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# PERFORMANCE PREDICTION OF ULTRAFILTRATION TREATMENT OF POST-PROCESS COKE WASTEWATER BASED ON THE ASSUMPTIONS OF HYDRAULIC FILTRATION RESISTANCE MODEL

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**Keywords:** Coking wastewater, pressure membrane technique, ultrafiltration, reverse osmosis, hydraulic model of filtration resistance.

**Abstract:** The post-processes coke wastewater treatment was carried out using flat ultrafiltration membranes with a variable polysulfone concentration in membrane solution (15 wt% – 17% wt.) and variable evaporation time of the solvent from the polymer film surface (0s, 2s, 5s). The ultrafiltration process was carried out with the transmembrane pressure of 0.4 MPa and the linear speed of water flow over the surface of the membrane at 2 m / s. For all the membranes transport characteristic of de-ionized water describing the dependence of the volumetric flow on the transmembrane pressure was done. Since none of the ultrafiltration membranes prepared had provided a sufficiently high degree of pollutants removal from wastewater, it was post-treated by RO method. The wastewater treated this way can be used as technical water for coke quenching. The calculations based on the assumptions of the hydraulic model of filtration resistance allowed to predict the efficiency of ultrafiltration membranes used in the process. To that end, for each of the membranes, the following parameters were determined experimentally: the alterations of effluent stream volume over the time of the low-pressure filtration, the total hydraulic resistance and the resistance constituents such as „new" membrane resistance, the resistance generated by polarization layer and the resistance caused by fouling – reversible and irreversible.

## INTRODUCTION

A need to comply with the standards contained in the Regulation of the Ministry of Environmental Protection on the quality of treated post-processes industrial wastewater discharged into sewers or natural reservoir results in the search for increasingly efficient methods for wastewater treatment. Among the separation techniques, pressure membrane processes are more and more often used in conservation and environmental management. An integrated ultrafiltration and reverse osmosis system used in these studies guaranteed the high pollutant load removal from post-process water treated to such an extent that they could be recycled to the coking technology cycle as technical water for coke quenching.

However, as it is known, during the pressure membrane processes some unfavourable phenomena, such as the decrease of membrane permeability and the independence of permeate flux volume of transmembrane pressure, are observed [1]. They contribute

to the reduction of membrane process efficiency having an adverse impact on its economics. There are many mathematical models that describe the process of permeate flux volume reduction during membrane filtration process carried out for steady and transient operating conditions. The results obtained allowed us to verify the hydraulic model of filtration resistance. The conclusions were based on the measurement of ultrafiltration permeate flux volume changes over time, graphically determined time constant characterizing the decrease in the process efficiency (down to below economic viability) and the experimentally determined resistance values: total membrane resistance, "non-working" membrane resistance and the resistance related to fouling phenomenon and concentration polarization.

## RAW WASTEWATER

The treated primary wastewater came from Coke Plant „Koksownia Częstochowa Nowa" Ltd. Initially it was purified mechanically in order to remove tar substances, oils and solids and then it was subjected to phenol removal processes and gas desorption to remove ammonia.

Table.1. Characteristics of wastewater from Coke Plant "Koksownia Częstochowa Nowa" lp.d

| Determination | Unit | Raw wastewater | Allowable values of sewage pollution which is carried away to the receiver[1)] |
|---|---|---|---|
| pH | - | 9.4 | 6.5–9.0 |
| COD | $mgO_2/ dm^3$ | 4519.6 | 125 |
| TC | $mgC/dm^3$ | 1186.4 | nn. |
| TOC | $mgC/dm^3$ | 963.1 | 30 |
| Ammonium nitrogen $NH_4^+$ | $mg/dm^3$ | 131.6 | 10 |
| Free cyanide | $mg/ dm^3$ | 27.3 | 0.1 |
| Phenol index | $mg/ dm^3$ | 381 | 0.1 |
| Conductivity | µS/cm | 8410.0 | - |

[1)] The Minister of Environment of 28 January 2009 on conditions to be met for the introduction of sewage into the water or the ground, and on substances particularly harmful to the aquatic environment (Journal of law 2006 no. 137 item. 984).

