No. 7

COKING PLANT WASTEWATER TREATMENT IN INTEGRATED SYSTEMS COMBINING VOLUME COAGULATION AND ADVANCED OXIDATION WITH PRESSURE MEMBRANE TECHNIQUES

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The article compares the efficiencies of coke plant wastewater treatment in the integrated systems that combine physicochemical and chemical purification methods with ultrafiltration and reverse osmosis. The wastewater treated came from the "Coke Plant Czestochowa New". In the process of volume coagulation the coagulant used was sulfate (VI) iron (III)-PIX-113, with the dosage of 400 mg/dm³ and pH was 9. In the advanced oxidation Fenton's reagent was applied, with the concentration of iron equal to 0.8g/dm³ and hydrogen peroxide 2.4 g/dm³. Coke plant wastewater treatment after its initial purification was performed by means of ultrafiltration process with the use of a polysulfone membrane with 16% weight content of polysulfones in the film-forming solution and a 5-second time of solvent evaporation from the membrane surface. In spite of the fact that treated coke oven wastewater presented high values of pollution indicators, making it impossible to drain into the natural receiver or sewer, it was posttreated in the process of reverse osmosis using a nylon membrane (ADF), of an American company Osmonics. The article also presents an attempt of modelling of low-pressure membrane filtration efficiency based on the relaxation model assumptions.

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Key words: coke plant wastewater, volumetric coagulation, Fenton reagent, pressure membrane techniques, modeling efficiency of the process of ultrafiltration

1. INTRODUCTION

In the process of coal coking there are many noxious and environmentally damaging post-process waste streams. These include coke plant contaminated water and coal preparation water.

The amount of coke wastewater produced depends mainly on the size of the plant production and the moisture content in the coal feed. They are one of the most hazardous industrial waste. These include among others: PAHs, heterocyclic compounds, oils, tar substances and inorganic compounds i.e. cyanides, sulfides, sulfates, thiosulphate, ammonia and heavy metal ions[1-4].

Therefore, before discharging into the receiver, coking plant wastewater must undergo a process of purification which should guarantee the lowest cost but provide the highest degree of impurities removal. Increase in the sewage treatment efficiency is often associated with the expansion of new unit processes in the treatment systems, or the modification of existed systems. Among the physicochemical methods used for wastewater-bearing contaminants remaining in the form of colloids and fine suspensions very often is used the process of coagulation, while for the removal of phenols and other nonbiodegradable organic pollutants advanced oxidation process with Fenton's reagent is applied [4,5].

The authors attempted to assess the effectiveness of post-process waste water treatment in the integrated systems combining coagulation and advanced oxidation with the pressure membrane ultrafiltration and reverse osmosis. In addition, they attempted to predict polysulfone ultrafiltration membranes efficiency based on the relaxation model assumptions.

2. THE SUBSTRATE OF THE RESEARCH

The treated wastewater came from Coke Plant Coke "Koksownia Czestochowa Nowa" Ltd. It was the subject of mechanical treatment, so tar substances, oils and solids were removed. This process was conducted in decanters, from which tar was transported by pipeline to the underground tank and then trough the intermediate tank to the storage tanks. Post-gas water separated from the tar and oils was subjected to phenol removal and sent to the ammonia stripping columns. Table 1 shows the values of selected pollutants indicators characterizing coke oven effluent after pre-purification.

Determination	Unit	Value	The indexes of sewage pollution which is carried away to the receiver (Journal of law, 2006 no. 137, item. 984)
pH	-	9,34-9,05	6,5 - 9
COD	mg O_2/dm^3	3860-3138,6	125
TC	mg C/dm ³	898,3-770,0	-
TOC	mg C/dm ³	708,8-614,6	30
TN	mg N/dm ³	2186-1130	30
Phenol index	mg/dm ³	534-435	0,1
Ammonium nitrogen	$mg NH_4^+/dm^3$	392,0-386,4	10
Free cyanide	mg/dm ³	9,92-6,05	0,1
Sulfides	mg/dm ³	1,86-1,19	0,2
General Iron	mg/dm ³	7,5-2,2	10

Table 1. Coking industry wastewater from Coking Industry "Koksownia Częstochowa Nowa" ltd

3. APPARATUS

Coke oven wastewater physical-chemical treatment i.e. volume coagulation and the chemical process of advanced oxidation was conducted with the use of vascular reactor with a capacity of 3.0 dm^3 , whose contents were stirred with a magnetic stirrer [3].

