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Removal of organic pollutants and surfactants from laundry wastewater in membrane bioreactor (MBR)

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ABSTRACT

This research carried out membrane bioreactor (MBR) pilot plant tests on the treatment of industrial laundry wastewater as well as making an analysis of MBR pilot plant operational conditions. The experiments were carried out in-place at the large-scale industrial laundry in Poland with usage of real wastewater. The MBR pilot plant used worked under aerobic conditions. The laundry wastewater, containing mainly surfactants and impurities originating from washed fabrics, was supplemented with a solution of urea to increase nitrogen content and a solution of acid to adjust pH. Daily flow of raw wastewater was equal to 0.25–0.5 m^3/d and hourly flow was equal to 13–25 L/h. The removal efficiency of organic pollutants, determined as 5-d biochemical oxygen demand and chemical oxygen demand, amounted to 95%–98% and 89%–94%, respectively; whereas in the case of other parameters, it was 32%–84% for total N, 55%–71% for total P, 94.5%–99.5% for anionic surfactants, and 98.8%–99.4% for nonionic surfactants. The quality of the purified wastewater meets the legal requirements regarding the standards for wastewater discharged to the environment. However, due to the hydraulic instability and short failure-free periods of membrane operation, the investigated system needs further optimization to be used for industrial laundry wastewater treatment plant.

Keywords: Laundry wastewater; Membrane bioreactor (MBR); Surfactants

1. Introduction

Chosen laundry is a large industrial laundry localized in Nowe Czarnowo, nearby Szczecin city (Poland). The laundry washes about 80 tons of linens and generates a daily average of about 600 m^3/d of industrial wastewater. The maximum wastewater flow is registered at a level of 800 m^3/d.

A significant part of industrial laundry running costs result from wastewater treatment and discharging. Continuous increases in the regulated discharging fee within communal sewage systems, as well as expansion of laundries, mean that there is a greater need to analyze the potential for wastewater management system improvements.

Laundry wastewater may be biodegradable, but this depends on the level of impurities; which is, in turn, related to the type of washing [1]. To analyze the removal of surfactant, as a specific laundry pollutant, membrane bioreactor (MBR) wastewater treatment technologies were tested. MBR technology is a combination of the conventional biological sludge process, combined with a microfiltration (MF)

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or ultrafiltration (UF) membrane system [2]. For treatment of laundry wastewater containing surfactants, both aerobic [3–8] and anaerobic [9] membrane reactors were used. Nicolaidis and Vyrides [3] achieved 99% efficiency in the removal of turbidity as well as total solids and 70%–99% efficiency in the removal of chemical oxygen demand (COD) in a full-scale submerged aerobic MBR (9 m^3) used for laundry wastewater treatment [3]. The MBR pilot plant, with submerged plate and frame MF membranes as the principal treating unit, has been successfully tested over a period of 5 years in a laundry located in Darmstadt, Germany. The average COD and total organic carbon removal efficiency in the MBR reactor with immersed Kubota membranes was higher than 90%. Part of the MBR permeate is subsequently treated using reverse osmosis filtration [4,5]. In another research study, a submerged MBR with a cross-flow MF membrane (pore nominal diameter of 0.4 μm) was used. The treatment of an anionic surfactant-rich wastewater by a powerful *Citrobacter braakii* strain was investigated in a large-scale MBR [6]. The anionic surfactant concentration in the permeate varied from 0 to 40 mg/L. After 2 months of running, the permeate flux slightly decreased. González et al. [7] reported treatment of wastewater containing anionic and nonionic surfactants using different methods. Higher efficiency was obtained when a pilot plant MBR was used in comparison with a full-scale wastewater treatment plant (WWTP) with a conventional activated sludge system [7]. MBR technology was also used for the treatment of sludge obtained in the textile industry [8]. An MF membrane with a nominal pore diameter of 0.2 μm was used. The COD removal efficiency was 92%, and total suspended solids removal efficiency was 99.5%. Treatment of wastewater polluted by anionic surfactant (linear alkylbenzene sulfonate [LAS]) in a submerged anaerobic MBR was investigated in a 243-d operation. Addition of LAS decreased COD removal and biogas production rate [9].

