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# The influence of membrane and water properties on fouling during ultrafiltration

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## ABSTRACT

Membrane techniques are an alternative for classical processes of water treatment. However, one of their main drawbacks is that their capacity and membrane lifetime are limited by the phenomenon termed fouling. Natural organic matter (NOM) has been reported as the main foulant during water ultrafiltration. The character of NOM fouling depends on many factors. The results of the study on the effect of water properties such as pH, kind of foulant and ionic strength on the extent of membrane fouling are discussed in this article. Membranes used in the experiment vary in contact angle. Unified Modified Fouling Index (UMFI) was used to describe the intensity of fouling during ultrafiltration. The investigations was made with the use of the Ultrafiltration Cell Millipore CDS10 System. The device was equipped with flat sheet membranes and was operated in the dead-end mode. The study showed that the increase of ionic strength and pH resulted in the decrease of fouling. It was also found that the most severe fouling was caused by humic substances, followed by polysaccharides (dextrans) and the lowest fouling rate  was observed for the mixture of humic substances and polysaccharides. Membrane hydrophobicity and pore size distribution had also a significant impact on fouling in case of low ionic strength waters.

*Keywords:* Ultrafiltration; NOM; Fouling; Contact angle; pH; SUVA

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## 1. Introduction

Membrane techniques are an alternative for water treatment method for classical processes of water treatment. Due to gradual deterioration of water quality and the increasingly stringent standards on drinking water quality, membrane processes are becoming common for solution for drinking water production. However, in Poland, only three water treatment plants are using low-pressure membrane processes (i.e., Sucha Beskidzka-2006, and Jaroslaw, Water Treatment Station  KWK "Piast") in their treatment system. The main limitation of the implementation of membrane systems is related to accumulation of organic and/or inorganic substances on the surface and in pores of the membrane [1]. This phenomenon, termed fouling, hinders capacity and lifetime of membrane. Natural organic matter (NOM) has been reported as the main foulant during water ultrafiltration. The intensity of NOM fouling depends on many factors, among which properties of filtrated water, membrane type and parameters are of the greatest importance. It is caused by both, electrostatic repulsive forces between charges of foulants and membrane and adsorptive properties of membrane material related to its hydrophobicity and hydrophilicity [2]. Hydrophobicity/hydrophilicity can be defined using contact angle which is measured between water drop and membrane surface [3]. It is established that membrane is highly hydrophilic if the contact angle value

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is equal to 0° (complete wetting) and highly hydrophobic when the value is above 90° (no wetting) [4,5]. Generally, it is stated that the greater is the contact angle the more hydrophobic is the membrane.

Ionic strength, as one of the main properties of filtrated water, has a great influence on fouling, especially in the presence of calcium cations which affects the solubility of organic substances and change their molecular distribution [2,6,7]. Composition of organic substances is also an important factor. $SUVA_{254}$ (specific UV absorbance at 254 nm) is the parameter widely used to describe properties of organic substances. $SUVA_{254}$ is defined as the ratio of absorbance at 254 nm wavelength ($UVA_{254}$) to dissolved organic carbon (DOC) content [8].

Several fouling modes can be mathematically characterized using Hermia's model. Originally, the model was developed for constant pressure filtrations and in order to apply it to constant flow filtration conformal transformation must be performed [9]. Several membrane fouling indices (MFIs) have been proposed based on Hermia's model. One of them is Unified Modified Fouling Index (UMFI). The UMFI value can be determined from the dependence between normalized membrane specific flux $J_s'$ (dimensionless) and unit permeate capacity $V_s$ (dm³/m²), regardless of hydrodynamic process conditions. The higher is the value of UMFI the faster decline in normalized membrane specific flux is observed [10]. UMFI is defined based on the cake layer (as the dominant form of membrane fouling) formation equation:

$$1/J_s' = 1 + \mathrm{UMFI}\ V_s \qquad (1)$$

where $J_s'$ is the normalized membrane specific flux [-], $V_s$ is the unit permeate capacity [dm³/m²], and UMFI is the Unified Modified Fouling Index [m²/dm³].

It should be noted that normalized membrane specific flux is also relative flux (ratio of momentary flux to initial flux), which allows to compare fouling of membranes varying in cut-off.

The aim of the study was to investigate the effect of water properties such as pH, kind of foulant and ionic strength on the extent membrane fouling.