In the process of high-pressure membrane filtration for coke- plant wastewater treatment an apparatus with a slab-type membrane module SEPA CF-NP from American company Osmonics., a sewage tank with a capacity of 8 dm^3 with a cooler, rotameter, high-pressure pump and pressure gauges and valves were used [1].

The module consisted of two steel plates between which a flat membrane was placed in a shape of a rectangular sheet with dimensions of $190 \times 140 \text{ mm}$ (total surface of the membrane was 155 cm^2 , and the filtration area 144 cm^2). The whole was introduced into a steel enclosure in order to provide the sealing arrangement.

4. THE METHODOLOGY OF RESEARCH AND ANALYTICAL DETERMINATIONS

Coke-plant wastewater was treated in three integrated systems, whose block diagrams are shown in Fig. 1.



Fig. 1. Scheme of treatment coke-making wastewater

The first system consisted of two combined pressure membrane processes such as ultrafiltration and reverse osmosis. In the second system a volume coagulation process was introduced. Its target was the initial wastewater pretreatment (the removal of colloidal substances causing ultrafiltration membrane fouling). Next, the wastewater was subjected to a post-treatment by means of ultrafiltration and reverse osmosis. In the first stage (I) of the last system the wastewater was purified in the process of advanced oxidation with Fenton's reagent assuring the mineralisation of toxic and non- biodegradable organic pollutants present in raw wastewater such as phenol. Then, it was successively cleaned in the process of low pressure and high pressure membrane filtration.

In the first system coke plant wastewater was initially pre-treated by means of ultrafiltration process, using prepared in laboratory a polysulfone membrane with 16% weight content of polysulfones in the film-forming solution and a 5-second time of the solvent evaporation from the membrane surface equal to 5 s. Initially, it was subjected to conditioning, which consisted of its filtering with de-ionized water of varying trans-membrane pressure in the range of 0.2 MPa - 0.8 MPa. Linear velocity over the surface of the membrane was 2.0 m/s. This process was performed until the stabilization of the water flow volume in the time, which indicated the formation of the permanent

membrane structure. In the next stage of experiment the effectiveness of wastewater treatment processes was assessed, consequently in the coke wastewater ultra-filtration process ($\Delta P=0.4MPa$, u=2m/s) and reverse osmosis ($\Delta P=2MPa$, u=2 m/s).

The coke-plant wastewater final treatment after the initial screening process was conducted on a commercial polyamide osmotic membrane ADF, manufactured by U.S. Company Osminics. It was determined the characteristics of de-ionized water transport for the membrane with the diaphragm (Δ =0.5-2.0 MPa, u = 2 m/s, then under the pressure of 2.0 MPa and linear velocity of 2 m/s the wastewaters were subjected to purification process. The effectiveness of the treatment was evaluated according to the relativity between the experimental and temporary permeate fluxes, and there was also determined the change in the pollution indicators for the raw and cleaned sewage. Next the chemical oxygen demand (COD), total organic carbon (TOC), total coal (TC) and the concentration of ammonia nitrogen and total, phenol index, free cyanides and sulphides were determined. COD factors were established through a test method on a spectrophotometer HACH DR 4000; TOC, TC and total nitrogen concentrations through a method of high temperature catalytic oxidation using a gas chromatograph Multi N/C 2100 and the concentration of free cyanide, phenol index and sulphide were determined using cuvette tests from HACH LANGE spectrophotometer DR 2800th.

In the second proposed system after the initial volume coagulation and sedimentation of coke plant wastewater it was purified in the process of ultrafiltration and reverse osmosis.

The earlier studies had shown that the most useful coagulant applied in coke plant wastewater treatment was the sulphate (VI) iron (III) under the trade name PIX-113, manufactured by Chemical Plant Kemipol [Bielsko-Biala 2011]. Therefore, in the process of coagulation this coagulant was used in order to obtain the initial adjustment pH = 9. The coagulant dose was 400 mg/dm³. The process of mixing the sewage with a coagulant was conducted in two stages. The quick stirring lasting 1 minute was to mix the entire contents of the reactor, while the slow stirring that ran for 30 minutes ensured the flocks formation, forming larger agglomerates subsequently. After 30 minutes of sedimentation the effluent was introduced into the ultrafiltration membrane module.

