Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2023.29865

309 (2023) 49–57
October

Control of ionic composition of wastewater treated by reverse osmosis membranes to increase product total dissolved solids and reduce concentrate utilization costs

Alexei Pervov*, Konstantin Tikhonov

Department of Water Supply and Wastewater Treatment, Moscow State Research University of Civil Engineering, 26 Yaroslavskoye Highway, 129337 Moscow, Russia, email: ale-pervov@yandex.ru (A. Pervov)

Received 25 June 2023; Accepted 27 July 2023

ABSTRACT

The severe requirements for wastewater post treatment quality prompts us to look for new solutions to produce quality water with reduced biochemical elements concentrations. One of the most promising solutions is the use of reverse osmosis. Reverse osmosis membranes reject pollutions in ionic state and seem more efficient as does not depend on biological process. Reverse osmosis is already efficiently used for post treatment of wastewater after biological process to improve water quality. The significant disadvantage of reverse osmosis process is the concentrate handling. Concentrate stream should be disposed and often conditions should be provided. This article is a continued research program to utilize concentrate streams by reduction of concentrate volume by 100–300 times. This enables us to reduce radically the concentrate amounts to eighter withdraw these amounts by road or together with the sludge. The necessity to remove monovalent ions and reduce their concentration by 20–100 times (such as ammonia ion) requires to use membranes with high rejection, thus removal of small amounts of ammonia provides large amounts of concentrate with high total dissolved solids (TDS) values. In this article an attempt is undertaken to apply the newly developed technique to separate monovalent and divalent ions to increase the TDS value of permeate (product water) and increase the amount of salts discharged into water sources. The increase of permeate TDS provides the smaller TDS in concentrate that provides easier reduction of concentrate volume with lower costs.

Keywords: Reverse osmosis; Concentrate handling; Nanofiltration; Low rejection membranes; Reduction of concentrate flow; Rejection of biogenic elements

1. Introduction: main problems of the use of reverse osmosis to reach drinking quality

Reverse osmosis (RO) is being successfully applied to purify and reuse wastewater after biological treatment to achieve high quality to meet discharge regulations or technical requirements. A lot of information is provided about using RO for wastewater reuse [1–8]. In many cases it seems reasonable to apply reverse osmosis to reach required removal of monovalent pollutants (such as: ammonia) than to apply complicated improvements to biological process of biogenic elements removal [9–13]. Application of reverse osmosis process always meets a problem of concentrate disposal. In majority of applications concentrate is discharged. But to widely apply this technique for reconstruction and modernization of conventional wastewater treatment plants there is no sites to discharge concentrate. A zero-discharge techniques are being successfully developed but they have a limited application due to high energy requirements and operational costs [14–23]. As the improvement is applied to improve wastewater quality to discharge in the water body. In our articles authors

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* Corresponding author.

Presented at the European Desalination Society Conference on Desalination for the Environment: Clean Water and Energy, Limassol, Cyprus, 22–26 May 2023

demonstrated the developed techniques to reduce concentrate flow value by 100–200 times to facilitate it's withdrawal together with the sludge "by road" as it is usually applied when small and medium-size facilities are operated [22,24]. This process was experimentally developed and applied [19–24]. Meanwhile, it seems not reasonable (from membrane operation point of view) to use reverse osmosis membranes to remove ammonia (the ion that has lowest rejection rate by reverse osmosis membranes) and simultaneously produce deionized water [14,17,25–29] which requires high operational costs. Moreover, further concentrate flow reduction also requires high costs, as to reach high recovery a lot of membrane area is required [29].

Principles of concentrate reduction were discussed and described in previous publications [16–19]. In this article authors have undertaken a step to use nanofiltration membranes to reach the desired ammonia removal level and at the same time provide a certain increase of permeate total dissolved solids (TDS) value. Higher penetration of salts in permeate provides lower requirements to reach concentrate reduction [18,23–27]. And this also provides a certain reduction of costs required to achieve low concentrate volume. To achieve this effect, we have developed an ion separation technique whereby monovalent ions concentrations (such as ammonia concentrations are reduced in product water and are increased in concentrate). Principles of this approach are demonstrated in Table 1 and on the Fig. 1. Table 1 shows the composition of the biologically treated wastewater and permeate qualities after treatment of this water composition with different membranes (reverse osmosis and nanofiltration membranes with different rejection characteristics) [28,29].