## 2. Materials and methods

The study was performed with the use of the Ultrafiltration Cell Millipore The CDS10 System. The CDS10 device was equipped with flat sheet membranes and was operated in dead-end mode. The operational pressure was kept constant at 0.1 MPa. The construction of ultrafiltration system was discussed in [11]. Characteristics of membranes provided by manufacturers are presented in Table 1.

Table 1
Membranes characteristics given by manufacture

| Membrane material | Manufacturer | MWCO [kDa] |
|---|---|---|
| Polyether-sulfone (PES) | Millipore Sp. z o.o. | 10 |
| Polyacrylonitrile (PAN) | KOCH Membrane Systems | 20 |
| Poly(vinylidene fluoride) (PVDF) | KOCH Membrane Systems | 30 |

Contact angle measurements of contact angle were carried out using pocket goniometer and the sessile drop method was applied. The contact angle is the angle formed by water at the three phase boundary where the water, air, and membrane surface [12].

Additionally, the cut-off of applied membranes was determined using standard substances i.e., polyethylene glicols (PEGs). PEGs of various molecular weight i.e., 0.2, 8, 20, 40 and 108 kDa were used to prepare solutions of concentration range 8–10 mgC/dm³. The retention degree was calculated based on measurements of organic carbon concentration. Membrane cut-off was determined as a molar weight of compounds which were rejected by the membrane in 90% [13].

The mean pore sizes and corresponding geometric standard deviations can be determined from diagram of relation between solute separation and mean pore size. The Einstein-Stokes (ES) diameter, which that corresponds to 50% of the solute separation, is taken as the mean pore size ($\mu_{50}$), while the ratio between the ES diameter corresponding to 84.13% of solute separation and that corresponding to 50% is taken as the geometric standard deviation ($\sigma_p$) [14]. The ES diameters were calculated based on molecular weights [15]. For polyethylene glycol:

$$r_{\mathrm{PEG}} = 16.73 * 10^{-10} M^{0.557} \qquad (2)$$

where $r_{\mathrm{PEG}}$ is the ES radius [cm], and $M$ is the the average molecular weight of PEG [g/mol].

UMFI was employed to describe intensity of fouling during ultrafiltration. Humic substances and dextrans were the main components of the simulated waters. The pH of the waters used in the range 5–9. Table 2 lists the characteristics of the feed waters (value of parameters and standard deviations) used in the study.

## 3. Results and discussion

Properties of membranes are an important parameter that affects degree of fouling. Some of the membrane properties and operational paremeters, such as: fluxes of deionized water, contact angles, determined cut-off, mean pore size and geometric standard deviation obtained for new membranes are shown in Table 3.