In the last test system after oxidation process with Fenton's reagent and sedimentation as well as in the second system it was purified with the use of pressure membrane techniques. Previous studies had shown that advanced oxidation process is best carried out for a dose of iron and hydrogen peroxide, respectively at 0.8 g/dm³ and 2.4 g/dm³, while the most favorable pH of the effluent in this process should amount 5. After 5 minutes of rapid stirring with an oxidizing mixture wastewater were subjected to four-hour oxidation process.

After this time the contents of the reactor was neutralized with 30 wt%. NaOH solution to pH 7, stirred 30 minutes and then subjected to a 30-minute sedimentation. The supernatant liquid was directed to the UF and the osmotic models.

5. RESULTS AND DISCUSSION

5.1. Effect of advanced oxidation and volume coagulation of the coke wastewater treatment efficiency

Figure 2 compares the changes in the quantity of permeate flux obtained in the process of coke plant wastewater ultrafiltration treatment (system I) and ultrafiltrate fluxes obtained in wastewater low-pressure filtration pre-treated with coagulation and advanced oxidation processes (system II and system III).



Fig. 2. The dependence of the experimental temporary permeate flux on the time of postprocess coke plant wastewater low-pressure membrane filtration

The highest efficiency was observed in the membrane system in which sewage was subjected to ultrafiltration process after the advanced oxidation with Fenton's reagent. Stabilized permeate flux equilibrium after 90 minutes of low-pressure membrane filtration was about 54.9% higher compared to the permeate flux obtained in the direct coke plant ultra-filtration process and shaped at the level of $0.394 \cdot 10^{-5} \, [\text{m}^3/\text{m}^2 \cdot \text{s}]$. These streams were respectively 1.33 and 1.19 times lower in comparison with a de-ionized water stream. In the case of the first system the permeate flux equilibrium stabilized after 150 min and amounted to $0.463 \cdot 10^{-5} \, [\text{m}^3/\text{m}^2 \cdot \text{s}]$. This shows that the post-process coke plant waste water pre-treatment resulted in the removal of pollutants that cause the

fouling phenomenon. Table 2 shows the characteristics of wastewater treated in the I system while table 3 displays the sewage treatment in the integrated systems combining physical-chemical and chemical process with the pressure membrane techniques.

Table 2. Effectiveness of coke wastewater integrated system ultrafiltration-reverse osmosis system

Indiastons of pollution	Raw	Treatment wastewater in UF		
indicators of pollution	wastewater	Values of pollutants	R, %	
pН	9,34	9,05	-	
$COD, mg/dm^3$	3860	2307,9	40,2	
TC, mg/dm^3	898,3	708,8	21,1	
TOC, mg/dm^3	770	560	27,3	
Phenol index, mg/dm ³	534	430,5	19,4	
TN, mg/dm^3	2189	569	74,0	
Ammonium nitrogen, mg/dm ³	386,4	302,4	21,7	
Free cyanide, mg/dm ³	9,925	2,42	75,6	
Sulfides, mg/dm ³	1,86	0,689	62,9	
General Iron, mg/dm ³	2,19	0,893	59,2	

Table 3. Effectiveness of coke wastewater treatment in integrated systems combining volume coagulation and advanced oxidation process with ultrafiltration (system II and system III).

	Dow]	reatment w	vastewater	
Indicators of	Kaw	Volumetric coagu	lation-UF	Advances oxidation-UF	
pollution	wastewat	Values of	D 04	Values of	D 0/4
	CI	pollutants	к, %	pollutants	к, %
pН	9,34	9,1	-	7,18	-
COD, mg/dm^3	3183,6	1652,3	48,1	1456	54,3
TC, mg/dm^3	898,3	673,3	25,0	387,6	56,9
TOC, mg/dm^3	708,8	551,9	22,1	356,4	49,7
Phenol index, mg/dm ³	435	349	19,8	156	64,1
TN, mg/dm^3	1330	1170	12,0	1130	15,3
Ammonium nitrogen, mg/dm ³	392	322	17,9	297,6	24,1
Free cyanide, mg/dm ³	6,05	5,6	7,4	5,6	7,4
Sulfides, mg/dm ³	1,195	0,175	85,3	0	100
General Iron, mg/dm ³	7,5	0,945	87,4	8,6	-

It is clear that the use of post-process coke plant wastewater pre-cleaning improved the effectiveness of its treatment in the ultra-filtration process. However, none of the systems tested did not provide a sufficiently high degree of pollutants removal from treated wastewater, which resulted in the lack of the possibility of the direct discharge to a receiver. This prevented also the use of treated water as a post-process technical water in the coke oven production.