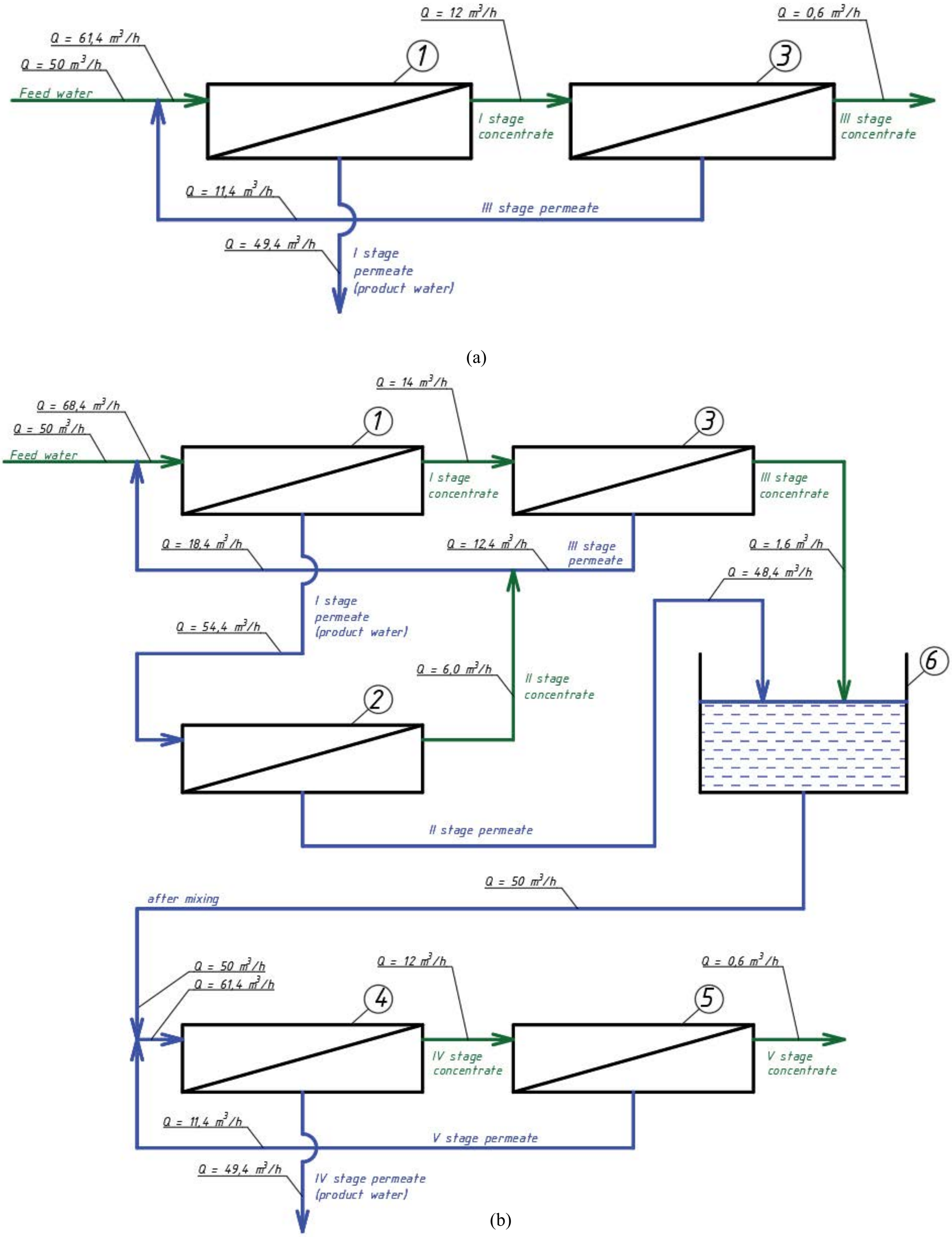
Conventional approach to treat wastewater consists of application of high rejection membrane to remove all salts and ammonia [1,14]. Above ammonia ion that has lowest rejection values [14], all other ions are removed (Table 1). The use of membranes with high rejection characteristics also requires recoveries do not exceed 0.7–0.8 values to escape low soluble salt scaling hazard. To reduce concentrate, an additional low rejection membrane stage is used [4,14,28]. Product water after this stage is forwarded to the reverse osmosis inlet. To reach high recoveries (to reduce concentrate by 100 and more times) a cascade of membrane stages is used. Permeate of each stage is forwarded to the entrance to the previous stage [17]. The flow diagram to describe flows and concentrations of pollutants is presented in Fig. 1a.