As can be seen, the pollutants' indicators which characterize coke oven effluents are much higher than standardized values and this prevents their direct discharge into a natural receiver.

## POLYSULFONE MEMBRANES

The process of asymmetric polysulfone membranes preparation was carried out under laboratory conditions. In the first stage the film-forming solution was prepared by introducing an appropriate amount of polysulfone (UDEL 1500 U.S. production) into an organic solvent (N, N-dimethylformamide) and then the mixture was stirred for 24 hours.

The conditions for polymer membranes preparation and their composition are summarized in Table 2

Table.2. The composition of polysulfone membranes and their conditions of preparation

| Type of membrane | The composition of the film-forming solution | | Conditions of preparation |
|---|---|---|---|
| | Polymer [g] | Solvent [g] | |
| PSF - 15 | 15 | 85 | - polymer film thickness: 0,2 mm<br>- evaporation time:0s, 2s, 5s<br>- bathing gelling agents: de-ionized water<br>- gelling bath temperature: 281-283 K<br>- active surface membrane: 0,0144 $m^2$ |
| PSF - 16 | 16 | 84 | |
| PSF - 17 | 17 | 83 | |

## TESTING EQUIPMENT

In the process of high-pressure membrane filtration for coke-plant wastewater treatment an apparatus with a slab-type membrane module SEPA CF-NP of American company Osmonics, a sewage tank (8 $dm^3$) with a cooler, rotameter, high-pressure pump and pressure gauges and valves were used. The membrane module consisted of two steel plates with a flat membrane (the surface of the membrane was 144 $cm^2$). The setting operated in cross-flow closed system in which the retentive was recycled to feed tank [2, 4].

## ANALYTICAL METHODS AND ANALYTICAL IDENTIFICATION

Ultrafiltration membranes with varying polysulphone concentrations in film forming solution (15% wt. – 17% wt.) and different times of the solvent evaporation from poured polymer film (0 s – 5 s) were conditioned and then used for the treatment of post-process coke plant water. The transmembrane pressure of the process stood at 0.4 MPa and the linear flow velocity over the membrane surface was 2 m/s [3]. The effectiveness of coke wastewater treatment was evaluated on the basis of the degree of pollution load removal. The following pollution indicators were determined: conductivity, chemical oxygen demand (COD), total organic carbon (TOC), total coal (OW) and the concentration of cyanide and phenol index. For the indication of COD a test method was performed using spectrophotometer HACH DR $4000^{th}$. In the conductivity measurement, conductivity meter of Cole-Palamer Instrument Company was used. The determination of TOC and OW indicators was performed by high temperature catalytic oxidation using GC Multi N/C 2100, while the concentration of cyanide and phenol index was established using cuvette tests of HACH LANGE firm on spectrophotometer DR $2800^{th}$.

Since the post-process coke water after ultrafiltration purification process was still characterized by high values of pollution indicators which prevented their direct discharge to the natural receiver, the water was subjected to a reverse osmosis post-treatment process. In this stage polymer membranes of ADF and SE type provided by Osmonics (U.S.A.) were used. The effectiveness of wastewater post-treatment was assessed, as in the filtration process, basing on the change in pollution indicators' values character-

izing raw and cleaned sewage. The last stage of the study included the verification of the hydraulic model of filtration resistance which allowed to predict the ultrafiltration membranes effectiveness. The studies performed enabled us to calculate experimentally the resizing of permeate fluxes volume in the course of low-pressure filtration, the total hydraulic membrane resistance and the resistance constituents such as „new" membrane resistance, the resistance related with polarization layer and the resistance caused by fouling - reversible and irreversible.

## RESULTS AND DISCUSSION

***Coke wastewater treatment using polysulfone ultrafiltration membrane***

Figure 1 shows the changes in the size of the instantaneous permeate flow volume in the course of coke plant wastewater ultrafiltration process.

