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Novel membranes for industrial laundry wastewater treatment

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ABSTRACT

This work focuses on the development of new polymeric membranes with hydrophilic properties for use in industrial laundry wastewater treatment. Composite heterogeneous membranes containing polymer matrix and organic and inorganic fillers were prepared. Different fillers were sought that would improve the wettability of the membrane surface. Based on the literature data, both inorganic compounds–metal oxide nanoparticles (ZnO, ZrO_2, Al_2O_3) as well as organic compounds such as polyacrylic acid–PAA and polyethylene oxide–PEO were selected. Process tests were carried out using modified membranes and the contact angle of the developed materials was measured as well. The results show that for the membranes modified with ZrO_2 or PEO the obtained permeate volumetric flow was higher than for the unmodified membranes. This is due to the improvement of the hydrophilic properties of the modified membranes, which is confirmed by the reduction of the contact angle for new materials. The formation of a new thin layer on the surface of the support membrane has also been confirmed in microscopic photographs.

Keywords: Laundry wastewater treatment; Microfiltration; Modified membrane; Flow coating

1. Introduction

One of the industries where it is necessary to introduce a new technology is commercial laundries. It is related to the high consumption of water and detergents. The new technology will allow us to reduce the consumption of these products and energy as well as to decrease the negative impact on the environment by partly closing the circulation of detergents. The recovered water and detergents could be reused in the washing process.

The laundry wastewater contains solids (fibers, fabric residues), salts (nitrates, nitrites, phosphates, fluorides, bromides, chlorides), dyes, bacteria, bleaches (sodium hypochlorite, hydrogen peroxide), plasticizers as well as anionic and non-ionic surfactants [1]. The complex composition makes it difficult to choose a treatment technique that will make the recovery of water and detergents possible. The solution for this type of wastewater and the feed stream composition could be microfiltration. The range of membrane pores diameter makes it possible to transport the detergents. Besides, the microfiltration membrane retains solids, some insoluble salts, some proteins, fats, and bacteria [2]. The high content of insoluble particles influences membrane fouling, which decreases the process efficiency. To reduce the intensity of this phenomenon, a new hydrophilic membrane has been developed.

The aim of this work is to develop a new membrane for laundry wastewater treatment. The development of the new membrane will improve the efficiency of water and detergents recovery process from laundry wastewater.

2. Fouling and antifouling strategies

The efficiency of the microfiltration process depends on the type of membranes used. In many cases, it is necessary to develop a new membrane because commercial membranes

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have disadvantages that decrease the process efficiency. The specific physicochemical parameters of laundry wastewater make it difficult to use commercial membranes. It leads to an intensive fouling phenomenon.

The fouling phenomenon is related to the attachment, accumulation, or adsorption of materials on the membrane surface, in the pore of the membrane or both places. The intensity of this effect depends on [3,4]: feed properties: concentration, pH and ionic strength, interactions between particles in the feed stream and on the membrane surface, membrane structure: pore size, porosity, and pore size distribution physicochemical membrane properties: wettability, surface free energy, electric charge, and roughness, operating condition: transmembrane pressure, flow-cross velocity, temperature.

Fouling influences the increasing resistance of membranes, resulting in a decrease in the permeate volumetric flow and a change in the permeate quality. Additionally, in the case of laundry wastewater, the created cake layer influences the amount of recovered detergents. In the cake layer on the membrane, surfactant concentration increases and the critical micelle concentration could be reached. Micelles are created and the transport of surfactants through the membranes is reduced.