Along with treatment efficiency, membrane fouling and water flux decline are very important issues in MBR application. This is the main limitation in the use of full-scale reactors [2,10]. A fouled membrane needs chemical cleaning, which increases the cost of reactor maintenance. The frequency of chemical cleaning depends on the feed pretreatment processes used, and these are mainly concerned with the removal of suspended solids. Hoinkis and Panten [4] and Hoinkis et al. [5] used sieve and screen with a mesh size of 200 μm while Nicolaidis and Vyrides [3] used parabolic fine screen mesh of size 1.0 mm [3]. Sieving followed by coagulation–flocculation and clarification were used by Lubello et al. [8]. Alternatively, low-fouling membranes were proposed by Deowan et al. [11] and modificated with TiO_2 membrane proposed by Szwast and Polak [12].

The aim of the research described in this paper was the evaluation of the performance of MBR during treatment of real industrial laundry wastewater, as well as the analysis of the MBR pilot plant operational conditions. A pilot-scale test was carried out in-situ in the laundry with the usage of real laundry wastewater.

2. Materials and methods

The research was conducted in a period from October to December in 2015. During the investigations, a pilot-scale MBR WWTP (Veolia), fed with the real laundry wastewater, was used.

2.1. Materials and equipment

The pilot plant was continuously fed with wastewater pumped from a 350 m^3-volume equalization tank situated in the laundry. The equalization tank also collects wastewater from the regeneration processes of ion exchangers used for softening of the water. The contribution of that wastewater, which is polluted mainly by chlorides, is at a level of a few percent of total wastewater volume. The wastewater has been mechanically pretreated on a sieve and cooled down to the temperature of about 40°C. The washing processes in the laundry involve wet washing at temperatures to a maximum of 90°C. The wastewater is mainly polluted by the impurities washed out from linens as well as washing and auxiliary agents. Surfactants used in the laundry fulfill the criteria for biodegradation in the European regulation on detergents (Regulation EC no. 648/2004) [13]. The values of raw wastewater quality indicators are presented in Table 1.

The MBR pilot plant applied in the research consisted of biological reactors (three aerated chambers, installed in series, 200 L each), a 30-L separated membrane reactor and

Table 1
Quality of the mechanically pretreated laundry raw wastewater (sampling period: 10.2015–12.2015)

No.	Parameter/indicator	Allowable limit	Value	
			Maximum	Minimum
1	pH	6.5–9.5	8.7	7.6
2	Total suspended solids (TSS), mg/L	35	180	140
3	Chlorides, mg P/L	1,000	510	375
4	Total phosphorus, mg N/L	2	8.47	4.20
5	Total nitrogen, mg/L	30	20.00	8.75
6	Anionic surfactants, mg/L	5	8.96[a]	7.00[a]
7	Nonionic surfactants, mg/L	10	65.60[a]	53.60[a]

[a]Result according to the standard methods of surfactants determination (anionic surfactants EN 903 standard: determination of anionic surfactants by measurement of the methylene blue index MBAS; ISO 7875-2 standard: determination of non-ionic surfactants using Dragendorff reagent) [16].

auxiliary reagents dosing station. Raw wastewater flowed into a biological reactor (hourly flow 13–25 L/h, daily flow 0.25–0.5 m^3/d) was then pumped to the membrane reactor (125 L/h). The scheme for the used MBR pilot plant is presented in Fig. 1.

The polyvinylidene difluoride UF membrane with nominal membrane surface area of 0.93 m^2, nominal pore size of 0.04 μm, and outer/inner fiber diameter of 1.9 mm/0.8 mm (ZeeWeed 10 membrane module, GE Water & Process Technologies) was installed in the membrane reactor. The reactor worked in filtration, relaxation, and back-pulsing phases. The filtration cycle lasted for 350 s with air scouring and transmembrane pressure equal to approximately –0.05 bar. The relaxation phase lasted for 90 s and was followed by a 5 s of back-pulsing (with permeate).

The wastewater plant inflow was controlled by wastewater level in the bioreactor chambers. In the filtration phase, the outflow of purified wastewater was balanced by the raw wastewater inflow to maintain a steady level of wastewater in the bioreactors chambers. An internal recirculation of 25–50 L/h from the third to the first chamber of the biological reactor was applied, resulting in wastewater mixing in the chambers of the bioreactor. The excess sludge was periodically removed from the plant. The wastewater plant hydraulic retention time was equal at 12–24 h. The organic loading rate was equal at 0.625–0.958 kg COD_{Cr}/m^3/d.