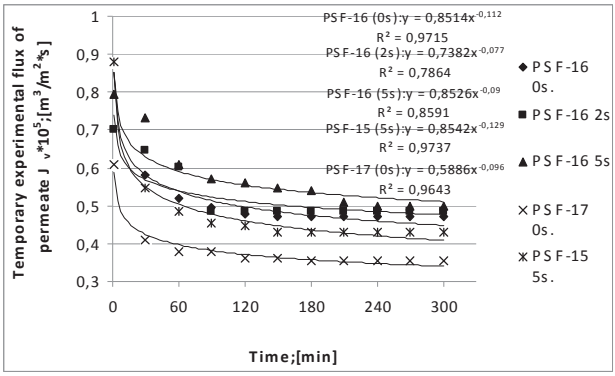


Fig.1. Dependence on temporary volume changes since the permeate flux of low-pressure membrane filtration plant coke

Among the membranes tested the polysulfone membrane with the most compact structure, i.e. 17% weight in the film-forming polymer solution and the solvent evaporation from the membrane surface of 0 s has the lowest permeate flux volume. The obtained equilibrium stream was up to $0.363*10^{-5}$ [$m^3/m^2*s$] after 150 minutes of water filtration. The highest permeate flux had a PSF-16 membrane (5 s) and it was 1.4 times higher than the lowest flux. Coke wastewater subjected to the treatment was characterized by the following indicators: COD-4519.6 $mgO_2/dm^3$, TC-1186.4 $mgC/dm^3$ and TOC-963.1 $mgC/dm^3$. None of the five polysulfone ultrafiltration membranes prepared, as expected, provided sufficiently high degree of pollutant load removal from the treated sewage (Tab. 3). As a consequence, it prevented their direct discharge to the natural receiver [2].

Among the membranes tested PSF membrane-16 (5 s) turned out to be the most profitable. Although the pollution indicators which characterized the sewage treated with this membrane were very close to the values of indicators describing the permeate obtained in the case of PSF membrane-15 (5 s), the equilibrium permeate flux was 15.2% higher.

The coke plant wastewater post-treatment after its initial ultrafiltration cleaning was performed by means of reverse osmosis system with the use of two polymer membranes

Table. 3. The degree of removal of pollution in the process ultrafiltration coke wastewater using polysulfone membranes

| Membrane | Treated wastewater | | | | | |
|---|---|---|---|---|---|---|
| | COD; mg/dm$^3$ | The degree of removal of pollutants [%] | TC; mg/dm$^3$ | The degree of removal of pollutants [%] | TOC; mg/dm$^3$ | The degree of removal of pollutants [%] |
| PSF-16 (0s) | 3926.3 | 13.12 | 850 | 28.35 | 738.5 | 23.32 |
| PSF-16 (2s) | 3577.8 | 20.83 | 883.6 | 25.52 | 767.4 | 20.31 |
| PSF-16 (5s) | 3238 | 28.35 | 772.5 | 34.88 | 613.3 | 36.32 |
| PSF-17 (0s) | 4006.5 | 11.35 | 827.9 | 30.22 | 699.3 | 27.39 |
| PSF-15 (5s) | 3223 | 28.68 | 724.6 | 38.92 | 583.9 | 39.37 |

TC- Total carbon; TOC- Total organic carbon.

ADF and SE manufactured respectively from polyamide and composite with a thin polyamide layer (TF).

Table 4 compares the values of indicators which characterize the sewage treated in the integrated system of ultrafiltration and reverse osmosis system with the use of both osmotic membranes.