The fouling phenomena could be limited by using different methods. These methods could be divided into two groups: avoidance and remediation. One of the basic methods is dividing the microfiltration process into the working and cleaning stages. Membranes could be cleaned by using hydraulic/mechanical techniques (cross–flushing, back–flushing) or by physicochemical cleaning, in which cleaning agents such as acids, base, oxidizing agents, surfactants, detergents, or enzymes are used. Fouling intensity could also be reduced by using flow velocity higher than 2 m/s; it ensures good shear force. Additionally, the microfiltration should be conducted at the maximum acceptable temperature. In this way, the viscosity of the fluid is decreased, so the membrane total resistance is lower [5,6]. Another way is to change the surface properties of the membrane during the production stage or by surface modification procedure. The aim of these methods is to change the physicochemical properties of the membrane surface, such as wettability, surface free energy, electric charge, and roughness. Modification of the structure might involve covering the surface with additional material or attaching specific chemical groups to it. The modification of the membrane surface is based on surface coating or surface grafting methods including plasma treatment, UV grafting, organic reaction, chemical vapor deposition or polymeric grafting [7,8]. The basic type of surface modification is to improve the hydrophilic properties of the membrane. In many works, the dependence between the increase in hydrophilic properties and the decrease in fouling intensity has been proved. This effect is the result of two mechanisms. With the decrease in contact angle (increasing wettability), the interactions between the membrane surface and foulants are lower, particularly in the case of fats and proteins. Additionally, the water layer could be formed on the hydrophilic surface. This is the result of creating hydrogen bonding (in case of polymers) or strong electrostatic interactions (in case of nanomaterials) between the used particles and water molecules [9,10]. The water layer limits the accumulation of pollutants on the surface and makes it easier to clean the membrane (self-cleaning surface). Additionally, in some cases, it is possible to improve the water permeation in this way. For the membrane modification, the following materials could be used: carbon materials (graphene oxide, acid oxidized multiwalled carbon nanotubes) [11,12], polymers (polyacrylic acid, Polyethylene oxide) [13,14], metal oxides (SiO_2, TiO_2, Fe_3O_4, Al_2O_3, ZrO_2, ZnO) [15,16] or zeolites [17].

2.1. Coating method

The coating method involves covering the solid surface with one or many layers of new material. The new layer could consist of one or more components. The purpose of the covering is to improve the existing properties or to give new properties such as hydrophobic/hydrophilic, antibacterial, antifouling, anti-corrosive properties or to improve the mechanical/chemical strength. For membrane modification, the flow coating method was used. In this method, the modification solution flows along the membrane and deposits on the membrane surface. After the evaporation of the solvent, on the membrane surface, a new layer is created. The quality of the layer depends on the solution flow velocity, the solution viscosity, and its surface tension. In the case of porous membranes, additional parameters such as the processing time and the transmembrane pressure should be considered. It seems to be related to the solution penetration through the pores, which could become partly or completely blocked by the new layer [18,19].

This method is easier to operate than chemical modifications and it makes it possible to modify all membranes that are in the module at the same time. However, for nanomaterial or some polymers, the coating process could not be efficient because interfacial interactions between the membrane and the modifying particles are too weak. For this reason, before the modification, the membrane is covered with the material that makes the adsorption of organic/inorganic particles possible, or it is covered with a polymer layer that will constitute the matrix for inorganic particles.

3. Experimental

3.1. Methods and materials

In the research, polypropylene microfiltration membranes were used. The parameters that characterize these membranes are presented in Table 1. This type of membranes has good chemical and mechanical strength but their disadvantage is the hydrophobic surface. To improve the wettability, the membrane surface has been covered with a new layer in the flow coating process.

The new layer has been made of the hydrophilic copolymer (PEBAX 2533) and organic or inorganic particles. The materials used are presented in Table 1.

The flow coating process was done with continuous recirculation of covering solution for 10 min. The solution volumetric flow was 890 ml/min, the transmembrane pressure was lower than 0.1 bar. After the covering, membranes were dried in atmospheric conditions. The cover solution contained 2% mass of PEBAX 2533 and different

