

# Coagulation – Submerged Membrane System for NOM Removal from Water

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## Abstract

Our paper presents the results of investigations on the treatment of waters involved in unit process ultrafiltration, hybrid process – coagulation/sedimentation/ultrafiltration and in-line coagulation/ultrafiltration. In membrane filtration that we applied immersed capillary membranes. Simulated water containing humic acids at 30 mg/l was ultrafiltered with a constant volumetric permeate flux amounting to  $5.7 \times 10^{-6} \text{ m}^3/\text{m}^2 \cdot \text{s}$ . Four coagulants were tested, for which the optimal process parameters were determined experimentally. We determined the effectiveness of ultrafiltration and that of the hybrid system coagulation-membrane filtration, basing on measurements of the membranes' yield (permeate flux) and physico-chemical analysis of raw water and permeates. Also residual concentration of metal ions (Al and Fe) in permeates were determined. The application of the hybrid system, combining coagulation and ultrafiltration, offers better effects of the removal of organic pollution.

**Keywords:** ultrafiltration, coagulation, PVDF immersed membrane, water treatment, hybrid process

## Introduction

In contrast to underground waters, surface waters are often characterized by the presence of a large amount of bacteria, viruses and microorganisms, high turbidity, a high concentration of organic substances and the presence of micropollution such as pesticides, odorous and flavour substances. UF/MF applied as a unit process are inappropriate due to hydraulic reasons (limited yield due to membrane fouling) and due to the unsatisfactory quality of the produced water [1, 2]. That is why they are combined with oxidation, adsorption on powdered activated carbon, coagulation or biological process [1, 3, 4]. Such systems can be directly applied in the treatment of raw water, or followed by pre-clarification, using the classical method. Comparative investigations involving different treatment systems have shown that the combi-

nation of UF/MF with the adsorption on activated carbon or with chemical coagulation are the most effective solutions in view of the hydraulic yield, the quality of the treated water and the most commonly applied systems [3, 4].

The hybrid method involving the addition of a coagulant (aluminium sulphate or ferric chloride) prior to the ultrafiltration or microfiltration process can increase the removal of natural and anthropogenic organic substances, also the disinfection of by-products [2]. Such an approach can also contribute to a better yield of the membranes, both polymer and ceramic ones [4], but the permeate flux during UF is most favourable when the coagulation conditions bring about the formation of flocs of the zeta potential close to zero.

There are three mechanisms to increase the permeate flux by the application of pre-coagulation of water preceding UF/MF [4]:

- a lowering of the penetration of substances causing fouling into the inside of the membrane pores,

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- the formation of filtration cake on the membrane surface with a lower porosity,
- better conditions of particle transport out of the membrane surface.

Hybrid systems for natural water treatment based on coagulation and UF/MF usually lead to two basic solutions [5, 6]: coagulation of water in a separate tank with fast and slow stirring, sedimentation and UF/MF and in-line coagulation (adding a coagulant before membrane filtration, i.e. without sedimentation) and then UF/MF of the suspension of post-coagulation flocules. Our investigations concerned the efficiency of the hybrid system of coagulation-sedimentation-ultrafiltration/microfiltration, using ceramic and capillary membranes [7, 8]. Filtration in the hybrid system brings about a considerable increase of the membrane yield, which differs only slightly from the permeate flux in the case of deionized water. This has been confirmed by the calculated values of the relative permeability of the membrane, which are in the hybrid system: 0.98 for simulated water with a TOC content of 7 mgTOC/l and 10 mgTOC/l [7, 8]. The retention coefficients of the particular indices of water loading are also much more favourable than in the case of coagulation alone and direct UF/MF.

The objective of our paper is to examine and to compare the efficiency of the hybrid coagulation-ultrafiltration process with UF alone in the treatment of water containing NOM. Another objective was to select the efficient and economical hybrid process. We used an immersed membrane module obtained from Zenon. The assessment criteria was based on the standards legally binding in Poland, i.e. the Regulation of the Ministry of Health of November 19, 2002, concerning the requirements involving the quality of potable water (Journal of Law nr. 203, item 1718) [9].

