



## The use of ultrafiltration in enhancement of chemical coke oven wastewater treatment

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### ABSTRACT

Coke oven wastewater is one of the most contaminated and toxic aqueous stream generated in thermal coal processing systems. It contains a significant amount of organic and inorganic pollutants, among which substances well recognized as environmental and living organisms toxicants can be found, that is, polyaromatic hydrocarbons, cyanides and sulphides. In the conventional coke oven treatment system, these contaminants should be eliminated from the stream at the chemical wastewater treatment site. However, due to operational limitations, a part of the compounds remains in the stream, which is introduced to further biological treatment, which may lead to the inhibition of biological processes. The main goal of the presented research was to investigate the enhancement of chemical treatment loop operation by means of ultrafiltration. Three types of polymeric, polyethersulphone membranes differed in cut-off equal to 20, 10 and 5 kDa (by Synder) were tested towards efficiency of complex cyanides and chemical oxygen demand (COD) removal at simultaneous monitoring of the capacity and the fouling affinity. The impact of transmembrane pressure and membrane cut-off on the process performance was checked. The evaluation of processes was made on the basis of flux stability, fouling intensity, complex cyanides and COD removal rates. The studies revealed that ultrafiltration process enabled to remove complex cyanides up to 75%, whereas COD was decreased by 27%. The satisfactory recovery of membranes initial capacity reaching 95% was observed.

*Keywords:* Coke oven wastewater; Chemical treatment; Complex cyanides separation; Ultrafiltration

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### 1. Introduction

Coke oven liquor generated during coking of coal is one of the most hazardous aqueous stream generated by thermal coal processing [1]. It is a highly loaded stream contaminated with tars, cyanides, sulphides, ammonia and phenols, the proper treatment and utilization of which is of the highest importance considering sustainable and environmentally acceptable coke production [2,3]. The coke oven liquor is originally generated during coke oven gas cooling [4], and after tars separation, eventual involvement in coke oven gas desulphurization and ammonia stripping [5], it becomes the influent of coke oven wastewater treatment plant (Fig. 1) [6,7].

Due to the presence of a number of contaminants in coke oven wastewater treatment plant influent, the stream treatment is performed with the use of chemical and biological processes [8], which may be supported by a preliminary mechanical treatment (e.g. sand filters) [9] or a final polishing, for example, coagulation, advanced oxidation or activated carbon adsorption [10,11]. The basic scheme of a coke oven wastewater treatment plant is presented in Fig. 2.

The proper operation of the biological loop of the plant depends on the efficient removal of inhibitors carried out at the chemical treatment site of the plant [12]. For the elimination of sulphides, cyanides and residual tars, coagulation with the use of iron-based coagulants is performed [13]. The elimination of organics is carried out based on the conventional coagulation mechanism [14], the removal

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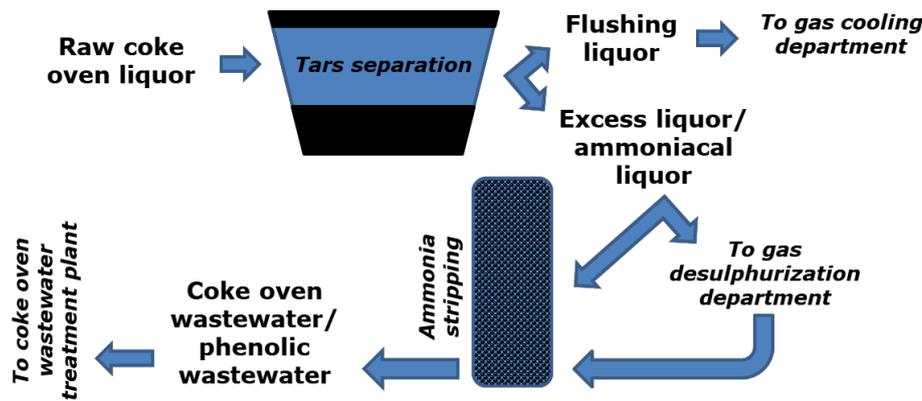


Fig. 1. The pathway of coke oven liquor processing and coke oven wastewater formation.