In the previous articles a method to evaluate the required membrane area to achieve high recovery was described [17]. Concentrate TDS can be recognized as a main factor that determines the costs of the concentrate reduction stage. In this article authors have undertaken an attempt to introduce a new technique to increase TDS of purified wastewater without the increase of ammonia concentration. This technique can reduce TDS of concentrate and help to reach the desired concentrate flow rate with less costs than in the conventional case. Fig. 1b describes the proposed technique. Production of quality purified water is implemented using the double stage scheme. For our case (Table 1) ammonia is reduced by 3 times on the first stage using 70 NE membranes and 80% recovery and by 3 times on the second stage using also 70 NE membranes and 80% recovery. The second stage permeate is forwarded to the entrance to the first stage membrane modules. The second stage permeate corresponds to the first stage permeate obtained in the scheme shown in Fig. 1a. The third stage modules aimed at reducing the first stage concentrate use also 70 NE membranes. For the first stage we implement achieving recovery value of 95%–96%, or reduction of concentrate volume only by 20–30 times. As it can be seen from Fig. 1b, concentrate collected after the third stage contains ammonia concentration increased by 7 times, TDS value increased by 10 times and calcium concentration increased by 20 times. After we mix the third stage concentrate and the second stage permeate, we obtain solution with the following concentrations: chemical oxygen demand (COD) – 31 mg/L; ammonia – 0.52 mg/L; TDS – 310 mg/L.

This water contains the same concentration of ammonia as the second stage permeate entering the second stage.

Table 1
Concentrations of main ingredients in wastewater, in permeate and in concentrate samples after treatment with different membranes under different conditions

No.	Ingredients	Feed water, mg/L	BLN membranes				Nanofiltration 70 NE membranes					
			RO permeate, mg/L % rejection		RO concentrate		Permeate			Concentrate		
			K = 2	*K* = 10	*K* = 2	*K* = 10	*K* = 10	*K* = 40	*K* = 100	*K* = 10	*K* = 40	*K* = 100
1	NH_4	1.27	0.15/80	0.5	2.6	10.1	0.9	1.7	3.5	8.0	18	57
2	PO_4^3	0.17	0.02/93	0.18	0.33	1.6	–	–	1.5	–	–	8.32
3	COD	190	32/82	48	220	384	100	300	400	280	490	600
4	BOD	7.24	0.7/90	2.16	7.7	13.8	–	–	4.61	–	–	124.5
5	SO_4	23	0.12/95	0.47	39	206	–	–	5.9	–	–	1218
6	CL	266	29/88	41.4	416	1,942	–	–	735	–	–	5,822
7	Oil	4.1	0.02/95	0.1	7.7	31.1	–	–	–	–	–	–
8	Detergents	1.8	0.05/98	0.25	2.5	10.2	–	–	–	–	–	–
9	TDS	465	50/95	250	725	3,580	400	700	1,215	3,500	11,000	17,160
10	PH	7.0	6.65	6.8	7.6	7.7	–	–	7.1	–	–	8.0



(a)

(b)

Fig. 1. Flow diagrams of treatment of biologically purified wastewater to remove ammonia: (a) Option 1: the use of reverse osmosis membranes to remove ammonia and further reduction of concentrate flow using low rejection nanofiltration membranes and (b) Option 2: application of double stage of nanofiltration membranes to reduce ammonia and the use of ion separation technique to increase product water TDS. 1 - the first stage membrane for product water; 2 - second stage membrane for product water quality improvement; 3- the third stage membrane for concentrate flow reduction; 4 - fourth stage for the blend treatment; 5 - fifth stage for concentrate flow reduction.