## Experimental

### Characteristics of an Immersed Membrane Module

In our investigations we applied the testing unit Zee Weed 10. The Zee Weed water treatment technology is a membrane process with very low energy consumption, based on an ultrafiltration module immersed directly in the water subjected to treatment. The ZeeWeed UF membrane system was operated at a constant flux of 21.5 l/m<sup>2</sup>h, and during several hours of operation the transmembrane pressure was also constant, so the pressure change had not been monitored. The ZeeWeed UF membrane is a submerged hollow-fiber membrane, that allows for operation under a slight vacuum (10-50 kPa), instead of under pressure. A vacuum can draw product water through the membrane. The membranes are outside/in hollow fibers. The filtration capillary constructed in such a way that the outward structure is covered with a polymer film with pores whose nominal diameter amounts to 0.03 µm and the absolute magnitude of the pores to 0.2 µm. It means that when the microbiological pollution exceeds 0.2 µm (including

also Giardia and Cryptosporidium), the suspension and oxidized compounds of iron and manganese have no access to the flux of the treated water [10]. The membrane surface chemistry is neutral and hydrophilic. A series of hollow fibers have a combined 0.93 m<sup>2</sup> surface area and are connected to top and bottom headers and submerged in a 30 l process tank. The length of the module is 692 mm and its width is 110 mm. The top and bottom headers are connected to the filtrate vacuum pump. A blower supplies air to a diffuser at the base of the process tank to continuously agitate the fibers and remove accumulated solids from the membrane surface. Airflow to the process tank was maintained at 60 l/min. The resulting scouring action mitigates the build-up of solids on the membrane surface. The system includes a clean-in-place tank of 15 liter volume where filtrate is stored for back pulsing the membrane. In the back pulse mode, the direction of flow through the membranes is reversed. Filtrate water from the tank is pumped from the clean water side of the membrane back to the feed water side in order to clean away material accumulated on the membrane surface. During back pulsing, at regular intervals of from 10 minutes, the flow through the membrane is reversed for 20 seconds to remove solids accumulated on the membrane surface. To limit the concentration of substances in the membrane, a flux of concentrate is extracted from the process tank. The characteristic of the module and membrane is presented in Table 1.

The hybrid process of coagulation/ultrafiltration and ultrafiltration alone were carried out in the pilot system presented in Fig. 1.

## Methodology

Simulated water used in the tests was prepared by dissolving powdered humic acid manufactured by Aldrich, in an amount of 30 mg/l tap water, which corresponds to 10 mg of TOC/l. The investigations involved five basic stages:

Table 1. Characteristics of membrane modules ZW10 [9].

Membrane type	Capillary, hydrophilic
Nominal area, m <sup>2</sup>	0.93
Membrane material	PVDF
Nominal membrane pore size, nm	40
Outside diameter of the capillary, mm	1.95
Inside diameter of the capillary, mm	0.75
Length of the module, mm	692
Width of the module, mm	109.5
Transmembrane pressure	max. 62 kPa at 40°C
Flux, l/m <sup>2</sup> h	15-35

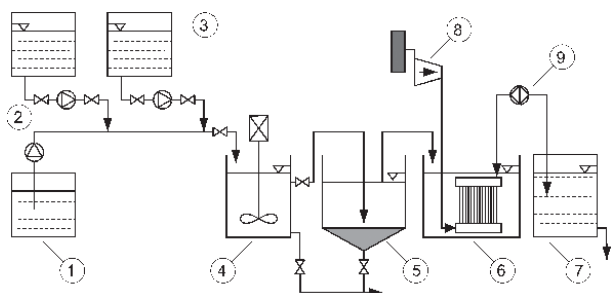


Fig. 1. Schematic diagram of the coagulation – ultrafiltration system (1) – water tank; (2) – coagulant tank; (3) – pH correcting solution tank; (4) – stirring cell; (5) – sedimentation tank; (6) – ultrafiltration pilot module; (7) permeate tank; (8) – blower; (9) – two-way pump