Table 2
Characteristics of the model waters

| Filtration number | | pH | $Ca^{2+}$ [mg/dm$^3$] | SUVA [m$^2$/gC] | TOC [mg/dm$^3$] | DOC [mg/dm$^3$] | $UVA_{254}$ [-] |
|---|---|---|---|---|---|---|---|
| 1 | PES | 6.95 ± 0.03 | 0 | 4.3471 ± 0.0011 | 6.14 ± 0.21 | 5.82 ± 0.15 | 0.253 ± 0.001 |
| | PAN | 6.70 ± 0.10 | 0 | 3.3429 ± 0.0003 | 7.06 ± 0.02 | 7.00 ± 0.02 | 0.234 ± 0.001 |
| | PVDF | 6.92 ± 0.07 | 0 | 3.5063 ± 0.0005 | 7.10 ± 0.12 | 6.36 ± 0.06 | 0.223 ± 0.002 |
| 2 | PES | 7.05 ± 0.07 | 98 ± 2.8 | 1.6935 ± 0.0007 | 5.73 ± 0.10 | 3.72 ± 0.09 | 0.063 ± 0.001 |
| | PAN | 7.09 ± 0.03 | 95.2 ± 0.0 | 1.5837 ± 0.0003 | 6.60 ± 0.02 | 4.42 ± 0.01 | 0.070 ± 0.002 |
| | PVDF | 6.96 ± 0.03 | 98.6 ± 2.9 | 1.6341 ± 0.0004 | 6.52 ± 0.09 | 4.10 ± 0.11 | 0.067 ± 0.001 |
| 3 | PES | 7.11 ± 0.03 | 199 ± 2.5 | 1.2336 ± 0.0006 | 6.09 ± 0.08 | 3.81 ± 0.08 | 0.047 ± 0.001 |
| | PAN | 7.00 ± 0.08 | 201.3 ± 3.8 | 1.5471 ± 0.0003 | 6.80 ± 0.04 | 4.46 ± 0.02 | 0.069 ± 0.001 |
| | PVDF | 7.01 ± 0.06 | 198 ± 2.9 | 1.3759 ± 0.0003 | 6.85 ± 0.09 | 4.07 ± 0.02 | 0.056 ± 0.001 |
| 4 | PES | 5.03 ± 0.05 | 0 | 3.1433 ± 0.0007 | 6.44 ± 0.07 | 6.49 ± 0.15 | 0.204 ± 0.001 |
| | PAN | 5.08 ± 0.01 | 0 | 3.3728 ± 0.0003 | 6.85 ± 0.04 | 6.76 ± 0.02 | 0.228 ± 0.001 |
| | PVDF | 4.99 ± 0.03 | 0 | 3.4745 ± 0.0005 | 6.91 ± 0.13 | 6.85 ± 0.08 | 0.238 ± 0.001 |
| 5 | PES | 9.02 ± 0.04 | 0 | 4.7687 ± 0.0017 | 6.20 ± 0.09 | 5.62 ± 0.20 | 0.268 ± 0.001 |
| | PAN | 8.99 ± 0.03 | 0 | 3.8462 ± 0.0006 | 6.91 ± 0.03 | 6.63 ± 0.06 | 0.255 ± 0.001 |
| | PVDF | 9.06 ± 0.02 | 0 | 3.7309 ± 0.0008 | 6.92 ± 0.01 | 6.54 ± 0.13 | 0.244 ± 0.001 |
| 6 | PES | 7.17 ± 0.03 | 0 | 1.5147 ± 0.0004 | 6.62 ± 0.05 | 6.47 ± 0.15 | 0.098 ± 0.000 |
| | PAN | 7.00 ± 0.10 | 0 | 0.7796 ± 0.0003 | 6.67 ± 0.08 | 6.67 ± 0.11 | 0.052 ± 0.001 |
| | PVDF | 6.96 ± 0.01 | 0 | 0.6577 ± 0.0002 | 6.68 ± 0.03 | 6.69 ± 0.15 | 0.044 ± 0.001 |
| 7 | PES | 7.01 ± 0.07 | 0 | 7.6331 ± 0.0017 | 5.25 ± 0.14 | 5.07 ± 0.11 | 0.387 ± 0.000 |
| | PAN | 6.98 ± 0.11 | 0 | 7.2258 ± 0.0006 | 6.80 ± 0.09 | 6.20 ± 0.05 | 0.448 ± 0.001 |
| | PVDF | 6.92 ± 0.03 | 0 | 7.0484 ± 0.0011 | 6.97 ± 0.02 | 6.20 ± 0.10 | 0.437 ± 0.000 |

Table 3
Membranes characteristics

| Membrane | Contact angle [°] | $J_m$[dm$^3$/hm$^2$] ($t$ = 20°C) | Cut-off [kDa] | $\mu_{50}$ [nm] | $\sigma_p$ [nm] |
|---|---|---|---|---|---|
| PES | 73.7 ± 2.1 | 143 ± 14 | 116 | 4.9 | 2.1 |
| PAN | 66.7 ± 5.4 | 91 ± 7 | 80 | 4.0 | 1.6 |
| PVDF | 57.5 ± 5.2 | 94 ± 7 | 6.5 | 1.4 | 1.6 |

Determined contact angles were in the range from 57.5 to 73.7°. This allowed classifying membrane materials as between strongly hydrophobic and strongly hydrophilic. PVDF membrane was found to be the most hydrophilic and PES membrane as the most hydrophobic (Table 3).

The determined cut-off values were significantly different from those provided by the manufacturers. It may result from different experimental conditions and standard substances. Membranes separation properties, except for particles size, depend on many factors e.g., particles shape, their flexibility and affinity to membrane material. Thus, the dependence between retention coefficient and molecular weight determined for ultrafiltration membranes during the study was not explicit [3,16]. It is stated that retention coefficient determined for particles of identical molecular weight but various shapes can be different. The study showed that PES and PAN membrane had similar mean pore size. However, PES membrane possessed wider pore size distribution in comparison with PAN membrane. Moreover, PAN and PVDF membranes were characterized by narrow pore size distribution, but mean pore size (i.e., compactness) of PVDF membrane was three times smaller than that determined for PAN membrane.