The source of an acclimated activated sludge, used for starting up of the pilot plant (600 L of biological chambers in total volume), was a municipal WWTP in Gryfino (Poland). The sludge used for inoculation originated from WWTP which is working with a mixture of municipal sewage and the investigated laundry wastewater. The sludge (160 L) containing ca. 15 g/L of dry mass was taken from the WWTP thickener and then it was sieved (1 mm sieve). After the starting-up period of the pilot MBR, the mixed liquor suspended solids were maintained at a level of 7–11 kg/m^3.

To improve efficiency of a biological treatment, the urea, as a nitrogen source, was dosed to the wastewater. A commercial product Ad-blue™ solution (32.5% by mass of urea content), containing 165.3 g/L of nitrogen, was used. The required dose of nitrogen was equal to 5 mgN/L, which is related to the added Ad-blue™ flow equal to 2 mL/min.

The pH of raw wastewater varied from 7.6 to 8.7 and, because of technological requirements concerning membrane, the pH was maintained at 7.7. The pH adjustment was achieved by automated dosing of 10% solution of H_2SO_4. The pH sensor was placed in the biological reactor, and 10 mL/min of acid was dosed until the pH dropped below 7.7. Moreover, antifoaming agent was added periodically to the wastewater.

Dissolved oxygen (DO) concentration was maintained at a level of 2–5 mgO_2/L. The DO sensor was immersed in the third bioreactor chamber, and the signal from this was used to switch on the air blower when DO dropped below 1 mgO_2/L.

2.2. Analytical methods

The pilot plant working conditions were controlled by the on-line measurements of raw wastewater flow, bioreactor chambers wastewater level, pH, temperature, transmembrane flow and pressure as well as DO concentration.

The wastewater pH was measured using the HI 991300 portable pH/EC/TDS/Temperature meter (Hanna Instruments, Olsztyn).

Oxidizable substances were determined as COD by dichromate method (ISO 6060 standard). The sample of wastewater was added to sulfuric acid–potassium dichromate solution in the presence of silver sulfate as a catalyst. The addition of mercury sulfate masked chloride. The remaining potassium dichromate was titrated with acidified Mohr's salt solution using ferroine as an indicator.