Table. 4. Effectiveness of coke wastewater in the system integrated ultrafiltration-reverse osmosis

| Determination | Unit | Raw wastewater | Treated wastewater | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | *UF - PSF-16(5s).* | | *RO - SE* | | *RO-ADF* | |
| | | | Conc. | Degree of pollutants removal [%] | Conc. | Degree of pollutants removal [%] | Conc. | Degree of pollutants removal [%] |
| COD | mg$O_2$/ dm$^3$ | 4519.6 | 3238.0 | 28.3 | 115.2 | 97.5 | 110.3 | 97.6 |
| TC | mgC/dm$^3$ | 1186.4 | 722.5 | 30.2 | 37.9 | 96.8 | 37.6 | 96.8 |
| TOC | mgC/dm$^3$ | 963.1 | 613.3 | 27.4 | 29.8 | 96.9 | 25.5 | 97.4 |
| Ammonium nitrogen $NH_4^+$ | mg/ dm$^3$ | 131.6 | 70.0 | 46.8 | 22.0 | 83.3 | 20.9 | 84.1 |
| Free cyanide | mg/ dm$^3$ | 27.3 | 20.7 | 24.2 | 0.05 | 99.9 | 0 | 100.0 |
| Phenol index | mg/ dm$^3$ | 381 | 355 | 6.8 | 0.1 | 99.6 | 0 | 100.0 |
| Conductivity | mS/cm$^2$ | 8410 | 7420.0 | 11.8 | 0.784 | 90.7 | 0.109 | 87.0 |

TC- Total carbon; TOC-total organic carbon;

As can be deduced from the research results, wastewater post-treated in the reverse osmosis process still did not meet quality standards set out in the Regulation of the Minister of Environment of 28 January 2009, on conditions to be met by the introduction of sewage into the water or soil, and on the substances particularly harmful to the aquatic environment due to the excessive concentration of ammonia nitrogen. A twofold excess of

the volatile ammonia concentration limit in terms of $NH_4^+$ was found. Therefore, before discharging into the natural receiver it should be additionally subjected to a desorption gas process. However, it can be successfully returned to the technological cycle of coke production and used as technical water for coke quenching [2, 5].

***Predicting performance of ultrafiltration membrane in the process of coke wastewater treatment based on the model of hydraulic filtration resistance***

In the course of pressure membrane separation processes it is observed a decrease in permeate fluxes volume over time caused basically by two negative phenomena, i.e. concentration polarization and fouling. They contribute to the emergence of additional resistance posed by membrane transport of substances. The following may also contribute to the lowering of its permeability: pore deformation under too high transmembrane pressure influence (especially in the case of pore membranes), hydrolysis of the polymeric membrane material or change in the filtered solution properties such as viscosity, pH, and osmotic pressure. In the present study attempts were made to predict polysulfone ultrafiltration membrane efficiency in the purification process of coke plant post-process wastewater on the basis of the assumptions of hydraulic model of filtration resistance [6, 7]. It takes into account both the changes in the resistance values posed by a new membrane to a "filtrating medium" and the interaction between the polymer membranes and the substances present in the coke wastewater. The dependence of permeate flux size on transmembrane pressure described by Darcy's equation (1) was used to determine the resistance of individual components of the membrane.

$$J_v = \Delta P/\eta * R_{total.} \quad (1)$$

where:

Jv – temporary volumetric flux of permeate [m3/m2*s],
ΔP – transmembrane pressure [Pa],
η – dynamic viscosity of liquid [Pa*s].
Rtotal – total hydraulic resistance of working membrane [m-1].

It assumes that the total hydraulic resistance of „working" $R_{total}$ membrane is the sum of constituents, which include: "new membrane" resistance and resistance which is a result of concentration polarization and fouling occurring on the membrane surface. The equation (1) can, therefore, be summarized as follows [3, 6, 7]:

$$J_v = \Delta P/\eta * (R_m + R_f + R_{cp}) \quad (2)$$

where:

$R_m$ – membrane resistance value of the „new" membrane [$m^{-1}$],
$R_{cp}$ – polarization layer resistance [$m^{-1}$],
$R_f$ – resistance due to the fouling phenomenon [$m^{-1}$].