Table 1
Materials used in the new membrane preparation

Materials	Role	Properties
Polypropylene membranes	Separation of ingredients	Hydrophobic surface, capillary, internal diameter 1.8 mm, outer diameter 2.7 mm, porosity 55%
PEBAX 2533 (Arkema)	Improvement of wettability, a matrix for additives	Hydrophilic properties
Organic additives (Sigma-Aldrich) - PEO-PAA	Improvement of wettability	Polymers
Inorganic additives (Sigma-Aldrich): - TiO_2-Al_2O-ZnO-ZrO_2	Improvement of wettability	Inorganic nanoparticles

concentrations of additives. 2-Butanol (Sigma–Aldrich, Poland) was used as a solvent. The own-made modules were prepared for the flow coating and microfiltration process. The modules were prepared using PVC housings and consisted of 20 capillary membranes with a length of 48 cm. The total filtration area was about 550 cm^2. In both processes (flow coating or MF), the feed stream (modification solution, water stream, or wastewater stream) flowed inside the membranes and the permeate (in case of MF) was collected in the space between the membranes.

The described process of new membrane production could be divided into two parts. At the first stage, new materials were prepared, and their contact angle was measured. The 2% PEBAX solutions with different additive concentrations were prepared. The samples were prepared on Petri dishes by casting a specific volume of solution and evaporating the solvent in room conditions. Samples prepared in this way had a flat geometry. Based on the results of the contact angle, the best material composition was chosen and then used in the process of further membrane modification.

The contact angle was determined by using the sessile drop method. The OCA 25 goniometer was used in the tests. The liquid probe droplets were deposited on the membrane surface by using a micro syringe with automatic dispenser, while the images were captured by a digital camera, which made it possible to measure static contact angles. The samples of flat materials and samples of the unmodified and modified membranes were tested. In the research, the testing liquid was reverse osmosis (RO) water at room temperature. The use of water makes it possible to compare the obtained results with the literature data. The volume of water drop was 0.5 µl. This volume provides the setting of a drop on the inside membrane surface. The example contact angle photos of the measurement are presented in Fig. 1.

In the second stage, membranes were modified with the chosen materials. In this part, the contact angle of the membranes with a new layer and ultrafiltration coefficient (UFC) were measured. Additionally, new membranes were tested in the laundry wastewater microfiltration process.

The impact of the modification on UFC and the quality of permeate were tested by a typical microfiltration plant (Fig. 2). The feed pressure was 2.5 bar and the volumetric cross-flow was 500 l/h. In the research, RO water or laundry wastewater was used as a feed stream. Wastewater was taken from "Holywood", the industrial laundry in Sierpc (Poland). The physicochemical parameters used to characterize the feed stream and the permeate were pH, conductivity, turbidity, chemical oxygen demand, and interfacial tension (IFT).

4. Results and discussion

The obtained results of the contact angle for different composites are presented in Table 2.

Based on the results, we can state that PEBAX has hydrophilic properties and the tested organic/inorganic particles improve the wettability of the prepared materials. In the case of inorganic particles, the hydrophilicity improvement could be noticed even for low concentration of particles, and with the increase of concentration, the contact angle change is small. It is related to the fact that only some particles are situated on the material surface while the rest is deeper in the sample. Additionally, nanoparticles have a tendency to agglomerate. Such agglomerates sediment under the surface. Agglomerates have also a lower specific surface area than the sum of single nanoparticles so their influence on the surface properties of a material is lower. A tendency to agglomeration by nanoparticles increases with the increase of their concentration, so the intensity of negative effects

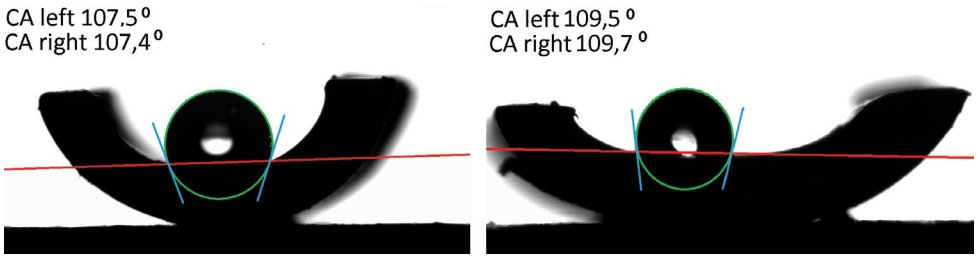

Fig. 1. Contact angle measurement using a sessile drop method.