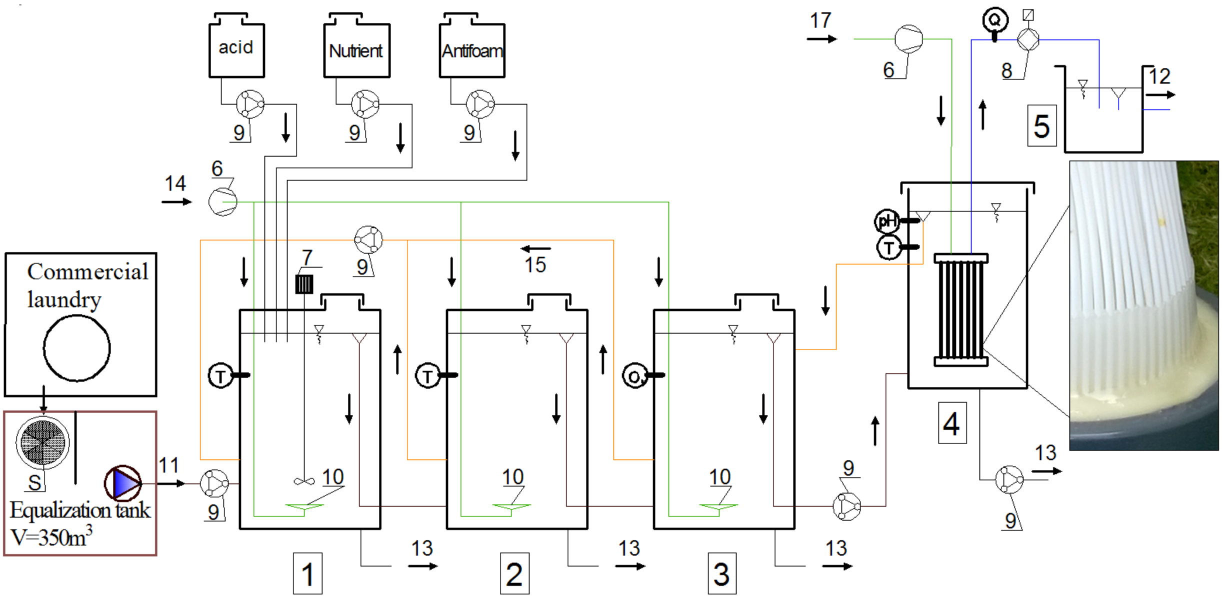

Fig. 1. The technological scheme of the MBR: (1)–(3) aerobic bioreactor, (4) membrane module, (5) permeate tank, (6) blower, (7) mixer, (8) vacuum pump, (9) peristaltic pump, (10) membrane diffuser, (11) raw wastewater inflow, (12) permeate outflow, (13) sludge outflow, (14) air, (15) recirculation of sludge, and (S) sieve.

Biochemical oxygen demand (BOD_5) was measured as the pressure difference within a closed system (respirometric BOD–Lovibond BOD-System Oxidirect).

Total nitrogen was determined using the Kjeldahl nitrogen method. This method is based on transformation of nitrogen compounds into ammonium sulfate in the process of sample mineralization with sulfuric acid with addition of potassium sulfate and selenium as a catalyst. Ammonia was released from ammonium sulfate by the addition of sodium hydroxide, and distillation to a solution of boric acid/indicator and then the determination of ammonium ion was carried out by titration with hydrochloric acid (EN 25663 standard).

Total phosphorus was determined by the ammonium molybdate spectrometric method (ISO 6878 standard). A sample was mineralized, in a Kjeldahl flask, with sulfuric acid and then with nitric acids. After cooling down, the content of the flask was neutralized with sodium hydroxide solution to pH of 3–10. Phosphate ions reacted with molybdate and antimony ions in an acidic solution to form an antimony–phospho-molybdate complex, which was reduced by ascorbic acid to phosphomolybdenum blue. Content of phosphate was measured photometrically at the wavelength of 880 nm (Spectroquant Pharo 300; Lambda 20, PerkinElmer, Krakow).

Anionic surfactants were determined as a methylene blue index (MBAS) according to the EN 903 standard as well as by the cuvette test LCK332 (Hach-Lange, Wrocław). Both methods are based on the reaction of anionic surfactants with methylene blue to form complexes, which are extracted in chloroform and evaluated photometrically. However, when using the standard method, surface-active agents are concentrated and isolated by gas stripping and the stripped surfactant is dissolved in ethyl acetate. To eliminate interference, the extraction is first effected from alkaline solution, and the extract is then shaken with acidic methylene blue solution. Both concentrating and interference eliminating is omitted in the simplified LCK332 method.

Nonionic surfactants were measured according to the ISO 7875-2 standard using Dragendorff reagent and by LCK333, the non-ionic surfactants cuvette test (Hach-Lange). Photometric determination was based on the tetrabromophenolphthalein ethyl ester (TBPE) method. Nonionic surfactants (ethoxylates with 3–20 ether bridges) react with the indicator TBPE to form complexes, which are extracted in dichloromethane and photometrically evaluated. The standard method, applicable to non-ionic surfactants containing 6–30 alkylene oxide groups, uses gas stripping for surface-active agent concentrations. The stripped surfactant is dissolved in ethyl acetate. After phase separation and evaporation of the solvent, the non-ionic surfactant is precipitated in aqueous solution with modified Dragendorff reagent ($KBiI^{4+}$ $BaCl^{2+}$ glacial acetic acid). The precipitate is filtered, washed with glacial acetic acid and dissolved in ammonium tartrate solution. The bismuth in the solution was titrated potentiometrically (Titrator TitroLine alpha plus, Schott Instruments, Germany) with pyrrolidinedithiocarbamate solution at pH 4–5 using a bright platinum indicator electrode and a silver/silver chloride reference electrode.

Filtration through glass fiber filters, according to the EN-872 standard was used for suspended solids determination. A sample was filtrated trough glass fiber filter on vacuum filtration apparatus. The filter was then dried at a temperature of 105°C and the mass of retained solids was determined by gravimetric method.