Non- working membrane resistance value ($R_m$) was determined from the relation (1) that describes the transport of de-ionized water over time ($J_{H2O} = f(t)$. Since in these process conditions $R_{total} = R_{m,}$ after the transformation of equation (1) we obtain the following relationship:

$$R_m = \Delta P/\eta \times J_{H20} \quad (3)$$

where:

$J_{H2O}$ – experimental temporary flux of de-ionized water [$m^3/m^2*s^{-1}$],
$\eta$ – dynamic viscosity of water at $20^0$C.

Resistance caused by fouling phenomenon involves the deposition of substances existing in the filtered medium on the membrane surface and/or in the pores of the substances presented in the filtered medium. It consists of the resistance caused by adsorption inside the membrane pores, resistance caused by gel layer resistance and the resistance resulting from the formation of a filter cake on the membrane surface, a so-called secondary diaphragm. It can, therefore, be described by the equation:

$$R_f = R_a + R_{gel} + R_p \quad (4)$$

where:

$R_a$ – resistance to the phenomenon of adsorption [$m^{-1}$],
$R_{gel}$ – generated resistance to gel layer [$m^{-1}$],
$R_p$ – resistance to the formation of secondary membrane [$m^{-1}$].

As it is known, fouling can be reversible or irreversible. In the first case, membrane cleaning provides its initial performance restoration, whereas in the second, it is impossible. It was assumed that the resistance caused by fouling phenomenon is the sum of both reversible and irreversible fouling, therefore [6, 7]:

$$R_f = R_{fn} + R_{fo} \quad (5)$$

where:

$R_{fo}$ – resistance to reversible fouling [$m^{-1}$],
$R_{fn}$ – resistance to irreversible fouling [$m^{-1}$].

The resistance generated by irreversible fouling is a result of permanent membrane pores blocking so that it is difficult to reconstruct its initial performance. To determine its value there was specified the size of the de-ionized water volumetric flux for the membrane after ultrafiltration coking wastewater treatment.

$$R_{fn} = (\Delta P/\eta * J_{pH2O}) - R_m \quad (6)$$

where:

$R_{fn}$ – resistance to irreversible fouling [$m^{-1}$],
$J_{pH2O}$ – experimental temporary flux of de-ionized water after pressure filtration plant, [$m^3/m^2*s$].

To determine the value of the experimental reversible fouling resistance of polysulfone membranes, the following formula should be used:

$$R_{fo\ exp.} = R_{total} - R_m - R_{fn} \quad (7)$$

A theoretical resistance value connected with reversible fouling was determined using the equation [3]:

$$\frac{d}{dt}\left(R_{\infty}-R_{fo}\right)+\frac{1}{t_{Ro}}\left(R_{\infty}-R_{fo}\right)=0 \tag{8}$$

After integrating the following equation is obtained [3]:

$$R_{fo}=R_{\infty}\left[1-\exp(-\frac{t}{t_{Ro}})\right] \tag{9}$$

where:

$R_{fo}$ – initial resistance fouling reversible ($R_{fo}$=0 at t=0) [$m^{-1}$],
$R_{\infty}$ – resistance fouling reversible after an infinitely long period of time [$m^{-1}$],
$t_{Ro}$ – time constant [$min^{-1}$].

The establishment of $t_{Ro}$ permanent allowed for rearrangement of the equation (9) to form [3]:

$$\frac{R_{\infty}-R_{fo}}{R_{\infty}}=\exp(-\frac{t}{t_{Ro}}) \tag{10}$$

After logarithming the equation of a straight line that passes through the origin of the system of coordinates was obtained. From the straight line inclination an coefficient, which allowed to calculate the theoretical reversible fouling resistance was determined.

Table 5 and Figure 2 show the examples of the graphical determination of time constants $t_{R0}$ for the prepared membranes.