Fouling intensity was characterized by means of Unified Modified Fouling Index, which was determined using Least Squares Estimation. The exemplary diagram of dependence between $1/J_s'$ and $V_s$ including confidence intervals determined for significance level ($\alpha$) equal to 0.05 is shown in Fig. 1. The regression lines fitted well experimental data, which was confirmed by high values of correlation coefficients (0.9104–0.9999). Low correlation coefficient ($R$=0.0443) was obtained only in one case, i.e., filtration no. 3, PAN membrane, which could be explained by the character of the obtained function (constant function – no fouling occurred). Small measurement uncertainties of UMFI were btained

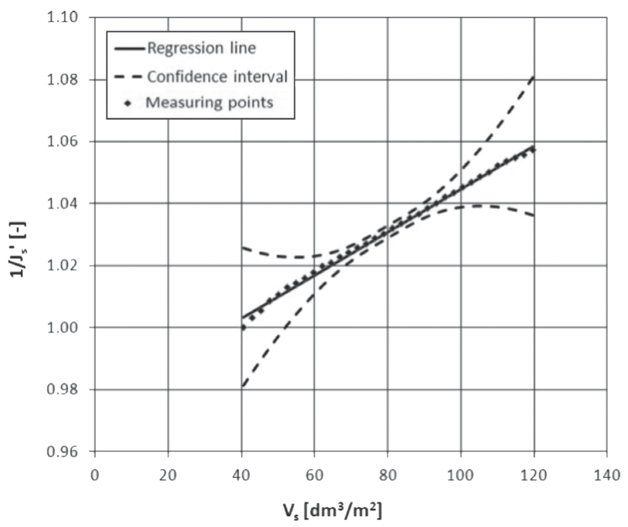

Fig. 1. The relationship between $1/J_s'$ and $V_s$ (filtration no. 3, PVDF membrane).

in most cases and did not exceed 10 %. (as shown in Figures 2-4). All measuring points and regression lines were covered by the determined confidence interval i.e. interval to which estimated parameter value belonged with 95% probability.

The plotted measuring points (on the basis of which UMFI was determined) represent the average of two replicates. The standard deviation for the average value of $1/J_s'$ ranged 0.03–0.14.

The pH of the feed is the crucial parameter affecting membrane fouling. The impact of pH on the fouling intensity occurred during filtrations 1, 4 and 5 (tab.2) is shown in Fig. 2. Fouling was lower for feed waters of higher pH. Manttari et al. showed that the membrane and humic acids were almost completely discharged at acidic pH (4–5), which promoted fouling [17]. The impact of pH was also studied by other researchers [18–21]. It was found that the same trend of adsorption of humic acids occurred at low pH for which electrostatic repulsion was lower. It was also reported in article published by Sutzkover-Gutman [22].

For pH 7, low fouling was observed for membranes of narrow pore size distribution and lower contact angle. Negative charge of functional group of organic substances prevented adsorption of NOM on negatively charged membrane surface, but on the other hand it improved the adsorption on positively charged materials (more hydrophobic) [2,23,24]. The tendency was no longer valid in acidic or basic environment. Fouling of PAN membrane was greater than of PES membrane in acidic environment. As mean pore size of both membranes was similar, the acidic environment caused greater hydrophobicity of PAN membrane. However, in basic environment PAN membrane fouling was lower than the one the obtained for PVDF membrane despite of its greater mean pore size and contact angle. In such an environment (i.e., high pH) PAN membrane seemed to act as more hydrophilic. However, measurements of contact angle were performed in pH 7. Probably, PAN material (and its properties) were more susceptible to pH change.

The ionic strength (expressed as the concentration of calcium ions) effect on UMFI index is presented in Fig. 3. It was observed that the increase of calcium ions concentration caused less severe fouling. It is generally accepted that the presence of divalent ions enhances fouling caused by humic substances [16,25–30]. This was owing to specific interactions between $Ca^{2+}$ and humic acids (HA–calcium complexation),which could reduce HA inter-chain repulsion and resulted in a compact HA molecular conformation, subsequently resulting in the formation of a less permeable and highly resistant fouling layer [31].