3. Discussion of results

3.1. Raw laundry wastewater quality

The values of the raw wastewater quality indicators including BOD_5, COD_{Cr}, anionic and nonionic surfactants (determined with standard photometric methods), conductivity and turbidity were presented in the previous paper [14]. Weekly measurements had been carried out during a 46-week period, including 11 weeks of MBR pilot plant testing. The determined wastewater quality indicators values varied in the following ranges [14]: from 368 to 626 mgO_2/L for BOD_5, from 750 to 1 150 mgO_2/L for COD_{Cr}, from 1.69 to 2.13 for COD_{Cr}/BOD_5 ratio, from 22.6 to 50.8 mg/L for anionic surfactants, and from 14.6 to 63.9 mg/L for nonionic surfactants.

In addition to the above presented results, the standard methods for anionic and nonionic surfactants determination have been used. The EN 903 standard: determination of anionic surfactants by measurement of the methylene blue index MBAS and ISO 7875-2 standard: determination of nonionic surfactants using Dragendorff reagent was applied. The content of the total nitrogen and total phosphorus were also determined. The quality of the raw (pretreated) wastewater was determined at the inlet to the first biological chamber of MBR pilot plant. The values of those wastewater quality parameters are presented in Table 1.

At the time of MBR pilot plant test, the ratio of BOD_5 to COD_{Cr} (COD_{Cr}/BOD_5) ranged from 1.9 to 2.8. The ratio of BOD_5 to total nitrogen and total phosphorus (BOD_5/N/P) was also calculated. Based on the subject literature, the value of that ratio required for proper wastewater biological treatment should be equal to 100/5/1 [15]. The median of the BOD_5/N/P ratio in the case of the examined laundry wastewater was equal to 100/2.21/1.15, which indicates that there was a deficiency of nitrogen.

3.2. The MBR pilot plant operational conditions

In order to analyze the plant working conditions, the basic parameters (wastewater flow rates, pH, temperature, DO, transmembrane pressure) were registered online. The variability of these parameters, in the period from 1 October to 10 December 2015, is presented in Fig. 2. The MBR pilot plant started on 18 September 2015. In Fig. 2 the sampling dates (green line) as well as the emergency periods (breaks in proper plant functioning—red line) are highlighted and the variability of the four parameters is shown (wastewater flow, pH, temperature, DO).

The emergencies (a1–a4) were caused by membrane fouling which was visible as a drop in purified wastewater flow rate and transmembrane pressure increase. In such cases, chemical cleaning of the membrane was applied. After an emergency was identified the cleaning in place (CIP), according to the built-in procedure was performed. The CIP was based on alternate usage of citric acid and sodium hypochlorite solutions. CIP was used in a2 and a4 emergencies. In the case that CIP was not effective in the recovery of the assumed membrane permeability, laboratory chemical cleaning (LCC), by immersing the membrane alternately in the solutions of