Thus, after we again treat the mixture with 70 NE membranes, we will get permeate with ammonia concentration 0.2 mg/L and TDS value by 10 times higher than obtained in the case when reverse osmosis membranes were used (Fig. 1a).The increase of TDS of the purified water can facilitate further concentrate treatment and concentrating as membrane costs to reach high recovery significantly depend on the concentrate salinity [30].

In this article authors aimed at investigation of the concentrate flow reduction process and finding the way to reduce total concentrate disposal costs. In previous publications [14,28] the scheme shown in Fig. 1a was investigated and main technical parameters of membrane plant were calculated.

2. Experimental program

To investigate post-treatment of biologically treated wastewater using reverse osmosis, an experimental program was developed. Water sample (150 L) of water after wastewater treatment plant was provided by Vodokanal of Narofominsk City in Moscow region. The flow diagram of membrane testing system is presented in Fig. 2.

A flow diagram of membrane test unit:

1 - feed water tank; 2 - pump; 3 - membrane module in pressure vessel; 4 - product water tank; 5 - heat exchanger; 6 - manometer; 7–9 - flow meters; 10 - bypass valve; 11 - flow and pressure regulation valve; 12 - pressure regulation valve; 13 - valve for adjusting the flow of cooling water; 14,15 - samplers.

Feed water was added in the feed water tank 1. The pump 2 delivered feed water to the membrane module 3. Permeate after membrane module 3 was collected in product water tank 4, and concentrate was returned back to the feed water tank. Samples of permeate and concentrate were taken simultaneously and product flow rate as well as total product water volume were fixed throughout experiment. Total volume reduction coefficient and concentration values of different pollutants were calculated depending on their values in the samples and volumes of product water and concentrate.

The experimental program was aimed at evaluation of technical parameters of the scheme shown in Fig. 1b and included six steps:

- Step 1: using 70 NE membranes on the first stage and obtaining dependencies of pollutant concentration values on *K* value when *K* is varied from 1 to 5 (up to 80% recovery);
- Step 2: continue to reduce concentrate volume using 70 NE membrane to reach recovery value of 97%;
- Step 3: treatment of the first stage permeate using 70 NE membranes to achieve desired permeate quality and 80% recovery;
- Step 4: mixing of concentrate and second stage permeate, evaluation of chemical composition of the blend;
- Step 5: treatment of the blend on the fourth stage using 70 NE nanofiltration membrane to reach 80% recovery;
- Step 6: continue to increase recovery to reach *K* value of 100.

Membrane elements of 1812 standard size were used in experiments. Membrane spiral wound elements were supplied by Toray Advanced Material, Korea Inc. (the Manufacturer of CSM membrane technologies). Spiral wound elements were furnished with reverse osmosis membranes (BLN-model, 95%–96% salt rejection) and nanofiltration membranes (70 NE model, 70% salt rejection). The total area of membrane surface in 1812 element was equal to 0.5 m^2.

During the experimental test run permeate was accumulated in tank 4 (Fig. 2) while the volume of the feed water in tank 1 constantly decreased. Thus, concentration values of all contaminants rejected by membranes and returned to