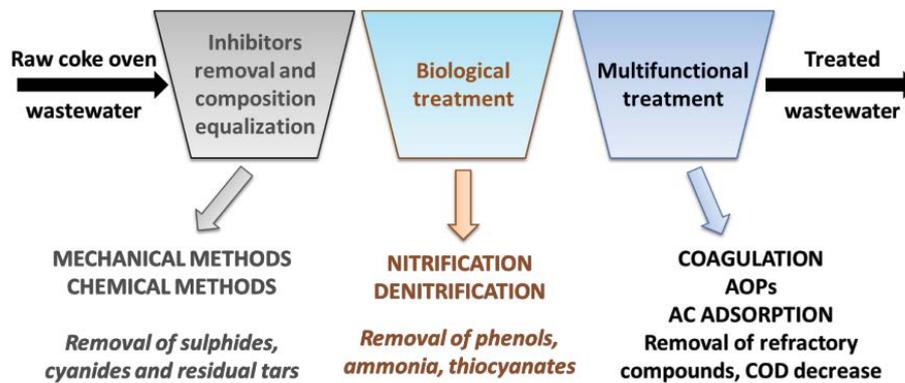
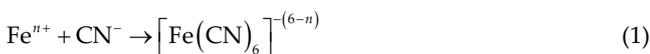
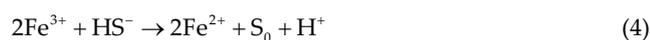


Fig. 2. Conventional coke oven wastewater treatment plant arrangement.

of cyanides is based on complexation and chemisorption/precipitation [15], while the elimination of sulphides is based on precipitation of iron sulphide [16]. The simplified mechanisms of the discussed elimination reactions of sulphides and cyanides are shown in Eqs. (1)–(6) as follows:



where  $\text{FeX}_{(s)}$  corresponds to iron-derived structures, which are formed during coagulant hydrolysis.



The removal of sulphur species (sulphides  $\text{S}^{2-}$ , bisulphides  $\text{HS}^-$  and polysulphides  $\text{S}_x^{2-}$ ) by means of reactions with iron ions of different valence is the efficient process, while the complexation (Eq. (1)) and especially precipitation of

cyanides is rather difficult (Eqs. (2) and (3)) [17]. It has been found that formation of iron-hexacyanoiron precipitates (products of reaction (2)) occurs in acidic environment, thus in basic environment of coke oven wastewater ( $\text{pH} > 9$ ), the elimination of complexes from aqueous phase is possible due to reaction of complexed cyanides with iron-derived flocks formed during coagulation (Eq. (3)) [18]. On the other hand, this reaction pathway (3) enables only partial elimination of hexacyanoiron complexes formed in reaction (1). Thus, these complexes inflow to biological reactors and have the ability to cumulate within activated sludge structures/flocks [19]. Additionally, the sensitivity of complex cyanides to photodegradation and their discussable stability in aqueous systems is dangerous to further biological treatment. At a high concentration (due to the accumulation), the decomposition of complex cyanides accompanied by the release of free cyanides, which are highly toxic to activated sludge microorganisms occurs and the biological treatment may be significantly inhibited or even completely stopped [20].

Membrane separation has already been tested in treatment of coke oven wastewater at various treatment stages and in the combination with different unit operations. Due to simple operation, ability to remove a wide range of contaminants and satisfactory chemical resistance, membrane processes seem to offer a promising alternative and/or support to industrial wastewater treatment, including

coke-making industry [21–23]. Mielczarek et al. [24] tested ultrafiltration (UF) for raw coke oven wastewater treatment, and the process was used as a direct filtration or in the combination with coagulation [24]. The self-prepared polymeric membrane was applied, while for coagulation commercial coagulant PIX113 (ferric sulphate acidic solution) was used. The studies revealed 15% elimination of free cyanides and 30% reduction of chemical oxygen demand (COD) by direct UF process, whereas in the combined system 50% elimination of free cyanides and 81% decrease in COD were achieved. The same authors tested a number of commercial UF membranes for the direct treatment of raw coke oven wastewater [25]. HZ15, PVDV, PW and DS-GM membranes made of polysulphone, polyethersulphone (PES), thin film and polyvinylidene, respectively, of cut-off 20, 10–12, 8 and 30 kDa, respectively, were used. The studies revealed that direct UF enabled to decrease COD of the wastewater by 11% (HZ15) to 40% (DS-GM), while the removal of free cyanides varied from 4% (HZ15) to 7% (DS-GM).