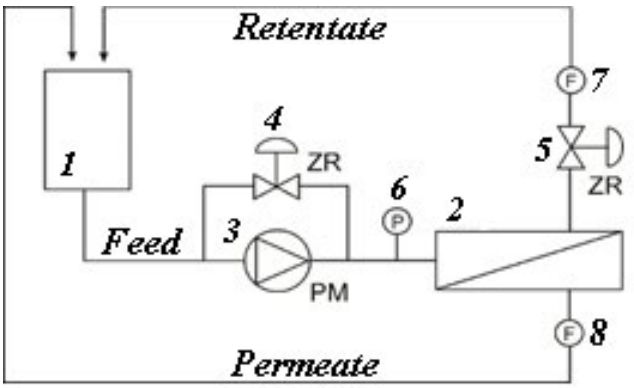

Fig. 2. The scheme of microfiltration plant. 1–feed tank, 2–membrane module, 3–pump, 4, 5–regulation valves, 6–pressure gauge, 7, 8–flowmeters.

also increases. In the case of polymeric additive, the change of contact angle is related to the concentration of polymer. It results from the steady distribution of the polymer additive in the polymer matrix and better interactions between the polymer chains than between polymer chains and nanoparticles. We can also state that the polymer additive improves the wettability of a sample prepared by PEBAX better than inorganic particles (Fig. 2). However, the concentration limit of inorganic (about 10%) and organic (about 30%) particles exists and above this value the preparation of the layer is impossible. In the next part of the research, materials with the lowest contact angle were chosen (one with organic and one with inorganic additives) so the modifying solution consisted of PEBAX 2533 with 10% ZrO_2 and PEBAX 2533 with 30% PEO.

The results of the contact angle for the flat samples of material, the contact angle of the modified membranes (capillaries) and the UFC of these membranes are presented in Table 3.

Small differences between the measured contact angles and the literature data indicate that the measurements were conducted correctly. The differences in results may be due to the level of water purity (a change in surface tension of the water), the temperature in which the measurements were conducted or the accuracy of the equipment used to determine the shape of droplets on the surface.

Based on the results, we can state that the measured contact angles of the prepared flat samples and the modified membranes are different. It is related to two effects. Firstly, it is related to the difference in structure between flat, nonporous samples and capillary porous membranes. Secondly, in the flow coating process, the created layer is thin, so it is possible that there exist some unmodified parts of the membrane surface. Additionally, the membrane pores may be blocked by a new layer so the porosity of the membrane is changed. However, it could be stated that when a lower contact angle characterizes the material, the membrane modified with this material also has a lower contact angle than the membrane modified with the material characterized by a higher contact angle. We can also state that for all the modifications, the improvement of membrane wettability properties was

Table 2
Contact angle results for the prepared materials

Continuous phase	Dispersed phase	Concentration of dispersed phase in continuous phase [%]						
		0	2	5	10	15	20	30
PEBAX 2533	TiO_2	83.2	–	–	68.9	66.2	–	–
	Al_2O_3	83.2	76.2	74.5	75.6	–	–	–
	ZnO	83.2	71.5	74.2	71.5	–	–	–
	ZrO_2	83.2	58.4	55.4	52.3	–	–	–
	PAA	83.2	–	–	78.6	58.2	50.5	48.4
	PEO	83.2	–	–	73.1	62.6	59.5	38.6

Table 3
Contact angle results and UFC for the modified membranes

Materials	Geometry	Measured contact angle (°)	Literature data of contact angle	UFC [ml/(min cm^2 bar)]
PP (unmodified)	flat	103	107 [20]	–
PP (unmodified)	capillary	112	99 [21], 118 [22]	1.55
PEBAX	flat	83.2	74 [23], 79.0 [24]	–
PP + PEBAX	capillary	97.5	–	1.64
PEBAX + ZrO_2	flat	52.3	–	–
PP + PEBAX + ZrO_2	capillary	85.3	–	2.26
PEBAX + PEO	flat	38.6	–	–
PP + PEBAX + PEO	capillary	83.5	–	1.66

obtained. It was confirmed by the UFC measurements as well. For the modified membranes, we obtained higher values of UFC than for unmodified membranes. The low value of UFC for the membrane modified by PEO is related to blocking the pores by the polymer. The high concentration of PEO influenced the increase in solution viscosity, which resulted in the higher polymer deposition on the membrane.