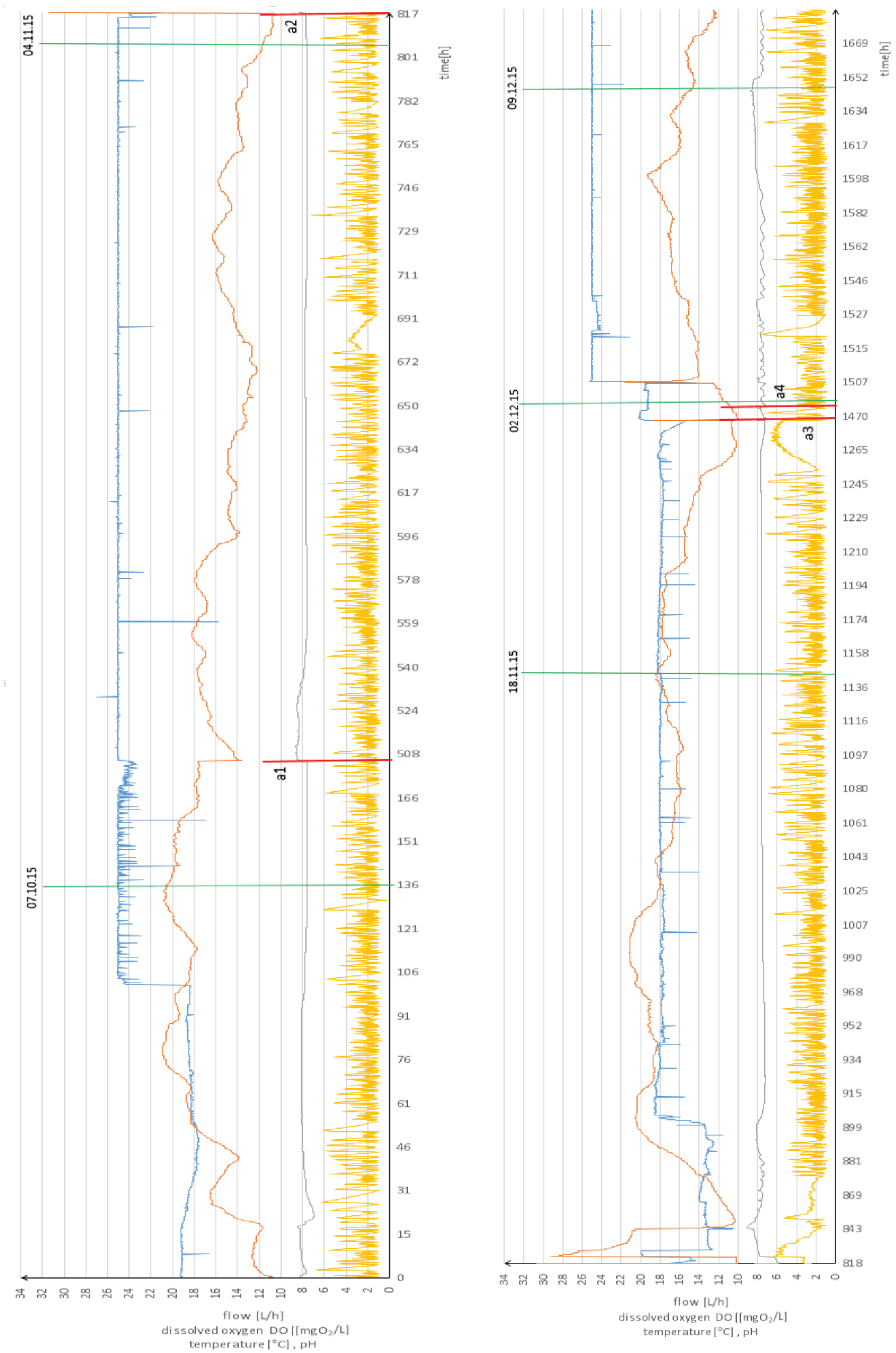

Fig. 2. MBR plant: —, flow (L/h); —, temperature (°C); —, pH; —, dissolved oxygen (DO) (mgO_2/L).

citric acid, sodium hypochlorite, alkaline solution (NaOH), P3-Ultrasil 11 solution, was applied. The chemical cleaning procedure and its efficiency has been described elsewhere [14]. LCC was used in a1 and a3 emergencies. Compilation of data on MBBR running time periods and emergencies is presented in Table 2.

MBR pilot plant hydraulic flow rate was disturbed by membrane fouling. The application of the LCC as well as the CIP allowed for maximum 31 d of undisturbed pilot plant operation. In fact the inflow rate equal to ca. 13 L/h has been maintained only directly after emergency a2 and lasted for less than 4 d (6.4% of total test time). Also at the starting-up phase, lasting for about 4 d, the inflow rate was lowered to ca. 18 L/h. Excluding these events, and other described emergencies, time-weighted average inflow rate was equal to about 23 L/h. In the time of stable operation, inflow rate was equal to 25 L/h and, for a few days, 18–20 L/h which gives daily inflow rate equal to 0.4 or 0.5 m^3/d.

3.3. Quality of the treated laundry wastewater

After 20 d of pilot plant operation, despite the hydraulic instability, the samples of the treated and untreated wastewater were collected periodically. The aim of analyzes was used to check whether the quality of treated wastewater meets the legal requirements to be discharged to the environment. Determined parameters depend on the functioning of activated sludge microorganisms, which prefer stable operational condition and needs some time to accommodate to the changing condition. Because the treated wastewater was sampled in the time periods not shorter than seven days and after, stable hydraulic operation of MBR pilot plant lasted for at least a few days. Taking into account given limitations the treated wastewater was sampled five times.

In Fig. 3, the values of COD_{Cr} and BOD_5 of the treated wastewater are presented. The acceptable aw values of the measured parameters should be [17] BOD_5 < 25 mg/L and COD_{Cr} < 125 mg/L.