Jin et al. [8] involved UF-based membrane bioreactor (MBR) for polishing of regular coke oven wastewater treatment biological loop effluent. Four modules of flat sheet polyvinylidene fluoride membranes of pore size 0.1  $\mu\text{m}$  (by Shanghai Sevoo, China) were used. The study revealed that application of the MBR enabled additional decrease of COD by 26%, whereas the removal of total cyanides was poor and reached 10% (4.6 mg/L in MBR influent and 4.1 mg/L in MBR effluent).

Pimple et al. [26] tested MBR/RO (reverse osmosis) and UF/RO system for coke oven wastewater treatment. The treatment was evaluated for the removal of cyanide, phenol and COD. The MBR was found to be more sufficient as it showed a higher treatment efficiency through aeration-enhanced biodegradation and, additionally, the determined SDI was <2.3. The final rejection of cyanide in the RO permeate was above 90%, phenol was above 95% and total suspended solids was 100%.

It has been found that membrane filtration should be suitable for polishing of effluent from chemical loop of coke oven wastewater treatment, and its main goal is to reject cyanide complexes and high-molecular weight organic compounds (ca. polyaromatic hydrocarbons and tars) from the stream, which is further directed to biological treatment. Thus, the main goal of the presented research was to test different UF membranes towards the enhancement of chemical coke oven

wastewater treatment and to evaluate the efficiency of the process in regard to capacity, fouling affinity and selected contaminants (complex cyanides, COD) removal rates.

## 2. Experimental

### 2.1. Membrane filtration

The laboratory scale installation for membrane filtration, KMS Cell CF 1 (produced by Koch Membrane Systems, USA) (Fig. 3), operated in a cross-flow mode was used for chemically treated coke oven wastewater treatment. The installation was equipped with the feed tank of a volume of 0.5  $\text{dm}^3$  and a flat-sheet membrane with an effective separation area of 28  $\text{cm}^2$ . The permeate was continuously collected outside the membrane module, while the retentate was recirculated to the feed tank.

During experiments, three types of PES UF membranes (by Synder Filtration, USA), that is, SM, ST and MT with corresponding molecular weight cut-off (MWCO) equal to 20, 10 and 5 kDa were used. The filtration of chemically treated coke oven wastewater was preceded with the characterization of membranes' transport properties by determining a dependence of deionized water volumetric flux on a transmembrane pressure (TMP) in the range of 0.1–0.3 MPa.

Real coke oven wastewater after chemical loop was filtered at a TMP of 0.1–0.3 MPa until 80% of initial feed volume was recovered in the form of permeate. After wastewater filtration, the deionized water flux was established for membranes neither chemically nor hydraulically cleaned in order to evaluate the impact of membrane fouling on the process capacity.

The volumetric flux across the membrane was calculated based on the measured volume of collected permeate according to Eq. (7) as follows:

$$J = \frac{V}{A \times \Delta t} \quad (7)$$

where  $J$  – volumetric permeate flux ( $\text{L}/\text{m}^2 \text{ h}$ );  $V$  – permeate volume collected over  $\Delta t$  period (L);  $A$  – membrane effective separation area ( $\text{m}^2$ ) and  $\Delta t$  – time of permeation and sample collection (h).

Relative fluxes of coke oven wastewater through new conditioned membrane and of deionized water after real

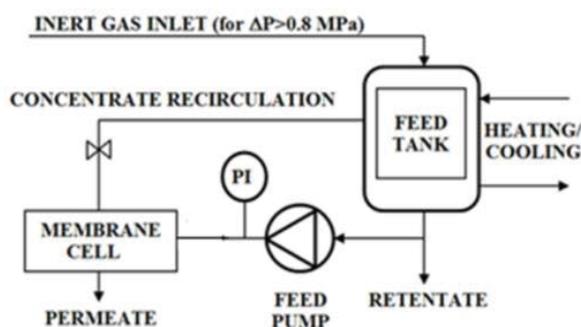


Fig. 3. KMS Cell CF1 laboratory-scale membrane filtration unit.

sample filtration were calculated according to Eqs. (8) and (9) as follows:

$$\alpha_p = \frac{J_p}{J_0} \quad (8)$$

$$\alpha_D = \frac{J_D}{J_0} \quad (9)$$

where  $J_p$  – volumetric flux of coke wastewater ( $L/m^2 h$ );  $J_D$  – volumetric flux of deionized water after real sample filtration ( $L/m^2 h$ );  $J_0$  – volumetric flux of deionized water established for new membrane ( $L/m^2 h$ );  $\alpha_p$  – relative permeate flux (–)  $\alpha_D$  – relative deionized water flux (–).