- the first stage was the selection of the optimal parameters of the coagulation process, i.e. the kind and amount of the coagulant and pH of coagulation. Four coagulants were tested: ferric chloride, aluminium sulphate, PIX-113 (aqueous solution of ferric sulphate) and PAX-25 (aqueous solution of polyaluminum chloride). The coagulation was carried out in four doses of coagulant, within the range of  $1 \div 7.2 \text{ mg/dm}^3$  of metal ion with three pH values for ferric chloride and aluminium sulphate and two pH values for PIX-113 and PAX-25. The pH range was within  $5.5 \div 8.8$ . The pH was corrected by a 1% solution of NaOH and HCl. Details involving the coagulation process are presented in [8].
- the second stage involved the conditioning of membranes by filtration of deionized water under constant process conditions for 5 hours in order to determine the dependence of the volumetric flux of deionized water ( $J_w$ ) on time for the investigated membranes and to obtain a constant value of this flux.
- the third stage was carried out in the hybrid system of coagulation/sedimentation/ultrafiltration of water, in order to assess the transport and separation properties of the investigated membranes after the pre-treatment of water in the coagulation process.
- the fourth stage was realized using “in-line” coagulation, i.e. after the addition of the coagulant fast stirring

was applied for over 1 minute and then the feed was passed directly to the process tank, in which ultrafiltration membranes were immersed. The measurements were carried out for 8 hours.

- the fifth stage covered the filtration of simulated water in the unit process of membrane filtration (8 h of process duration), in order to the efficiency of the UF process.

Membrane filtration was carried out with a transmembrane pressure of 0.02 MPa, temperature  $T=293 \pm 2\text{K}$  and the flux  $5.7 \cdot 10^{-6} \text{ m}^3/\text{m}^2 \cdot \text{s}$  ( $25 \text{ l/m}^2 \cdot \text{h}$ ). During 8 hours of experiments there was no observed pressure change. That is why the dependence of pressure on time was not monitored.

Membrane filtration (third, fourth and fifth stage) was carried out in the open mode of operation, i.e. the permeate was not reversed to the feed tank, and the water after coagulation (third stage) and simulated water (fourth stage) was fed to the tank in an amount equal to the volume of the removed permeate.

### Physicochemical Analyses of Water

The efficiency of the unit process (coagulation, ultrafiltration) and hybrid processes (coagulation/sedimentation/UF and in-line coagulation/UF) was assessed based on the physicochemical analysis of simulated water, water after coagulation or ultrafiltration, as well as on the permeates of two realized methods of the hybrid process. The analysis comprised the determination of organic carbon TOC, turbidity, and absorbance with a wavelength of 254 nm, conductivity, and residue of iron or aluminium in the treated water. Also, pH and conductivity were analyzed.

The analysis involving the content of chemical compounds was carried out in raw simulated water, in water after coagulation and in the collected permeates every 60 minutes during the filtration process, for all the investigated coagulants.

## Results and Discussion

### Selection of Optimal Coagulation Parameters

Before the studies on membrane filtration, the optimal parameters of the coagulation process were determined

Table 2. Optimal parameters of coagulation process and its efficiency.

Coagulant	pH	Dose	Efficiency, %	
			TOC	Absorbance, 254 nm
$\text{FeCl}_3$	7.0	$4.1 \text{ mg Fe/dm}^3$	63.3	97.8
$\text{Fe}_2(\text{SO}_4)_3$ (PIX-113)	7.5	$4.0 \text{ mg Fe/dm}^3$	42.4	72.5
$\text{Al}_2(\text{SO}_4)_3$	7.5	$4.1 \text{ mg Al/dm}^3$	61.3	90.2
PAX-25	7.5	$3.6 \text{ mg Al/dm}^3$	37.2	96.7

experimentally using a jar test, i.e. reagent dose and pH of water. The obtained results and the efficiency of the coagulation process for optimal doses of the coagulant and optimal pH are presented in Table 2.

The efficiency of the applied reagents was assessed based on the percentage of the removed organic compounds determined as the content of total organic carbon (TOC) and as the absorbance at a wavelength of 254 nm (Table 2). Also the dose, which ensured the appropriate removal of water loading, was taken into account. The lowest optimal dose was determined for PAX-25 and PIX-113 coagulants, due to the low removal of TOC. Higher doses were determined for the remaining two coagulants, and the removal efficiency of organic substance was 90-98% in the case of absorbance and 61-63% for TOC depending on the type of applied coagulant.