After 3 weeks of MBR initialization, the values of BOD_5 (2–5 mg/L) and COD_{Cr} (38–77 mg/L) were within the allowable limits. It was observed that stopping the raw wastewater feeding pump caused by break of the membrane reactor operation had no significant influence on the performance of biological processes, and after membrane was cleaned and put back into operation the biological processes started work with satisfactory efficiency. It means that maintaining the aerobic conditions and temperature above 6°C was enough for biological system to survive despite the periodical lack of raw wastewater inflow to the MBR pilot plant.

In Fig. 4, the concentration of surfactants in raw and treated wastewater is presented.

Observed significant differences between concentration of anionic and nonionic surfactants measured by photometric and standard methods may be explained by differences in used determination procedures which are described in analytical methods section. Both air stripping and elimination of interfering substances are omitted in simplified photometric methods which could lead to higher, in relation to standard methods, results of surfactants determination. According to the previously published determination results [16],

Table 2
Compilation of data on MBR running time periods and emergencies

No	Running hours	Pilot plant operation cycle	Time (d)	Flow rate (L/h)	Remarks
1	0–101	Run	4.2	18–19	(Start-up)
2	102	Run		25	CIP was applied
3	103–178	Run	3	25	
4	179	Emergency a1		0	TMP reached 0.5 bar. The flow through membrane module was blocked.
5	179–505	Shut-off	13.6	0	LCC was applied
6	505–818	Run	13.0	21–25	
7	818	Emergency a2		21	TMP rise, flow was blocked
8	818–842	Shut-off	1	0	CIP was applied
9	842–904	Run	18.2	13–17	Start-up after Emergency a2
	905–1,278			18–19	Gradual rise in wastewater temperature was observed (from 8°C to 20°C). When temperature reached 20°C wastewater flow rate reached 18–19 L/h.
10	1,279	Emergency a3		0	TMP reached 0.5 bar. The flow through membrane module was blocked.
11	1,280–1,460	Shut-off	7.6	0	LCC was applied
12	1,461–1,484	Run	1	19–20	
13	1,485	Emergency a4		0	TMP reached 0.3 bar. The flow through membrane module was blocked.
14	1,485–1,506	Shut-off	0.9	0	CIP was applied
15	1,507–1,687	Run	7.5	25	

TMP, transmembrane pressure.

the significant differences in the results of photometric and standard methods were noticed for wastewater sampling lasting for a few months.

In Fig. 5, the COD_{Cr}, BOD_5, surfactants, total N and P removal efficiency are presented. The efficiency of BOD_5 removal amounted to 98%–99%, COD_{Cr} 90%–95%, total N 32%–84%, total P 55%–71%, anionic surfactants 94.5%–99.5%, and nonionic surfactants 98.8%–99.4%. The efficiency of the laundry wastewater treatment was very high and the obtained parameters were far below the limits described in the regulations [17].

Real industrial laundry wastewater treatment in a full-scale submerged aerobic MBR is described by Nicolaidis and Vyrides [3]. COD_{Cr} effluent removal efficiencies were between 70% and 99%, and COD_{Cr} levels were below 100 mg/L [3]. In this study, the COD_{Cr} effluent removal efficiencies were between 89% and 94%. However, the research revealed some problems due to hydraulic instability and short failure-free periods of membrane operation. The chemical membrane cleaning should be carried out frequently, because of membrane fouling. After membrane examination, it was concluded that the main factor responsible for the membrane fouling was the presence of fibers originating from the laundry. Therefore, an additional pretreatment step, such as filtration through microsieves, was found to be necessary [3–5].

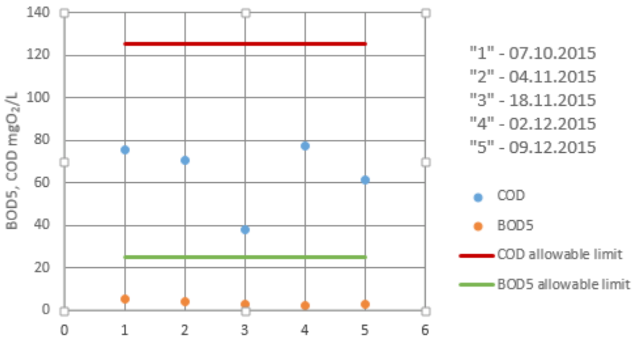

Fig. 3. COD and BOD_5 of the treated wastewater.