## 2.2. Analytical methods

The feed directed to UF separation as well as permeate samples were analysed for COD determined by a spectrophotometric method using HACH Lange procedures at DR-6000 spectrophotometer and for total cyanides concentration determined by means of modified DIN38405-13 used for sample preparation followed by ion chromatography analysis with pulsed amperometric detection. The removal rate of examined contaminants (COD and total cyanides) was calculated on the basis of Eq. (10) as follows:

$$R = \left( 1 - \frac{C_{p,i}}{C_{f,i}} \right) \times 100\% \quad (10)$$

where  $R$  – removal rate of  $i$  contaminant (%),  $C_{p,i}$  – concentration of  $i$  contaminant in the permeate (mg/L) and  $C_{f,i}$  – concentration of  $i$  contaminant in the feed (mg/L).

## 3. Results

### 3.1. Membrane characteristics

Transport properties of polymeric PES membranes were evaluated on the basis of the TMP dependency of deionized water volumetric flux in the range of 0.1–0.3 MPa (Fig. 4).

The results indicated on typical, linear increase of the deionized water flux with TMP in case of all tested membranes. On the other hand, membranes of cut-off 10 (ST) and 5 kDa (MT) characterized with the same capacity revealed for deionized water. Such results can be explained by either membrane porosity (number of pores in a unit area of a membrane) or a pore size distribution. Membranes with greater number of smaller pores may exhibit similar performance to a membrane, which possess less transportation channels, but of bigger average diameter.

### 3.2. Wastewater filtration

The operations of particular membranes at different TMPs from 0.1 to 0.3 MPa are shown in Figs. 5–7.

It was observed that at 0.1 MPa pressure the capacity of the process with SM-20 kDa membrane was the lowest, but on the other hand the most stable one (Fig. 5). The increase of the TMP resulted in the capacity increase, however, the flux decline in time was observed. It indicated on the occurrence of membrane fouling caused by penetration of membrane pores by contaminants present in the feed stream.

Filtrations at ST-10 kDa membrane showed that the process was stable at 0.1 and 0.2 MPa pressure, while in 0.3 MPa pressure the flux declines indicating on the membrane fouling occurred (Fig. 6). Nevertheless, the increase of pressure from 0.1 to 0.2 MPa resulted in the increase of the membrane capacity, while in case of further pressure increase to 0.3 MPa the capacity improvement was not observed, which also confirmed the affection of the process performance by fouling. On the other hand, the comparison of results obtained for ST membrane with ones obtained for SM membrane revealed the better operation of the former membrane, as at 0.2 MPa pressure the filtration was stable and the capacity was higher.

The performance of filtration using MT 5 kDa membrane indicated on the stability of the process at 0.1 and 0.2 MPa pressure, while at 0.3 MPa the temporary increase in permeate flux followed by the capacity decrease was observed (Fig. 7). The latter indicated on the deposition of contaminants on the membrane surface, which were periodically washed out and started to deposit again. The comparison of results obtained

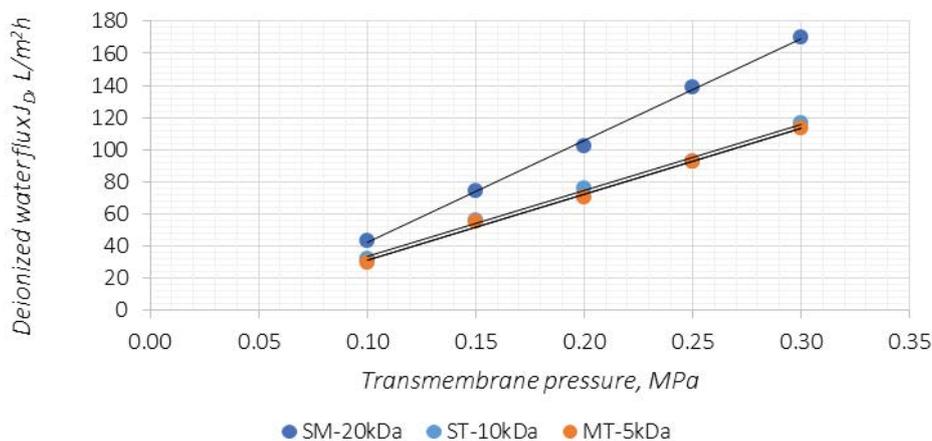


Fig. 4. Deionized water flux determined for tested PES membranes as a function of transmembrane pressure in the range of 0.1–0.3 MPa.