The coagulation process did not ensure a complete removal of organic compounds, and the concentration standards of metal ions remaining in the water exceeded the standards.

## Removal Efficiency of Organic Compounds

### Ultrafiltration Process

The results of the efficiency of the UF process alone have been gathered in Table 3, where the average concentrations of impurities and their retention coefficients are presented.

A considerable reduction of the turbidity and iron and medium removal of organics, especially COD, have been observed.

### Coagulation/Sedimentation/UF Process

Tables 4-5 present values of the parameters of physicochemical investigations, concerning the concentration of humic acids in simulated water amounting to 10 mgTOC/dm<sup>3</sup> in the hybrid system coagulation/sedimentation/ultrafiltration in the case of all the investigated coagulants.

Analyses of the investigated indices of organic impurities as well as iron and aluminium in the obtained permeates (Tables 4 and 5) show that the obtained level of raw water treatment after coagulation/sedimentation/

Table 3. Efficiency of water treatment with an ultrafiltration membrane alone (number of measurements – 6,  $\sigma$ -standard deviation).

Parameter	Feed		Permeate		Retention coefficient, %
	Average	$\sigma$	Average	$\sigma$	
pH	7.0		7.9		-
Conductivity, mS/cm	3.080		2.300-3.010		
Turbidity, NTU	8.02	0.01	0.14	0.01	98.3
Total iron, mg/dm <sup>3</sup> Fe	0.66	0.01	0.10	0.02	84.9
COD with KMnO <sub>4</sub> , mg/dm <sup>3</sup> O <sub>2</sub>	5.52	0.08	3.13	0.05	43.3
Absorbance UV ( $\lambda=254\text{nm}$ ), cm <sup>-1</sup>	0.329	0.002	0.081	0.001	75.4

Table 4. Average values of the physicochemical parameters of water subjected to treatment by coagulation/sedimentation/UF with the application of FeCl<sub>3</sub> and PIX-113 as coagulants.

Parameter	FeCl <sub>3</sub>				PIX-113			
	Feed		Permeate		Feed		Permeate	
	av	$\sigma$	av	$\sigma$	av	$\sigma$	av	$\sigma$
pH	7.0-7.1		7.7-7.9		7.0		7.5	
Conductivity, mS/cm	2.76-2.86		2.77-2.89		1.07-1.10		1.15-1.17	
Turbidity, NTU	6.5	0.10	0.11	0.06	7.78	0.01	0.21	0.02
Total iron, mgFe/dm <sup>3</sup>	0.21	0.02	0.03	0.02	0.32	0.01	0.07	0.01
Oxidizability with KMnO <sub>4</sub> , mgO <sub>2</sub> /dm <sup>3</sup>	4.47	0.37	2.37	0.46	6.07	0.08	3.00	0.00
Absorbance UV <sub>254</sub>	0.37	0.003	0.054	0.011	0.42	0.012	0.035	0.002

av-average,  $\sigma$ -standard deviation, no. of measurements – 6,

Table 5. Average values of the physicochemical parameters of water subjected to treatment by coagulation/sedimentation/UF with the application of  $\text{Al}_2(\text{SO}_4)_3$  and PAX-25 as coagulants.

Parameter	$\text{Al}_2(\text{SO}_4)_3$				PAX-25			
	Feed		Permeate		Feed		Permeate	
	av	$\sigma$	av	$\sigma$	av	$\sigma$	av	$\sigma$
pH	7.5		7.9-8.0		7.5		7.8-8.0	
Conductivity, mS/cm	2.67-2.74		2.78-2.83		2.70		2.76-2.78	
Turbidity, NTU	6.63	0.04	0.10	0.03	6.87	0.00	0.11	0.04
Total iron, mgFe/dm <sup>3</sup>	-	-	-	-	0.21	0.00	0.00	0.00
Oxidizability with $\text{KMnO}_4$ , mgO <sub>2</sub> /dm <sup>3</sup>	4.10	0.00	2.15	0.08	4.50	0.00	1.83	0.15
Absorbance UV <sub>254</sub>	0.417	0.006	0.148	0.013	0.415	0.001	0.118	0.013
Aluminum, mgAl/dm <sup>3</sup>	0.66	0.02	0.09	0.01	0.68	0.02	0.14	0.01

av-average,  $\sigma$ -standard deviation, no of measurements -6,

UF process meets the quality standards for drinking water specified in the Directive of the EU [6] and in Polish Regulations [9].