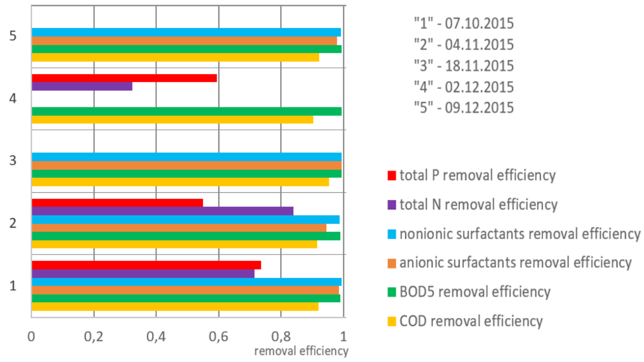

Fig. 5. COD, BOD_5, surfactants, total N, and total P removal efficiency of laundry wastewater.

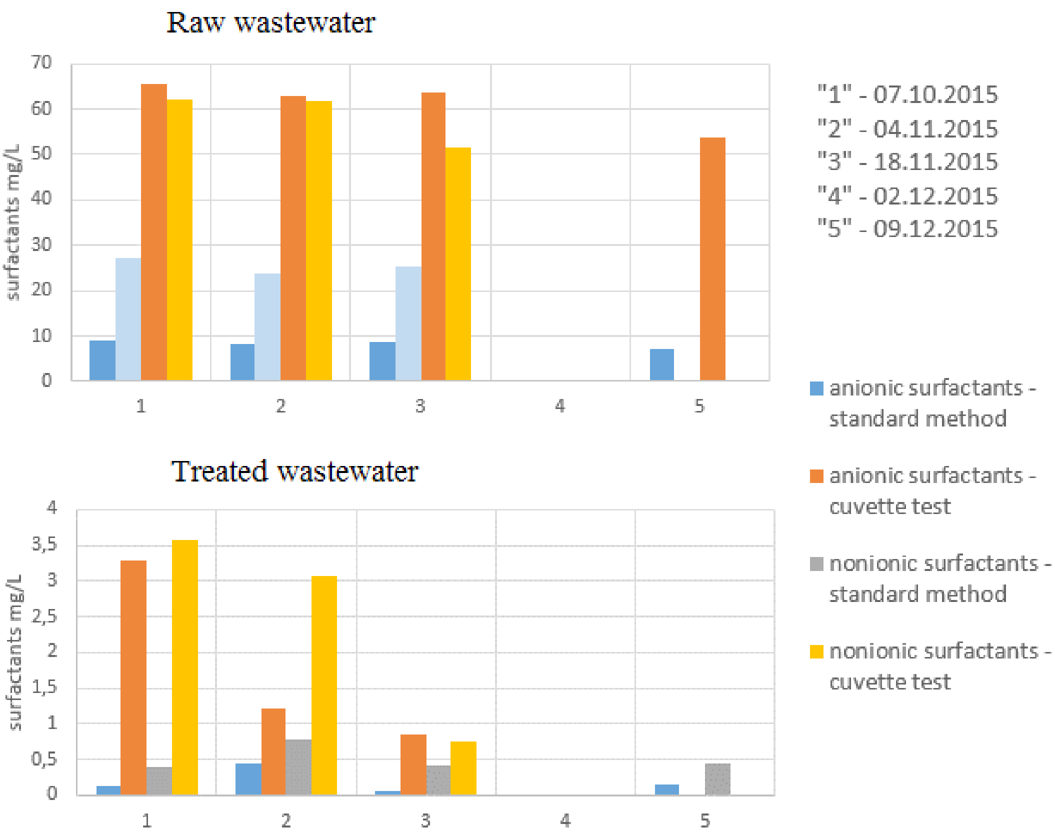

Fig. 4. Anionic and nonionic surfactants in raw and treated wastewater.

4. Conclusions

- Application of MBR technology for Fliegel Textilservice laundry wastewater treatment gives promising results. The quality of the treated wastewater is in accordance with the obligatory regulations [17]. Removal of nitrogen is not required.
- Removal efficiency of organic pollutants, determined as BOD_5 and COD_{Cr}, was equal to 95%–98% and 89%–94%, respectively. In case of other parameters, the treatment efficiency was 32%–84% for total N, 55%–71% for total P, 94.5%–99.5% for anionic surfactants, and 98.8%–99.4% for nonionic surfactants.
- Before a possible application in the investigated laundry, the MBR technology needs further optimization.

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