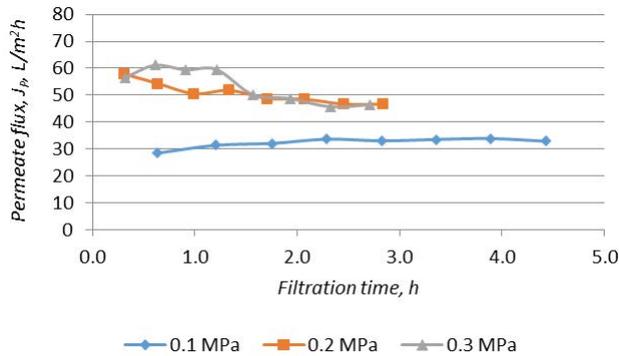


Fig. 5. Permeate fluxes observed during filtration using SM-20 kDa membrane.

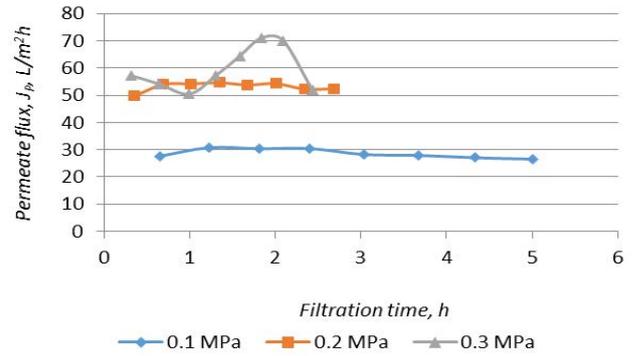


Fig. 7. Permeate fluxes observed during filtration using MT-5 kDa membrane.

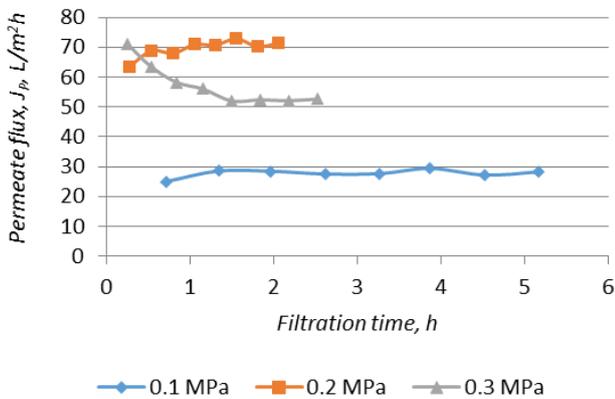


Fig. 6. Permeate fluxes observed during filtration using ST-10 kDa membrane.

and the longest duration necessary to achieve the assumed volume of permeate were observed. UF using SM membrane was also characterized with the most significant drop of capacity expressed as the ratio of permeate flux observed at the beginning of the filtration to final permeate flux, which reached 14%. On the other hand, ST membrane, revealed the shortest duration and the highest average permeate flux. Hence, it indicated that it would be the preferably used membrane.

3.3. Fouling

The filtration results had to be referred to fouling membrane affinity during process, thus, relative permeate fluxes at applied TMPs were determined (Fig. 9).

Obtained values of relative permeate fluxes indicated that filtration using MT 5 kDa membrane characterized with the lowest impact of membrane fouling, while it was ST membrane which is characterized with the highest capacity. Moreover, it was observed, that fouling severeness was the highest at the highest TMP regardless of tested membrane type. Thus, in order to indicate on the most preferable membrane for filtration of chemical loop effluent, the process capacity results had to be referred to capacity recovery results

for all tested membrane indicated that 0.2 MPa pressure was the preferable one, hence in Fig. 8 the presentation of results obtained for all membranes at this value of the parameter is shown.