Table 5. Determined values of time constants, and simple equations for the ultrafiltration polysulfone membrane

| Membrane | Regression equation in the forms of the linear equation | | Values of time constants |
|---|---|---|---|
| | y=ax+b | $R^2$ | $t_{R0}$;[min] |
| PSF-16 0s | y=-0.0039x-1.115 | 0.97 | 179 |
| PSF-16 2s | y=-0.0055x-1.45 | 0.99 | 182 |
| PSF-16 5s | y=-0.0056x-1.501 | 0.985 | 250 |
| PSF-17 0s | y=-0.0051x-1.984 | 0.987 | 196 |
| PSF-15 5s | y=-0.006x-1.6 | 0.966 | 179 |

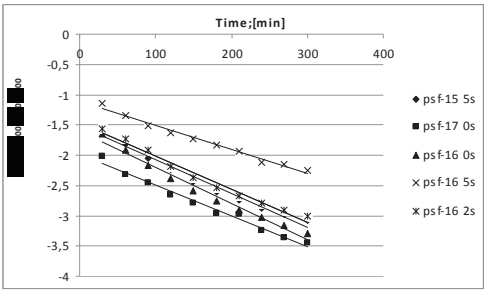


Fig.2. Time constants $t_{R0}$ designated for ultrafiltration membrane

Figure 3 presents the comparison between the experimental resistance values connected with the reversible fouling phenomenon obtained in the low-pressure filtration of coke plant effluent and the resistance of the theoretical reversible fouling values determined from the equation (9). Figure 4 illustrates the comparison between total membrane resistance, „new" membrane resistance and the resistance related to the fouling phenomena.