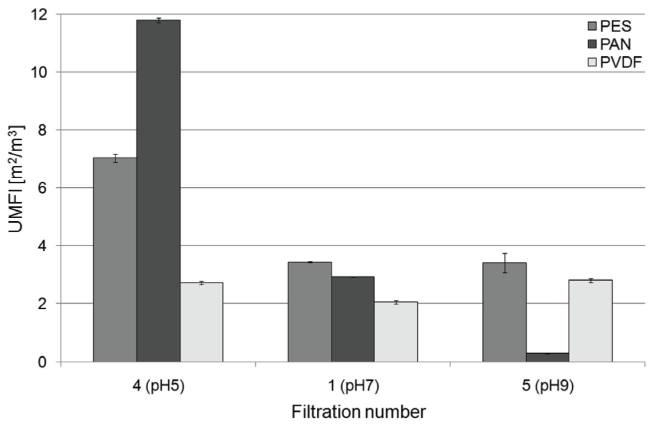

Fig. 2. Influence of pH on UMFI value.

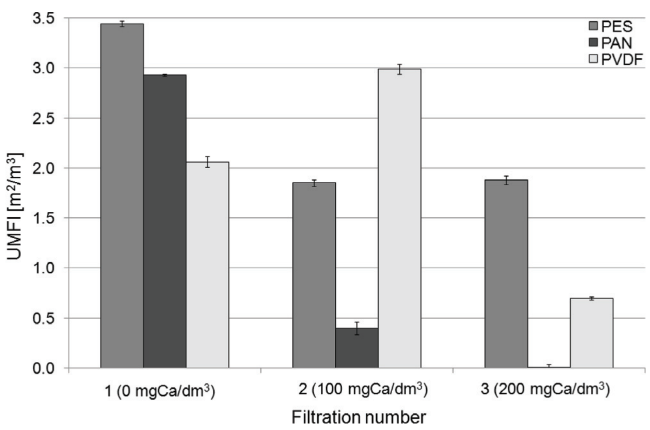

Fig. 3. Influence of ionic strength on UMFI value.

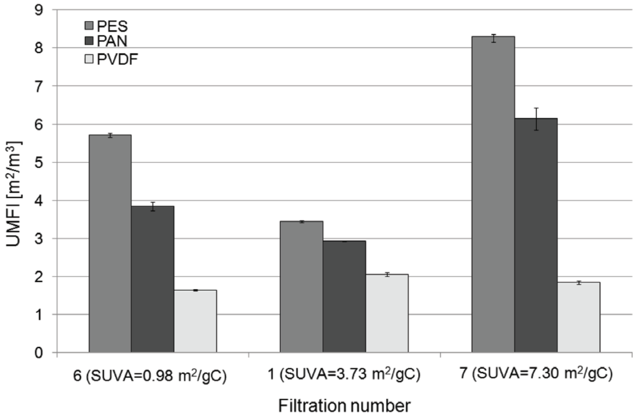

Fig. 4. Influence of organic substances composition on fouling.

It must be noted that $Ca^{2+}$ can reduce HA solubility and increase its aggregation by cancelling (protonation) the negative charge effect of the functional groups [32,33] or by bridging the negative membrane surface with the negative-charge functional groups (carboxylic, phenolics, and methoxyl carbonyls) of the humic substances [34]. According to Aoustin et al. as charge repulsion between ionized negative groups on HA is lowered by the $Ca^{2+}$, HA molecules tend to curl up to smaller molecular sizes [27]. Moreover, ionic strength and pH have an influence on mass distribution of organic substances [7].

Considering influence of calcium on fouling chemical properties and wide diversity of organic compounds present in feed water must be taken into account [29]. According to Yamamura et al. the presence of calcium ions causes more severe humic acids fouling than the one cancelling obtained for organic substances present in surface water [26]. According to Zularisam individual dextran fouling appeared to be unaffected by the increase in divalent calcium ions (from 0.1 to 1 mM) [35]. However, pronounced flux decline was observed in the case of individual and combined fouling of humic acids. According to Katsoufidou et al., as $Ca^{2+}$ concentration increases, humic acids aggregates grow larger in size forming a backwashable cake layer [16,29].

The gentle fouling observed in the presence of calcium ions (Fig. 3) could result from interactions between dextranes and humic substances, thus reducing the intermolecular bridging mechanism of $Ca^{2+}$, and subsequently implying minimal sorption of HA onto the membrane surface. The formation of backwashable filtration cake caused by particles aggregation seems to be the main mechanism. Moreover, the cake acts as a barrier that protects membrane pores from deposition of smaller particles. Such an explanation is in agreement with Katsoufidou et al. observations [16,29]. It should be emphasized that in a few cases, a small fouling in the presence of calcium ions was observed. This phenomenon was observed only at the highest dose of calcium (200 mg/l), which was reported in [16,29]. For smaller concentrations of $Ca^{2+}$, the authors noticed severe fouling. A discrepancy between the present results and the results reported by Katsoufidou is due to the fact that in both experiments different membranes cut-offs were used (150 kD PES vs.10 kDa PES) and the different concentrations of organics were applied in the feed. It can be concluded that considering the effect of calcium ions on fouling, the properties of the membrane and the other parameters of the feed need to be taken into account cumulatively.