The membranes were also tested by scanning electron microscopy (SEM) analysis (Fig. 3). The obtained photos confirm the development of a new layer on the membrane surface. We can also observe that some pores were blocked and the membrane surface was not covered by a homogeneous layer. However, based on the UFC measurements, we can state that the total effect of the modification process was positive.

The presented research work did not involve tests of strength and durability of new structures. However, in their previous research, the authors had developed PEBAX layers on polymeric membranes similarly, both for gas separation [25] and membrane filtration [2]. In both cases, no change in the new structure was observed throughout the process duration and no deterioration of the strength of polypropylene membranes was stated.

In the last part of the research, the membranes were tested in the microfiltration process. The process parameters and the test description were presented in the "Materials and methods" section. The obtained results of the permeate volumetric flow change in time are shown in Fig. 4. For all types of the tested membranes, the permeate flow decreases in time of the process and tends to a constant value. The permeate flow changes in the time of the microfiltration process could be divided into three stages. In the first stage, the decrease in permeate flow is very fast and related to the blocking of pores. In the second stage, the flow of the permeate decreases more slowly and the change of permeate volumetric flow is related to the creation of a cake layer on the membrane surface. In the last part, the permeate volumetric flow tends to a constant value because the rate of creating the cake layer and the rate of renewing the membrane surface by the flow of the retentate are equal. These three stages of the process are present for all modules but in the case of modules with modified membranes, the permeate volumetric flow is higher than for the module with unmodified membranes. It confirms the obtained results of the UFC. The highest UFC value and the permeate flow characterize membranes modified by ZrO_2, next by PEO and the lowest values characterize unmodified membranes.

In the research, the influence of membrane modification on the quality of permeate and on the change of the permeate quality in time during the microfiltration process was investigated. The obtained results are shown in Fig. 5 and Table 4. In Fig. 5, the values of physicochemical parameters of the feed stream and the last sample of the permeate are shown.

Based on the results, we can state that the modifications do not influence the quality of the permeate. In all cases, a clear and transparent stream was obtained after the MF process.

The turbidity of the permeate decreased under one nephelometric turbidity unit (NTU) for all types of membranes because all solids, such as insoluble salts, fabric residues, fats, and micelles made of surfactants and dirt, were retained on the membrane surface. The decrease in conductivity is the result of the retention of anionic surfactants by the cake layer, but for the membranes modified by ZrO_2, the decrease in conductivity is the lowest. We can state that the fouling effect was lower than for other membranes. The IFT increase is the result of the retention of some of the surfactants on the membrane surface. Surfactant particles and the dirt created micelles that could be stopped by the membrane. Similarly as in the case of conductivity, for the membranes modified by ZrO_2, a lower value of IFT confirms a less intensive fouling. Additionally, the sediment build-up may also impede the transport of proteins and other organic substances. These effects decrease the COD value.