5.2. The effectiveness of coke wastewater by reverse osmosis

Fig. 3 shows the dependency of permeate streams volume changes on the time of high-pressure membrane filtration post-treatment for all systems tested.





Tables 4a and 4b present the effectiveness of post-process coke plant wastewater treatment tested in the integrated systems.

	Treatment wastewater					
Indicators of	UF-RO		Volumetric coagulation-UF-RO			
pollution	Values of	D 0/	Values of	R, %		
	pollutants	K, %	pollutants			
рН	7,59	-	7,28	-		
COD, mg/dm^3	77,5	98,0	115	96,4		
TC, mg/dm^3	41,46	95,4	49,78	94,5		
TOC, mg/dm^3	27,38	96,4	27,41	96,1		
Phenol index, mg/dm ³	0	100	0	100		
TN, mg/dm^3	47,05	97,9	106,1	92,0		
Ammonium nitrogen, mg/dm ³	30,8	92,0	42,28	89,2		
Free cyanide, mg/dm ³	0	100	0	100		

Table 4a. Effectiveness of coke wastewater in the integrated systems.

Sulfides, mg/dm ³	0	100	0	100
General Iron, mg/dm ³	0,139	93,7	0,193	97,4

Table 4b.	Effectiveness	of coke	wastewater	treatment	in the	integrated	systems
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Indiastors of pollution	Advances oxidation -UF-RO			
indicators of pollution	Values of pollutants	R, %		
рН	8,05	-		
$COD, mg/dm^3$	52,6	98,3		
TC, mg/dm^3	34,58	96,2		
TOC, mg/dm^3	23,89	96,6		
Phenol index, mg/dm ³	0	100		
TN, mg/dm^3	47,05	96,5		
Ammonium nitrogen, mg/dm ³	21	94,6		
Free cyanide, mg/dm ³	0	100		
Sulfides, mg/dm ³	0	100		
General Iron, mg/dm ³	0,116	98,5		

The obtained results suggest that sewage treated in the process of reverse osmosis still did not meet quality standards set out in the Regulation of the Minister of Environment of 28 January 2009r., on conditions to be met by the introduction of sewage into the water or soil, and on substances particularly harmful to the aquatic environment due to the excessive concentration of ammonia nitrogen. All systems were tested several times exceeding the allowable concentrations of ammonia, volatile in terms of NH4 + ions. Therefore, post-process the water before discharge into the coke output of natural or drains should be further subjected to such a process of gas desorption. However, they may be recycled to the coke production cycle for quenching.

5.3. Modeling of low-pressure membrane filtration in coke wastewater treatment based on assumptions of the model relaxation.

This paper attempts to examine the possibility of forecasting the size of permeate fluxes in the coke wastewater ultrafiltration process in the integrated systems research.

Calculations are based on relaxation model assumptions, describing the changes in the permeate flux of membrane filtration process carried out in non-stationary arrangement [1,3].

The dependence of temporary permeate stream on the time of ultrafiltration process was experimentally determined and compared with the calculated theoretical temporary effluent streams. There were also compared the average experimental permeate streams and the theoretical values.

In the relaxation model the balance of mass transportation in the process of membrane filtration is presented by equation [1-3]:

$$\frac{d}{dt}\left(\mathbf{J}-\mathbf{J}_{\infty}\right) + \frac{t}{t_{0}}\left(\mathbf{J}-\mathbf{J}_{\infty}\right) = 0$$
(5.1)

at the assumptions that $J(t)_{t=0} = J_0$

It allows to specify the changes in the permeate flux during the filtration process, and the knowledge of the initial flow (J_0) , the equilibrium - saturation (J_{00}) and the time constant (t_0) allows to solve it. After integration of the equation (5.1) in the range of t and t0 = 0 we obtain the relationship:

$$\ln\left(\frac{J - J_{oo}}{J_0 - J_{oo}}\right) = -\frac{t}{t_0}$$
(5.2)

where: $J_{t=0} = J_0$,

 $J_{t\to oo} = J_{oo,}$ t₀ - time constant.