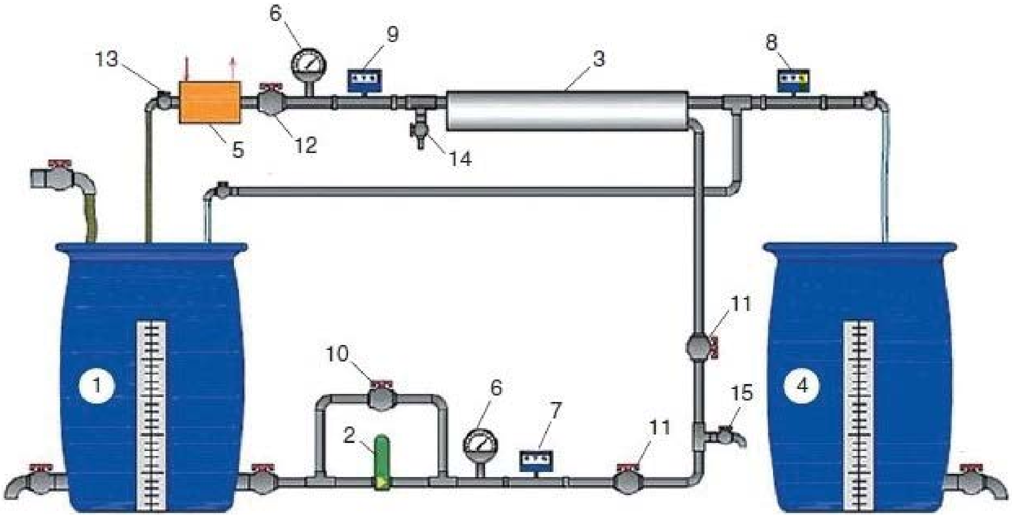


Fig. 2. Laboratory test unit flow diagram: 1 - source water tank; 2 - pump; 3 - membrane element in the pressure vessel; 4 - filtrate tank; 5 - heat exchanger; 6 - manometer; 7–9 - flow meters; 10 - bypass valve; 11 - valve for adjusting the flow of source water; 12 - valve for adjusting the working pressure and concentrate flow; 13 - valve for adjusting the flow of cooling water; 14, 15 - samplers.

the tank 1 (in ion and organic form) increased and the product water flow of membrane module constantly decreased with the growth of concentrate TDS. During the test program conductance, the dependencies of concentrations of the different water species on the value of initial volume reduction coefficient *K* were determined. The *K* value is equal to the ratio of the permeate flow rate to the feed water flow rate Q_p/Q_f. This value corresponds to the recovery value as follows: $Q_p/Q_f = 1–1/K$.

COD values were determined using titrimetric method. Concentrations of dissolved organic species were determined using the method of spectrophotometry. Atomic adsorption spectrophotometer of "A2" model was supplied by "Carl Stuart Group" (Lenster, Ireland, UK). Ammonia concentrations were determined using a photoelectric photometer of the "KFK-3-01-Z" model (supplied by "ZOMZ" Company, Sergiev Posad, Russian Federation). Electric conductivity, TDS and temperature values were evaluated using conductivity meter "Cond.730" supplied by WNW "Inolab-Akvilon" (Moscow, Prussian Federation). Calcium concentrations were evaluated trilometrically.

The volume of the sample was 100 L. At the first step we reduced the initial volume of the sample by 5 times using 70 NE membranes and product water amount of 80 L were produced. The last 20 L were treated further on the second step when the concentrate volume was reduced from 20 to 3 L thus providing reduction of initial volume by 30 times. At the third step product water of the first step was concentrated by 5 times and the product volume (64 L) was mixed with the second step concentrate (3 L). And then, on the fourth step this amount of the blended water was treated with NF-70 membranes to produce permeate (51 L) at *K* = 5 and further reduced by 100 times to achieve the volume of 6 L.