It was noticed that despite the highest MWCO of SM membrane, it was the one, for which the lowest capacity

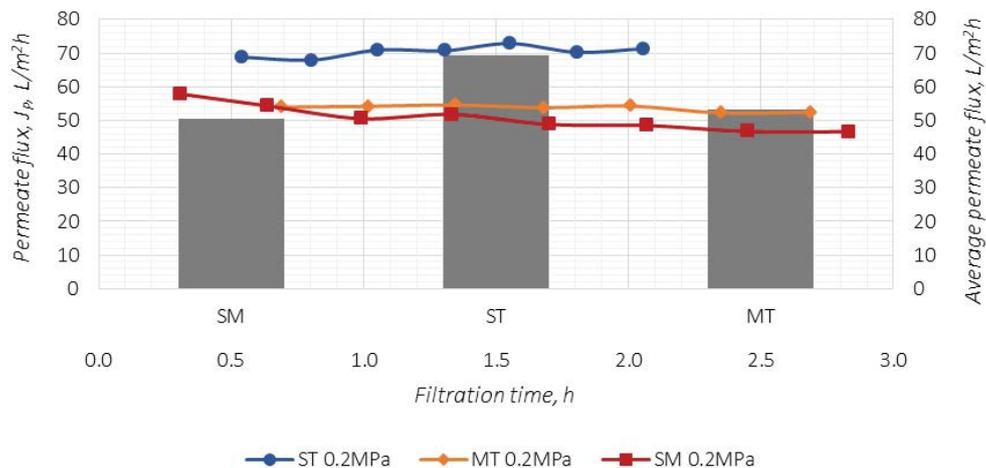


Fig. 8. The change of permeate flux with filtration time and its average value for chemical loop effluent ultrafiltration (TMP of 0.2 MPa).

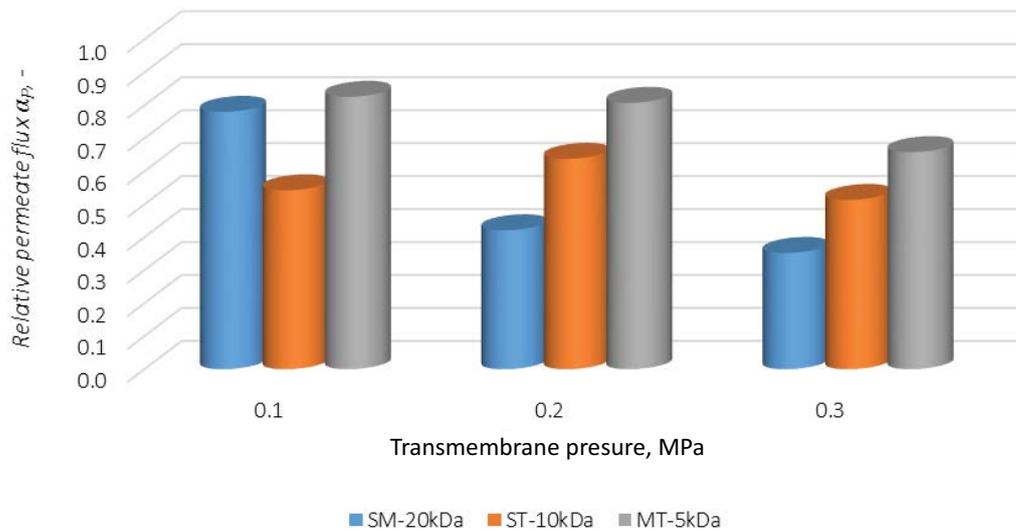


Fig. 9. Relative permeate fluxes  $\alpha_p$ , determined for membranes applied in the treatment.

and permeate quality analyses. In Fig. 10, relative deionized water fluxes determined for membranes tested during chemical effluent filtration are presented.

Relative deionized water fluxes determined for MT 5 kDa membrane were the highest ones and exceeded 85% regardless of the TMP applied in UF process (Fig. 10). Obtained results enabled to conclude that fouling phenomenon occurred during the filtration was the most severe in case of ST membrane, since calculated relative deionized water fluxes were the lowest ones (Fig. 10). Thus, the results on process capacity, fouling affinity and capacity recovery indicated that MT membrane would be the best for the stream filtration.