SUVA index was used to determine qualitative composition of natural water. Value of $SUVA_{254} \geq 4$ m$^2$/gC is characteristic for water which contains significant amounts of hydrophobic, aromatic and high-molecular weight organic substances. Value of $SUVA_{254} \leq 2$ m$^2$/gC indicates that mainly non-humic, hydrophilic, low-molecular weight substances are present in water. Mixture of hydrophilic and hydrophobic, low- and high-molecular weight substances occur in water with $SUVA_{254}$ equal to 2–4 m$^2$/gC [8]. The influence of organic substances composition on fouling is shown in Fig. 4.

Waters characterized by medium value of SUVA equal to 3.73 m$^2$/gC blocked PES and PAN membranes less than waters with low content of hydrophobic substances. The most severe fouling was observed for waters, which contain hydrophobic, aromatic organic substances. The intensity of fouling of PVDF membrane was quite constant regardless of hydrophobicity of organic substances. Apart for interactions between membrane surface and organic substances also foulant-foulant interactions had an impact on membrane fouling. Waters prepared for filtrations 6 an 7 were mainly composed from mixtures of polysaccharides (no. 6) and sodium salts of humic acids (no. 7). The heterogeneity of these solutions was much smaller than in case of feed water used in filtration 1. It resulted in additional intermolecular interactions appearing in more homogenic solutions and caused weaker interactions between particles and membrane surface. Thus, fouling observed during filtration no. 1 was the lowest. The influence of hydrophobicity and hydrophilicity on membrane fouling is questionable. On the one hand hydrophilic fraction is said to be the main foulant [24,36,37], on the other hand, oppositely, the hydrophobic fraction seems to be of greater importance [38,39]. According to Jung C-W et al. the adsorption of hydrophobic fraction resulting in membrane fouling is much more rapid in comparison with hydrophilic fraction regardless of membrane character [38]. Moreover, Lee S. et al. suggested considering the consideration of NOM character regarding of NOM regardless of membrane properties [39].

The most severe fouling was observed for the most hydrophobic membrane of the greatest mean pore size i.e., PES, and then for membrane of transient wettability and similar mean pore size i.e., PAN. The least membrane blockage appeared in the case of most hydrophilic membrane of smallest mean pore size i.e., PVDF. The dependence was established regardless of feed water composition. It can be concluded that hydrophobicity/hydrophilicity of membrane, apart from spherical exclusion, also must be considered, whereby the most severe fouling is obtained for hydrophobic membranes.

Zularisam A.W. et al. observed that hydrophobic membranes (polysulphone, polyethersulphone) possessed greater affinity to fouling than hydrophilic membranes [2]. Such a relationship was also determined by Nakatsuka S. et al. and Laine J.M. et al. and it is in agreement with kinetic adsorption test [40–42]. According to this test, the highest degree of adsorption is obtained for hydrophobic membranes regardless of organic fraction character [38].

## 4. Conclusions

Based on the obtained results of the study the following conclusions can be derived:

- pH increase causes less severe fouling during ultrafiltration;
- the decrease of electrostatic interactions and the increase of organic substances adsorption on membrane is observed for low pH; moreover, the impact of pH on fouling depends on hydrophobicity/hydrophilicity of membrane and its pore size distribution;
- high ionic strength (Ca concentration) causes less severe fouling during ultrafiltration;
- the presence of calcium ions favours the filtration cake formation mechanism as a result of particles aggregation; the appearing cake is quite compact but can be removed during back flushing;
- considering the effect of calcium ions on fouling, the properties of the membrane and the other parameters of the feed need to be taken into account cumulatively;
- the most severe fouling is caused by humic substances, then polysaccharides (dextrans) and the lowest is produced by mixture of humic substances, and polysaccharides, which is connected with interactions between membrane - organic substances and, additionally, with intermolecular interactions;
- regardless of the type of organic substances more severe fouling is observed for more hydrophobic membrane.

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