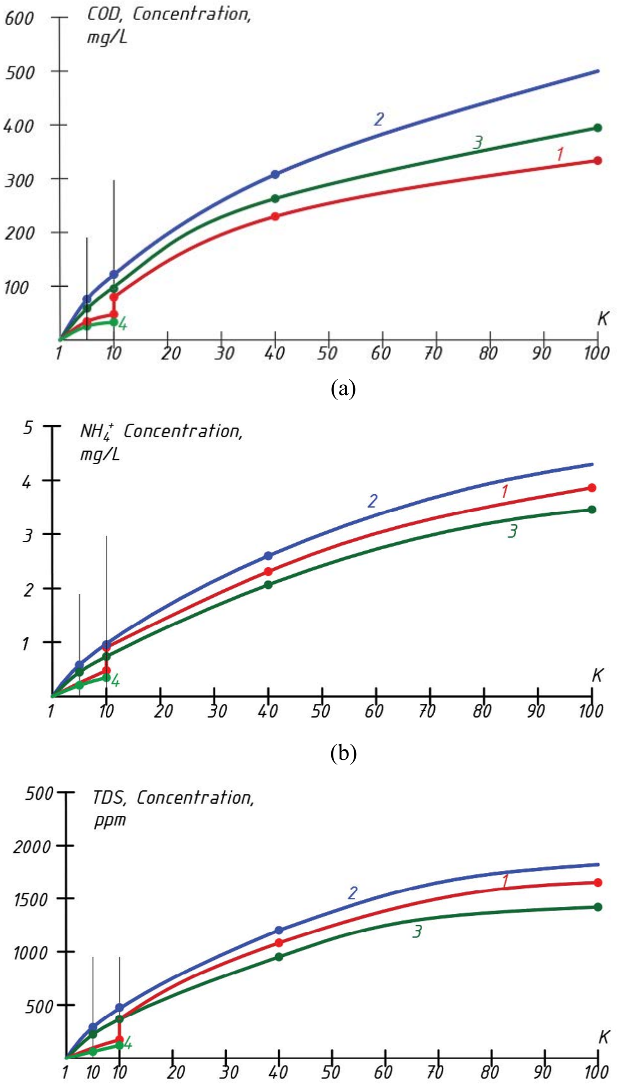
Fig. 3 shows results of experiments conducted on Steps 1 and 2. Concentrations of calcium (1), ammonia (2) and TDS (3) in permeate and concentrate are presented as dependencies on *K* value. Results of the first stage product water treatment with 70 NE membrane on the second stage (step 3) is shown in Fig. 4. Results of mixing of concentrate and second stage permeate are shown in Fig. 5 (as concentrations of feed water at *K* = 1). Fig. 5 demonstrates results of treatment of blend with nanofiltration membrane 70 NE to produce permeate at recovery of 80%.

Fig. 6 shows results of further concentrate reduction by 100 times to reach the recovery value of 99%.

Reduction of membrane permeability during experiments conductance is presented in Fig. 7.

3. Discussion of results

Also, during experiments conductance scaling and fouling rates were determined. To evaluate fouling rates, a method of mass balance calculation was applied. The experimental methods were fully described in Pervov et al. [14,28]. Figs. 7 and 8 demonstrate results of determination of calcium carbonate scaling rates and organic fouling rates in membrane modules throughout test runs conductance [28,30]. To predict decrease of membrane flux due to fouling and to prescribe cleaning schedules, dependencies of membrane flux on operational time and membrane flux on amount of accumulated foulant were developed [17] as well as efficiencies of removal of accumulated deposits by cleaning solutions were investigated [28–30]. To evaluate cost characteristics of described technological schemes, membrane areas were calculated. For the scheme shown in Fig. 1a, calculation of the specific membrane were performed in Pervov et al. [17]. Main steps of calculations are presented in Fig. 6. Principles of calculations consist of integration the function of product flow rate on *K* by the *K* ranges (Fig. 6b). Results of membrane area evaluation for the first, second, third and fourth stages of membrane technological scheme (Fig. 1b) are presented in Fig. 6c.