Time constant which characterizes the velocity of flux disappearing was determined from the equation (5.2) by means of graphic method:

$$t_0 = |1/a| \tag{5.3}$$

where: a - the straight line coefficient (y = and x t) characterizes the filtration process for the examined membrane..

The formula conversion (5.2) allows to determine the relation between the theoretical, temporary, volumetric stream of permeate (J_t) and the time of the filtration process:

$$J_{t}(t) = (J_{0} - J_{00}) \exp\left(\frac{t}{t_{0}}\right) + J_{00}$$
(5.4)

The theoretical average value of the permeate stream is determined by solving the equation (5.4):

$$\mathbf{J}_{e.} = \frac{1}{t_0} \int_{0}^{t_0} \mathbf{J}_{t}(t) dt = \mathbf{J}_0 - \frac{(J_0 - J_{00})}{e} = \mathbf{J}_0 - 0.37 (\mathbf{J}_0 - \mathbf{J}_{00})$$
(5.5)

within the integration limits: t = 0 i $t = t_0$.

Whereas experimental average value of stream was described by equation:

$$J_{a.e.} = \frac{1}{t_r} \int_{0}^{t_r} J_{e.}(t) dt$$
 (5.6)

where: t_r - time longer than: t_0 in which the volumetric permeate stream achieves the equilibrium value determined as: J_{oo} .

Table 3 shows the initial experimentally obtained permeate streams and saturation values. Fig.4 displays graphically determined time constants for ultra-filtration processes in integrated systems.

Table. 3. Values designated fluxes J_0 and J_{00} and the time constant t_0 in the treatment coke plant wastewater by ultrafiltration system

Type of system	$J_0 \cdot 10^5$, m ³ /m ² · s	$J_{00} \cdot 10^5$, m ³ /m ² · s	t ₀ , min.
UF	0,525	0,394	370,4
Volumetric	0,555	0,463	196,1
coagulation - UF			
Advances oxidation -	1,119	0,718	83,3
UF			





Fig 5. compares the size of temporary experimental permeate fluxes obtained in the process of the low-pressure membrane filtration in the cokeplant wastewater post-treatment after the pre-cleaning methods such as coagulation and advanced oxidation with the values of theoretical fluxes.



94 a)

b)

c)



Fig. 5. Comparison of temporary average permeate fluxes in the ultrafiltration process with temporary theoretical fluxes definite from relaxation model.

In all integrated systems tested there was observed a decline in the permeate streams in the course of low-pressure membrane filtration. In a unitary ultrafiltration treatment of post-process water the difference between the initial permeate stream volume and a stream of equilibrium was 23.5% while for the initial purification by means of volume coagulation and advanced oxidation was much lower and shaped at, respectively, 20.0% and 22.5%.

The comparison of experimental temporary ultrafiltration permeate streams with the temporary theoretical streams definite from relaxation model allows to conclude that relaxation model applied permits to predict the size of temporary permeate flux in the system that combine the pre-treatment methods, advanced oxidation and ultrafiltration (R^2 =0.974), and coagulation with ultrafiltration (R^2 =0.973) as well as for systems in which the membrane ultrafiltration processes were combined with reverse osmosis (R^2 =0.969).

Figure 6 shows the comparison between the average experimental permeate streams and the average theoretical streams obtained in the coke plant post- process wastewater treatment in the systems tested.



Fig. 6. Comparison of average experimental and theoretical obtained for coke wastewater treatment in the integreted systems

The highest average theoretical ultrafiltrate flux was obtained for the system with the initial advanced oxidation. It shaped at the level of $0.97 \cdot 10^{-5} \text{m}^3/\text{m}^2$ ·s and was 12.3% higher in comparison with the experimental stream. A similar relationship was observed for systems with pre- volume coagulation and the system where raw sewage ultrafiltration process was applied. The average theoretical permeate streams values were almost the same as the average values of experimental streams. For the system where pre-coagulation process was used the average experimental ultrafiltrate flux value was 0.526 \cdot 10^{-5} \text{m}^3/\text{m}^2·s, while the average theoretical flow value was 0.8% higher. In the raw sewage low-pressure filtration the difference between the theoretical and experimental flux values was 0.7%.