In the time of the microfiltration process, the change of physicochemical parameters of the permeate is little. Despite the fouling effect, which should block the transport of the substance, a part of the collected particles is pushed through the membrane and the average value of parameters remains on a similar level. Membrane modification improves the efficiency of the surfactant recovery process. In microfiltration, by using the modified membranes, the permeate was characterized by a lower value of the IFT than in the case of

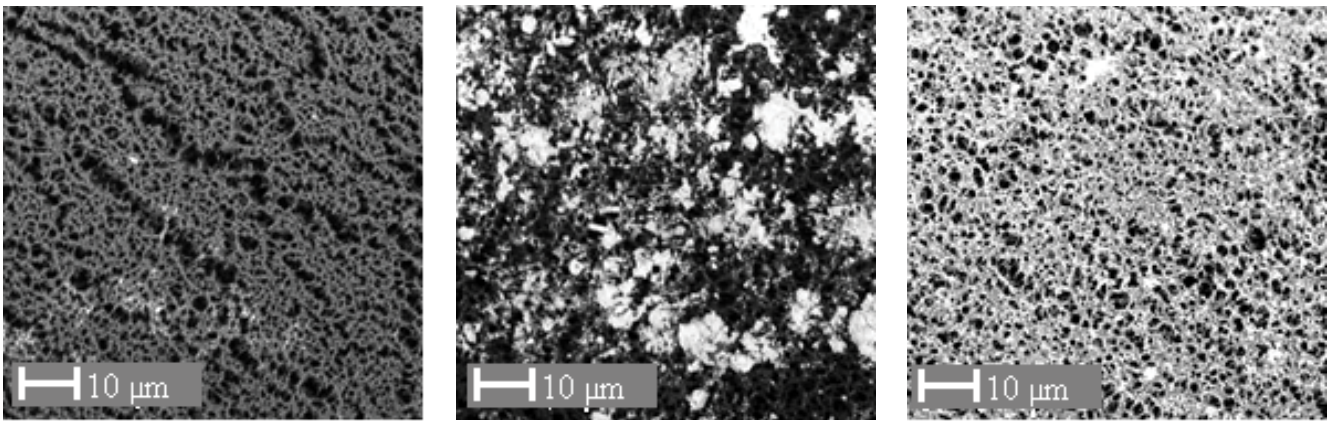

Fig. 3. SEM photos of membrane surface. From the left: unmodified membrane, membrane modified with ZrO_2, membrane modified with PEO.

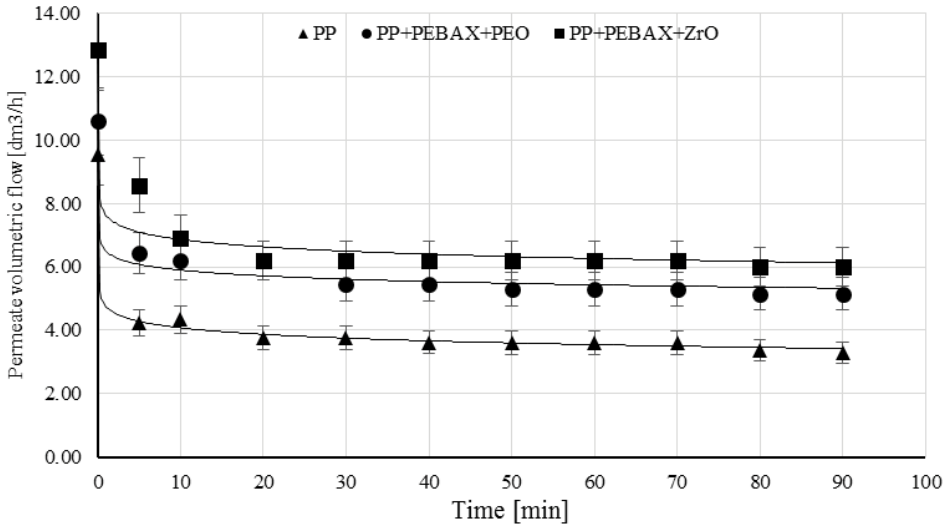

Fig. 4. Permeate volumetric flow changes in time.