### 3.4. Quality of permeate

The efficiency of the process towards the removal of contaminants was evaluated according to the rate of removal of cyanides, which remained in the stream after chemical

treatment, and to organic compounds removal indicated as COD. In Figs. 11 and 12 total cyanides (which correspond to complex cyanides – free cyanides in the feed water were not detected) and COD removal rates, respectively, are presented.

The most effective rejection was observed for iron-cyanide complexes indicated as  $CN_{TOT}$  removal rate of which varied from 45.4% to 76.7% (Fig. 11). The filtration at SM and ST membrane at 0.3 MPa pressure enabled to decrease the contaminant concentration from 14 to <4 mg/L, while for MT membrane it was <5 mg/L at 0.2 and 0.3 MPa. The better removal of contaminant observed at higher pressure for wider membranes was probably due to the pores blocking and narrowing of their diameter. Generally, removal of  $CN_{TOT}$  using tested PES membranes increased with the TMP increase. It was assumed that two different mechanisms of separation could be taken into consideration, that is, the size rejection and electrostatic interactions – negative charge of PES membrane could contribute to the rejection of negatively charged

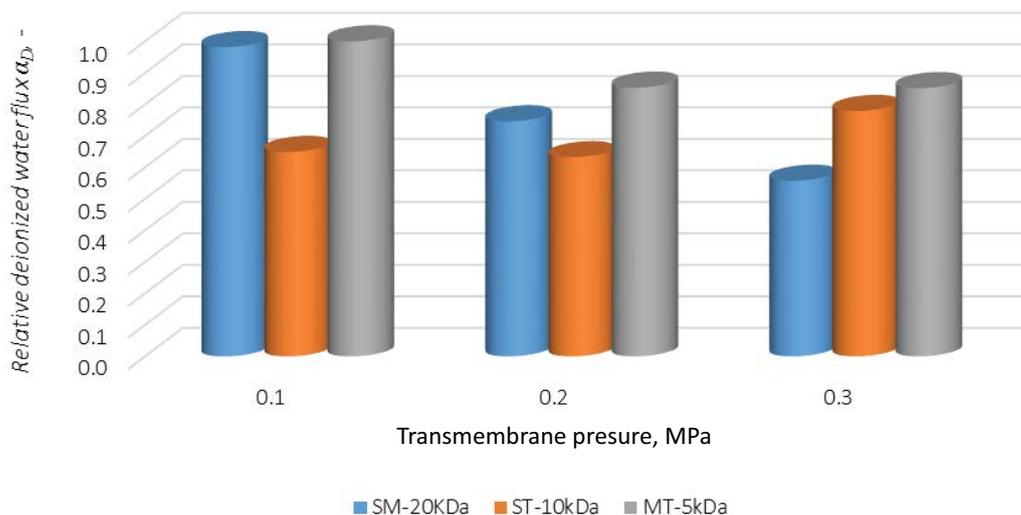


Fig. 10. Relative deionized water flux determined for membranes applied in treatment of chemical site effluents.

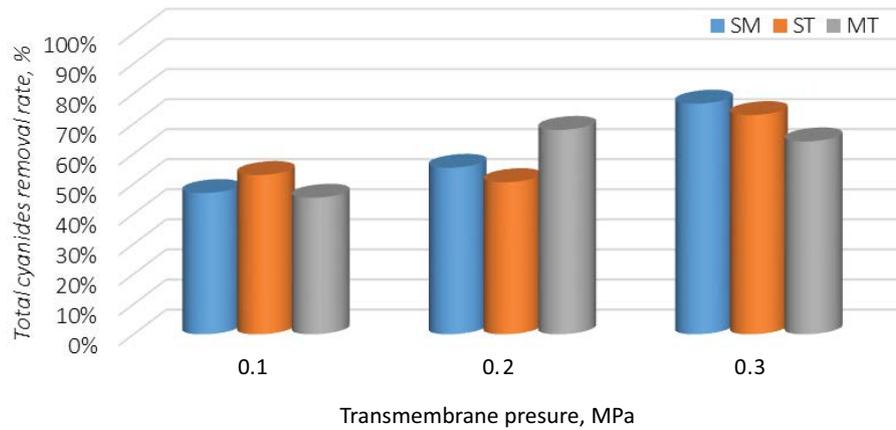


Fig. 11. Total cyanides removal by UF process with PES membranes.