6. CONCLUSIONS

1. The use of coke plant waste water pre-pretreatment methods such as coagulation and advanced oxidation processes resulted in a temporary increase in the experimental permeate flux, respectively, of 82%, $(0,718\cdot10^{-5} \text{ [m}^3/\text{m}^2\cdot\text{s}])$ and 17.5% $(0.463\cdot10^{-5} \text{ [m}^3/\text{m}^2\cdot\text{s}])$ and higher treatment efficiency. Sewage treated in the most advantageous integrated system that combined advanced oxidation- ultrafiltration process and reverse osmosis had the lowest pollution indicators values, namely: COD-1456mg/dm³, TOC-356.4 mg/dm³, OW-387.6 mg/dm³, the concentration of cyanide-free-5.6mg/dm³, phenols-156mg/dm³, sulfides- 0 mg/dm³, N-NH₄⁺-297.6 mg/dm³.

2. The introduction of coke plant wastewater pre-treatment processes allowed to obtain higher permeate streams in the membrane cleaning process.

3. As in all integrated systems wastewater post-treated by means of reverse osmosis had a high concentration of ammonia nitrogen (UF-RO-42,28mgNH₄⁺/dm³, volume coagulation UF-RO-30,8 mgNH₄⁺/dm³, advanced oxidation-UF-RO-29,7mgNH₄⁺/dm³), before being discharged into the natural receiver should be subjected, for instance, to a gas desorption process. However, they may be directly used in the coke plant as technical water for quenching.

4. The quantities of theoretical temporary permeate streams determined by means of calculations based on relaxation model assumptions are similar to the experimental ones. Therefore, there is possibility to predict the changes in the permeate flux quantity in the post-process coke plant waste water ultrafiltration process considering the knowledge of the initial permeate streams ,saturation (equilibrium) and time constant.

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OCZYSZCZANIE POPROCESOWYCH WÓD KOKSOWNICZYCH W UKŁADACH ZINTEGROWANYCH ŁACZĄCYCH METODY KOAGULACJI OBJETOŚCIOWEJ I POGŁĘBIONEGO UTLENIANIA Z CIŚNIENIOWYMI TECHNIKAMI MEMBRANOWYMI

Streszczenie

efektywności oczyszczania ścieków koksowniczych artykule porównano W pochodzących z zakładu koksowniczego "Koksownia Częstochowa Nowa" w układach zintegrowanych, które łączyły ze sobą fizykochemiczną oraz chemiczną metodę oczyszczania z ultrafiltracją i odwróconą osmozą. Proces koagulacji objętościowej prowadzono siarczanem (VI) żelaza (III)-PIX-113, którego dawka wynosiła 400 mg/dm³ a pH środowiska wynosiło 9 natomiast pogłębionego utleniania z wykorzystaniem odczynnika Fentona, stosując stężenie żelaza równe 0,8 g/dm³ i nadtlenku wodoru 2,4 g/dm³. Oczyszczanie poprocesowych wód koksowniczych po ich wstępnym oczyszczeniu prowadzono stosując proces ultrafiltracji z wykorzystaniem płaskiej polisulfonowej membrany o 16% wag. zawartości polisulfony w roztworze błonotwórczym i 5-cio sekundowym czasem odparowaniem rozpuszczalnika z powierzchni membrany. Ponieważ tak oczyszczone ścieki koksownicze nadal charakteryzowały się wysokimi wartościami wskaźników zanieczyszczeń, co uniemożliwiło odprowadzenie ich do odbiornika naturalnego lub kanalizacji doczyszczono, je w procesie odwróconej osmozy z zastosowaniem poliamidowej membrany (ADF) amerykańskiej firmy Osmonics.

W pracy zaprezentowano również próbę modelowania wydajności procesu niskociśnieniowej filtracji membranowej w oparciu o założenia modelu relaksacyjnego.

Słowa kluczowe: ścieki koksownicze, koagulacja objętościowa, odczynnik Fentona, ciśnieniowe techniki membranowe, modelowanie wydajności procesu ultrafiltracji.