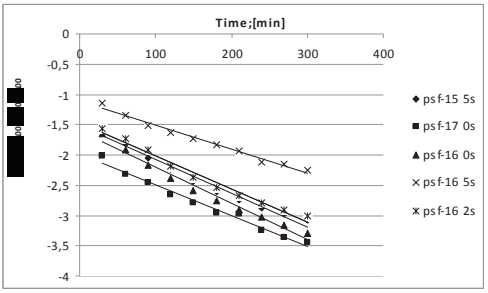


Fig.3. Time constants $t_{R0}$ designated for ultrafiltration membrane

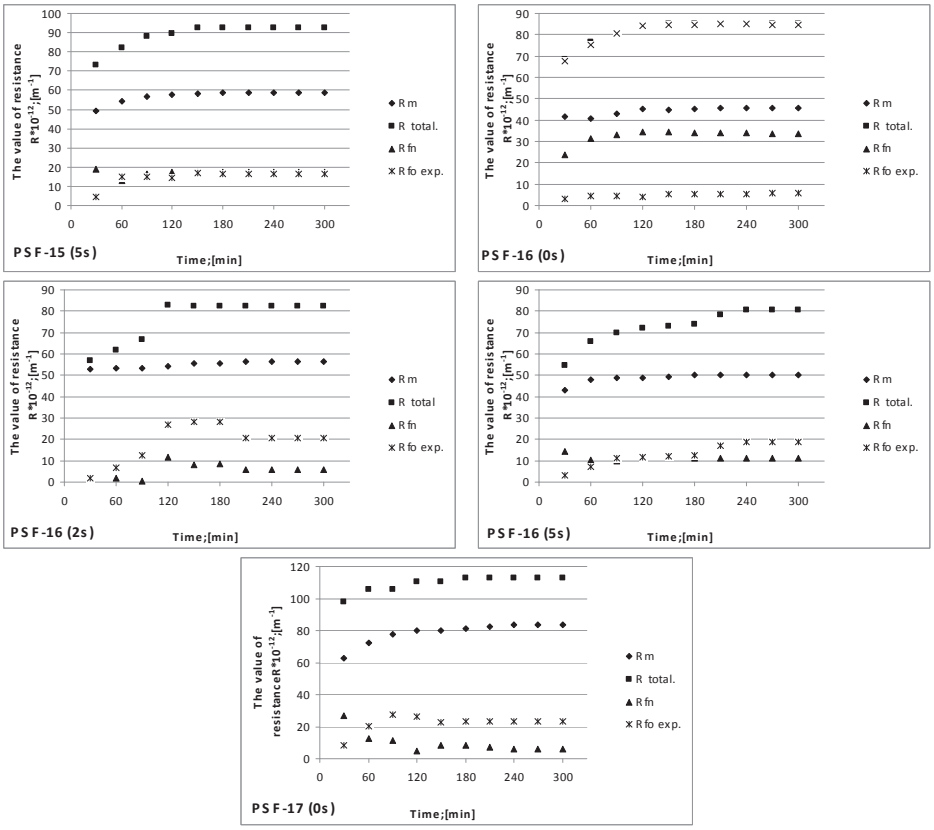


Fig. 4 Comparison of changes in resistance R total., Rm, Rfn, Rfo.exp. polysulfone membrane flat during treatment the post-trial ultrafiltration coke wastewater

The last of the designated resistance components is the layer polarization resistance. The phenomenon of concentration polarization is the formation of the boundary layer solution with a concentration above the average of the feed solution in the immediate membrane vicinity. It contributes to decrease of membrane productivity and changes in the membrane separation properties. The following relationship [7] describes the resistance induced by generated polarization layer:

$$Rp = \phi \Delta P \tag{11}$$

Where:

$\phi$ – resistance index characterizing the ability of mass transport through the membrane [s/m].

The value of resistive index can be calculated from the following mathematical relation [6]:

$$\phi = 1/J_{lim} \tag{12}$$

for the following limit values:

$\Delta P = 0$, $J = 0$;

$\Delta P \rightarrow \infty$, $J = J_{lim}$

where:

$J_{lim}$ – temporary volumetric flux of permeate [$m^3/m^2*s$].

Table 6 summarizes the values of resistance indices for the polysulfone membranes. Figure 5 shows the dependence of the resistance values changes for polarizing layer of ultrafiltration membrane on time of coke pre-processed water ultrfiltration treatment.

Table 6. The values of resistance indices for the polysulfone membranes

| Time;[min] | Membrane | | | | |
|---|---|---|---|---|---|
| | PSF-15 ( 5s) | PSF-16 (0s) | PSF-16 (2s) | PSF-16 (5s) | PSF-17 (0s) |
| | Resistance index $*10^5$;[s/m] | | | | |
| 30 | 1.822 | 1.715 | 1.425 | 1.364 | 2.446 |
| 60 | 2.057 | 1.919 | 1.543 | 1.647 | 2.645 |
| 90 | 2.196 | 2.024 | 1.663 | 1.751 | 2.645 |
| 120 | 2.234 | 2.092 | 2.232 | 1.802 | 2.757 |
| 150 | 2.314 | 2.123 | 2.232 | 1.825 | 2.757 |
| 180 | 2.314 | 2.123 | 2.232 | 1.851 | 2.817 |
| 210 | 2.314 | 2.123 | 2.232 | 1.963 | 2.817 |
| 240 | 2.314 | 2.123 | 2.232 | 2.009 | 2.817 |
| 270 | 2.314 | 2.123 | 2.232 | 2.009 | 2.817 |
| 300 | 2.314 | 2.123 | 2.232 | 2.009 | 2.817 |

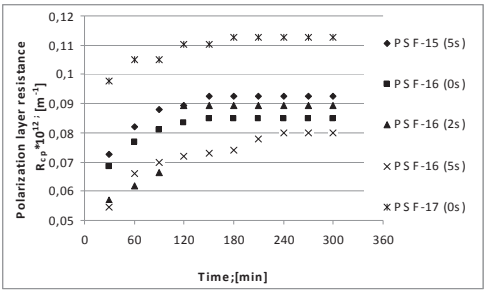


Fig.5. Dependence of the resistance values changes for polarizing layer of ultrafiltration membrane on time of coke pre-processed water ultarfiltration treatment

It is clear that for all ultra-filtration membranes tested the layer polarization resistance values are several orders of magnitude smaller in comparison to other resistance constituents and slightly increase in the course of low-pressure membrane filtration. Then, using the determined experimentally resistance constituents' values, theoretical values of total polysulfone membrane resistance were calculated (Equation 2). This enabled the determination of theoretical volumetric values of ultrafiltration permeate fluxes obtained in coke wastewater ultrafiltration treatment. Figure 6 presents the comparison between theoretical permeate fluxes calculated from the hydraulic model of filtration resistance and the size of temporary fluxes determined experimentally.