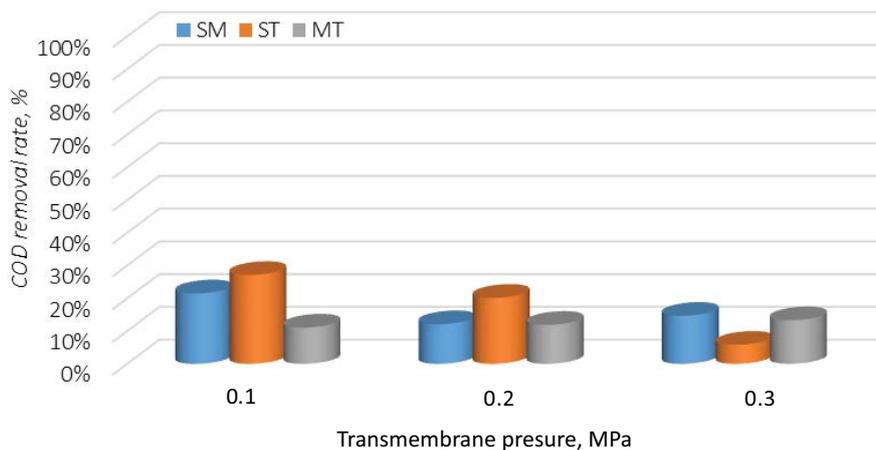


Fig. 12. COD removal by UF process with PES membranes.

iron-cyanide complexes. On the other hand, COD reduction was found to be very poor and it did not exceed 27.1% for ST membrane for TMP of 0.1 MPa (Fig. 12). It was assumed, that the size of uncharged organic contaminants present in the feed stream was below MWCO of tested membranes. Finally, it was concluded that among all tested membranes, the treatment of chemical site effluents at MT membrane would be preferable due to its capacity, lowest fouling affinity and contaminants removal efficiency. The obtained COD removal rates were compared with ones observed by other authors, who applied low pressure-driven membrane filtration to direct or in combination with coagulation raw coke oven wastewater treatment [23,24]. It was found, that COD elimination (27.1%) was in agreement with other results (30% [23] and 11%–40% [24]).

#### 4. Conclusions

- Three types of polyethersulphone membranes of MWCO 20, 10 and 5 kDa were applied in UF of chemical treatment effluents.
- Membranes characterized with the smallest MWCO

(5 kDa), exhibited the best permeate capacity and were the least vulnerable to fouling phenomenon resulting of pore blocking.

- In case of more open membranes (10 and 20 kDa), fouling was caused by deposition of contaminants inside membrane pores making the capacity recovery process less effective.
- Evaluation of contaminants removal efficiencies indicated that UF process could be successfully combined with chemical treatment not only to prevent membrane from adverse fouling, but also to remove iron-cyanide complexes.
- The obtained reduction rate of total cyanides varied from 46% to 75%. Removal of organic contaminants expressed as COD was less efficient (27%) indicating that most of the contaminants were below MWCO of tested membranes.
- Among all tested membranes, the treatment of chemical effluents at MT membrane of cut-off 5 kDa would be preferable due to its capacity, lowest fouling affinity and contaminants removal efficiency.

## Symbols

$V$	–	Permeate volume collected over $\Delta t$ period, L
$A$	–	Membrane effective separation area, m <sup>2</sup>
$\Delta t$	–	Time of permeation and sample collection, h
$J_p$	–	Volumetric flux of coke wastewater, L/m <sup>2</sup> h
$J_D$	–	Volumetric flux of deionized water after real sample filtration, L/m <sup>2</sup> h
$J_0$	–	Initial volumetric flux of deionized water, L/m <sup>2</sup> h
COD	–	Chemical oxygen demand, mg O <sub>2</sub> /L
MWCO	–	Molecular weight cut-off, kDa
TMP	–	Transmembrane pressure, MPa
$\alpha_p$	–	Relative permeate flux, –
$\alpha_D$	–	Relative deionized water flux, –
$R$	–	Removal rate, %
$C_{p,i}$	–	Concentration of $i$ contaminant in the permeate, mg/L
$C_{f,i}$	–	Concentration of $i$ contaminants in the fee, mg/L

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