#### Coagulation “in line”/UF Process

Tables 6 and 7 present the results of water treatment using hybrid process coagulation “in line”-UF, also for all the applied coagulants.

Comparing the determined quality standards [6.9] we may say (Tables 6 and 7) that the process of “in-line” coagulation/UF improved the parameters of raw water very efficiently. The results obtained in all the investigations comply with the regulations involving the quality of drinking water and are comparable to those obtained by applying the previous treatment method.

Based on the average concentration of the parameters of the substances present in the investigated water, the retention coefficients (R) were calculated using the equation  $R = 1 - (C_n/C_p)$  (where:  $C_n$  - feed concentration,  $C_p$  - permeate concentration). Table 8 presents the retention coefficients of the physicochemical parameters concerning the ultrafiltration process and two hybrid processes, i.e. coagulation/sedimentation/UF and in-line coagulation/UF. The results show that the treatment on ultrafiltration membranes is efficient, particularly with respect to the removal of substances, which cause turbidity. The introduction of coagulation prior to ultrafiltration brings about a rise of the retention coefficient, especially with respect to organic substances determined as absorbance UV<sub>254</sub> and oxidizability ( $\text{KMnO}_4$ ). The application of coagulation does not lead to a decrease of turbidity of the treated water as compared to the process carried out directly on an ultrafiltration membrane, independent of the type of the hybrid process (classical coagulation or “in-line” coagulation) and the type of the applied coagulant.

Yet the application of coagulation influences considerably the removal of organic substances from water, determined as absorbance UV<sub>254</sub>. The highest removal

efficiency of these substances was obtained when the coagulant PIX-113 was used in the process coagulation/sedimentation/UF. The process “in-line” coagulation/ ultrafiltration turned out to be less effective, not only when compared with the hybrid system with coagulation and sedimentation but also with direct ultrafiltration.

Organic substances determined as to their oxidizability ( $\text{KMnO}_4$ ) are more efficiently removed from water in hybrid processes. The coagulant PAX-25 turned out to be the best reagent in the removal of organic substances, in the process coagulation/sedimentation/UF, and the coagulant  $\text{FeCl}_3$  in the process of “in-line” coagulation/ ultrafiltration. In this case the removal effectiveness is influenced both by the type of coagulation and the type of the applied coagulant, for example, coagulation with the use of  $\text{Al}_2(\text{SO}_4)_3$  and  $\text{FeCl}_3$  is more effective in the “in-line” coagulation, and for the remaining ones coagulation with sedimentation. Substances determined as to their oxidizability with  $\text{KMnO}_4$  are removed from water to a smaller degree than the organic substances determined as absorbance UV<sub>254</sub>. This can be explained by a scope of different measurements by means of these two methods. The oxidizability with  $\text{KMnO}_4$  covers also some inorganic substances, which oxidize in the determination process.

The application of ferric and aluminium coagulants entails a rise of concentration of these elements in water, and therefore the content of aluminium and iron in the treated water was also assessed. In all these cases, independently of the applied coagulant, trace amounts of iron were found in the treated water after the hybrid process (below the standards for drinking water), both ferric chloride and ferric sulphate (within 0–0.1 mg/l) (Table 4). With respect to aluminium ions, a small amount of aluminium was found in the treated water, depending on the type of the coagulant (0.09-0.14 mg/l) (Table 5). The treated water obtained in the hybrid process with “in-line” coagulation did not contain any residual amount of iron or aluminium (Tables 6 and 7). The content of aluminium and iron in potable water should not exceed 0.2mg/l [9].

Table 6. Average values of the physicochemical parameters of water subjected to treatment by coagulation “in line”/UF with the application of FeCl<sub>3</sub> and PIX-113 as coagulants.

