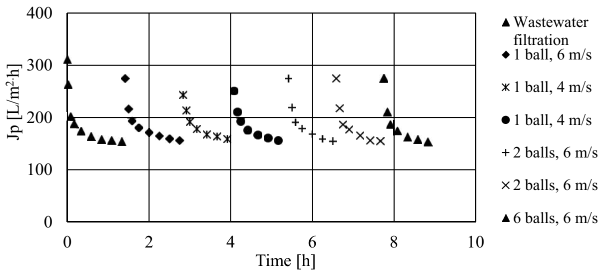

Fig. 9. Effects of PCI membrane regeneration by sponge balls for 1 min (ΔP = 0.2 MPa, w = 4 m s^{-1}, T = 25°C).

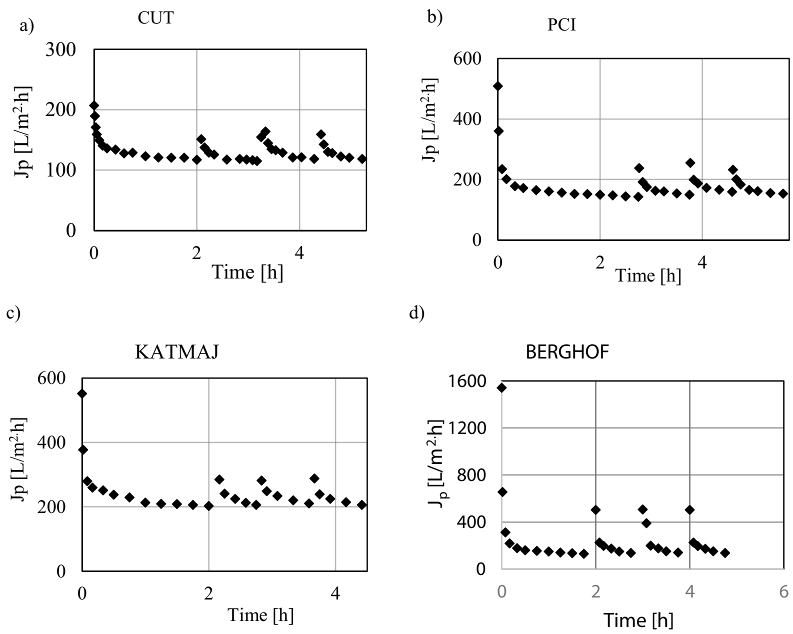

Fig. 7. Effects of the membrane regeneration by hydraulic flushing every 1 h (the first after 2–3 h) for 10 s (ΔP = 0.2 MPa, w = 4 m s^{-1}, T = 25°C); (a) CUT membrane, (b) PCI membrane, (c) Katmaj membrane, and (d) Berghof membrane.

Table 4
Cleaning degree of tested membrane

Membrane	Wastewater hydraulic flushing	Backflushing	Sponge balls
CUT 50 kDa	0.78	–	–
Katmaj 500 kDa	0.52	–	–
PCI 20 kDa	0.49	–	0.87
Berghof 0.03 μm	0.33	0.66	–

the membrane to generate the desired turbulence effect. At 6 m s^{-1}, the surface regeneration was the same for one, two and six balls.

Table 4 presents the degree of surface cleaning of the tested membranes, defined as a fraction of primal stream obtained for demineralized water filtration.

The degree of membrane surface cleaning with balls turned out to be relatively high (approx. 87% recovery of the initial stream). It indicates that the main impurities causing a decrease in permeate flux are localized on the surface of the membrane and not in its pores.

4. Summary and conclusions

The use of an ultrafiltration process allows pretreatment of coke oven wastewater. Four different membranes with different pore sizes were tested. It was surprising because the membrane with the biggest pores (0.03 μm) had the highest retention. Retention concerns mainly solids and substances responsible for turbidity and colour. Among the analysed chemical compounds, the highest retention relates to iron ions. The COD removal efficiency was in the range of 23%–40%. These are better results than obtained for the membranes with lower cut-off values [33].

The decrease in permeate flux was considerable in already the first minutes of filtration. A similar effect was observed by other researchers [32]. Intensive fouling is the main reason for the lack of application of membrane processes in coke oven wastewater treatment. Membrane blocking was similarly high, regardless of the kind of membrane, the material from which it was produced and its pore size.

Therefore, mechanical regeneration of the membrane during the filtration process is necessary. The above research has shown this possibility. The most common in membrane techniques is backflushing. However, hydraulic flushing is the cheapest method; for some of membranes, it was quite efficient. For the CUT (50 kDa) and Katmaj (500 kDa) membranes, it was possible to obtain the permeate stream above 50% of the initial value. Backflushing was a more efficient method than hydraulic flushing for Berghof membrane. Similarly, in the paper [37], it was shown that the initial deposit of particles could be removed easily by physical cleaning. The prolonged operation yielded irreversible clogging, which can be rectified by chemical cleaning.

The choice between hydraulic flushing and sponge balls comes down to analysing the costs of expanding the installation for sponge balls using. For the second method, the diameter of the membrane is an additional limiting factor. However, sponge balls have proved to be the most effective method of regeneration. If the membrane geometry allows it, this method is recommended at coke oven wastewater filtration.

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