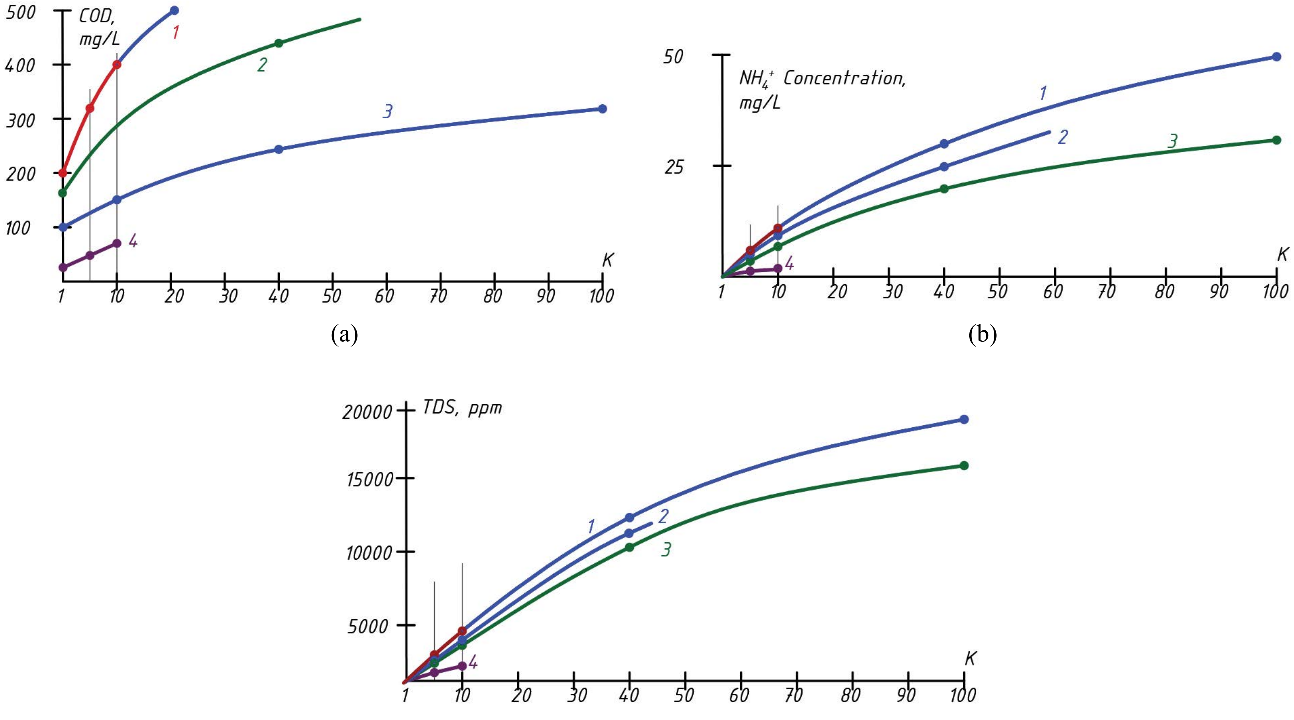


(a)

(b)

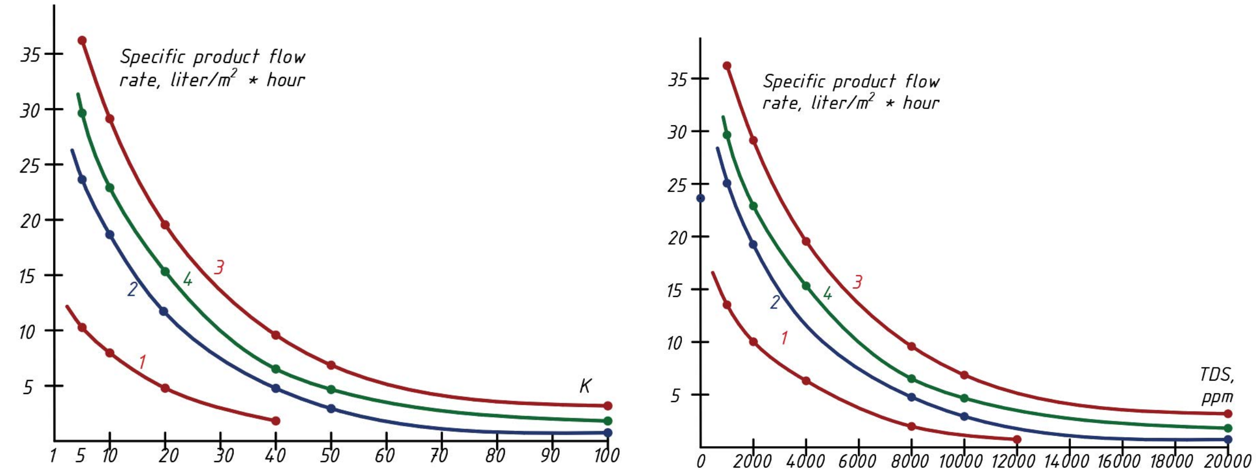
(c)

Fig. 3. Dependencies of calcium (a), ammonia (b) concentration and TDS (c) values in permeate on the value of initial volume reduction coefficient *K* values. 1 - treatment of wastewater with BLN membranes (Option 1, Fig. 1a), stages 1 and 3; 2 - treatment of wastewater, stages 1 and 3 (Option 2, Fig. 1b); 3 - stages 4 and 5 (option 2, Fig. 1b); 4 - stage 2, (Option 2, Fig. 1b).