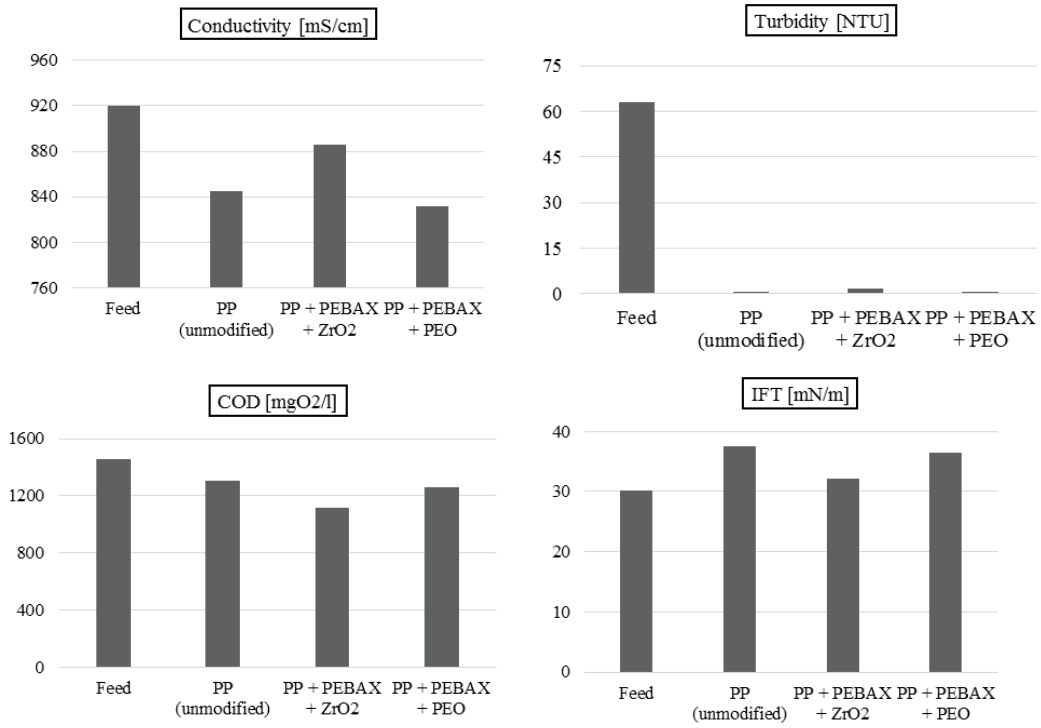

Fig. 5. Comparison of physicochemical properties of the feed stream and of the permeates.

unmodified membranes. It is related to a higher concentration of surfactants in the permeate.

5. Conclusions

The obtained results show that a higher permeate volumetric flow was obtained for the membranes modified with both ZrO_2 and PEO than for the unmodified ones. This is due to the improvement of the hydrophilic properties of the modified membranes, which is confirmed by the reduction of the contact angle for new materials. The formation of a new thin layer on the surface of the support membrane has also been confirmed in microscopic photographs. Additionally, the modifications do not decrease the quality of the permeate.

The developed membranes could be useful in the water and detergent recovery process in industrial-scale conditions. It has been confirmed by the preliminary tests carried out on the semi-industrial pilot plant connected with the tunnel laundry machine.

Table 4
Physicochemical parameters of permeates

		Parameter				
	Time (min)	pH (–)	Conductivity (mS/cm)	Turbidity (NTU)	COD (mgO_2/L)	IFT (mN/m)
PP (unmodified)	5	9.6	845	0.59	1,303	37.45
	10	9.5	842	0.64	1,280	37.64
	20	9.5	848	0.66	1,224	38.24
	60	9.5	847	0.98	1,183	39.57
	100	9.5	860	0.42	1,195	40.69
PP + PEBAX + ZrO_2	5	9.7	886	1.66	1,117	32.08
	10	9.7	845	0.7	1,150	32.37
	20	9.6	851	1.1	1,084	33.08
	60	9.6	878	0.23	1,059	34.33
	100	9.6	870	0.5	1,044	35.97
PP + PEBAX + PEO	5	9.6	832	0.69	1,258	36.52
	10	9.5	835	0.81	1,254	37.25
	20	9.5	845	0.91	1,250	37.58
	60	9.5	840	0.72	1,252	38.01
	100	9.5	841	0.58	1,262	38.29

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