Parameter	FeCl <sub>3</sub>				PIX-113			
	Feed		Permeate		Feed		Permeate	
	av	σ	av	σ	av	σ	av	σ
pH	7.0-7.2		7.7-7.9		7.1		7.9	
Conductivity, mS/cm	2.6-2.65		2.61-2.74		1.36		1.31-1.32	
Turbidity, NTU	7.15	0.34	0.15	0.08	7.7	0.01	0.17	0.02
Total iron, mgFe/dm <sup>3</sup>	0.30	0.01	0.01	0.01	0.45	0.01	0.00	0.01
Oxidizability with KMnO <sub>4</sub> , mgO <sub>2</sub> /dm <sup>3</sup>	5.30	0.26	2.30	0.21	6.15	0.09	3.09	0.08
Absorbance UV <sub>254</sub>	0.389	0.003	0.130	0.005	0.441	0.001	0.117	0.006

av-average, σ-standard deviation, no. of measurements - 6,

Table 7. Average values of the physicochemical parameters of water subjected to treatment by coagulation “in line”/UF with the application of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and PAX-25 as coagulants.

Parameter	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>				PAX-25			
	Feed		Permeate		Feed		Permeate	
	av	σ	av	σ	av	σ	av	σ
pH	7.6		8.0-8.1		7.5		7.9-8.0	
Conductivity, mS/cm	2.70		2.73-2.82		2.62		2.61-2.78	
Turbidity, NTU	7.12	0.02	0.16	0.05	7.23	0.00	0.12	0.01
Total iron, mgFe/dm <sup>3</sup>	-	-	-	-	0.26	0.00	0.01	0.01
Oxidizability with KMnO <sub>4</sub> , mgO <sub>2</sub> /dm <sup>3</sup>	4.70	0.00	2.215	0.12	4.70	0.00	2.28	0.13
Absorbance UV <sub>λ=254</sub>	0.439	0.004	0.127	0.028	0.43	0.00	0.155	0.003
Aluminum, mgAl/dm <sup>3</sup>	0.66	0.02	0.011	0.01	0.68	0.02	0.16	0.01

av – average, σ – standard deviation, no. of measurements - 6,

Table 8. Retention coefficients for the investigated processes and applied coagulants.

Parameter	UF alone	Retention coefficient (%) of hybrid system with							
		PIX-113		FeCl <sub>3</sub>		Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>		PAX-25	
		c/s/UF <sup>*)</sup>	in-line	c/s/UF <sup>*)</sup>	in-line	c/s/UF <sup>*)</sup>	in-line	c/s/UF <sup>*)</sup>	in-line
Turbidity	98.3	97.3	97.8	98.4	97.9	98.4	97.8	98.4	98.4
Total iron	84.8	79.3	99.3	87.0	97.8	----	----	100.0	96.8
Oxidizability with KMnO <sub>4</sub>	42.9	50.6	49.7	47.0	56.6	47.6	52.1	59.3	51.4
Absorbance UV <sub>254</sub>	75.1	91.6	69.5	85.4	66.5	64.4	71.1	71.6	64.0
Aluminium	----	----	----	----	----	87.0	84.0	79.4	77.2

c/s/UF<sup>\*)</sup> – coagulation/sedimentation/ultrafiltration



### Conclusions

1. As expected, coagulation applied as a unit process does not cause a complete reduction of organic-containing NOM. The removal of TOC amounted to 37-63%, depending on coagulant type. Coagulation process in water treatment is identified with the removal of turbidity rather than TOC [11].
2. Analyzing the results involving the unit process (ultrafiltration), we found a considerable drop of turbidity (in 98.3%) in the water and a low removal of organic substances determined as to their oxidizability (COD) with  $\text{KMnO}_4$  (43%), compared to the hybrid process. Similar results have been obtained by other research [2, 6, 11-14].
3. The application of the hybrid system results in a higher efficiency of the removal of organic substances from water, depending on the kind of the applied coagulant and used mode of coagulation (in line or ordinary coagulation with sedimentation). The hybrid system with in line coagulation seems to be more efficient and economical.
4. The metals (Fe, Al) were efficiently removed to the level required by the Polish standards.

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