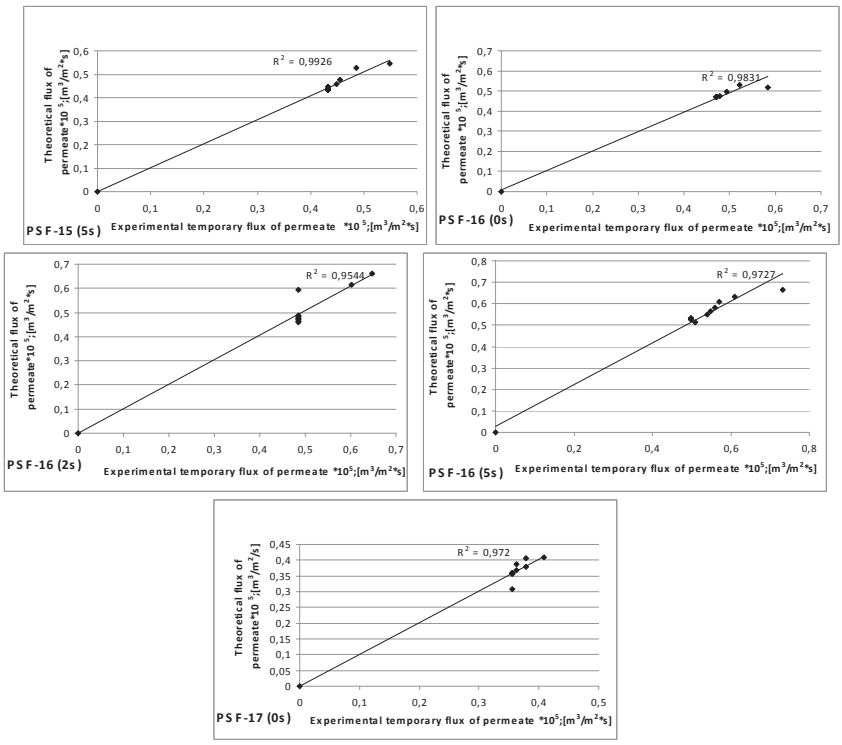


Fig. 6. Comparison between theoretical permeate fluxes calculated from the hydraulic model of filtration resistance and the rates of temporary streams determined experimentally.

The high correlation coefficients suggest that the hydraulic model of filtration resistance used in the calculation allows to predict the size of the instantaneous permeate flux in the process discussed.

## CONCLUSIONS

The sequential ultrafiltration and reverse osmosis membrane systems used for post-trial coke plant water treatment did not provide an adequate level of pollutant removal due to too high concentration of ammonia in treated sewage. Before discharging into the natural receiver, it should be subjected to additional gas desorption process. It can be successfully returned to the technological circulation as technical water for coke quenching.

Among the prepared polysulfone ultrafiltration membranes with different structure compactness, the PSF-16 membrane had the best transport and distribution properties. However, since none of the membranes tested provided the required degree of pollutant load removal from the treated coke sewage it was thoroughly cleaned by the means of reverse osmosis system. Due to the excessive concentration of ions $NH_4^+$ in the permeate after reverse osmosis process (22.0 and 20.9 mg/dm$^3$-SE mg/dm$^3$-ADF), post-process purified coke water cannot be discharged into the natural receiver but may be returned to technological cycle of the coking plant.

High values of correlation coefficients obtained by comparing the instantaneous experimental ultra-filtration permeate streams to the instantaneous theoretical streams suggest that the hydraulic model of filtration resistance used in the calculation allows to forecast the membranes performance in the process discussed.

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