(a) (b) (c)

Fig. 4. Dependencies of calcium (a), ammonia (b), concentration and TDS (c) values in concentrate at all stages of membrane treatment on the value of initial volume reduction coefficient K values: 1 - stages 1 and 3, Option 1 (Fig. 1a); 2 - stages 1 and 3 (Option 2, Fig. 1b); 3 - stages 4 and 5, Option 2 (Fig. 1b).

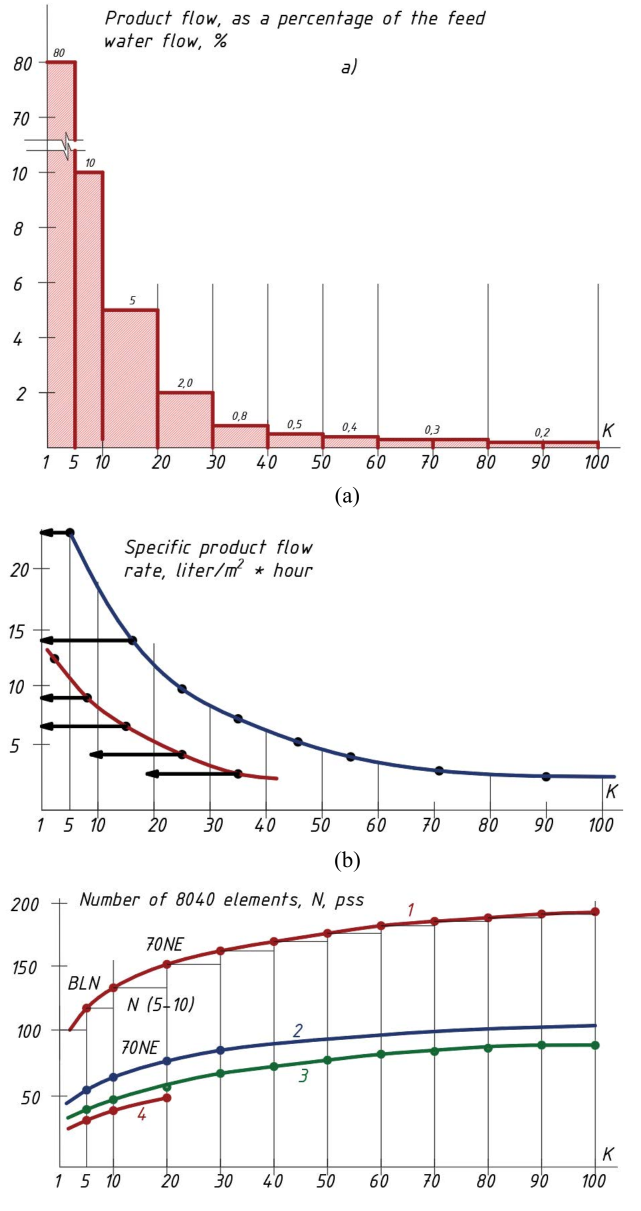


(a) (b)

Fig. 5. Dependencies of membrane specific product flow rate on K (a) and on TDS (b): 1 - Option 2, stages 1; 2 - Option 2, stages 1 and 3; 3 - Option 2, stage 2; 4 - Option 2, stages 4 and 5.

The last step was evaluation and comparison of technical parameters of two discussed technological schemes. Table 2 shows results of evaluation of membrane areas and number of membrane elements (to calculate membrane replacement costs and cleaning reagent consumption), energy costs and others.

Fig. 3 shows dependencies of calcium, ammonia concentration and TDS values on K in permeate and concentrate of NF-70 membrane on the first stage. Also, TOC values were determined to further evaluate organic adsorption rates on membrane surface. As it could be seen, rejection of organic pollutants is higher than rejection of monovalent ions (such as ammonia). At the second stage ammonia concentration is also reduced to reach the designed value. Dependencies of calcium, ammonia and TDS values on K are presented in Fig. 4. Results of TOC, ammonia and TDS in the third stage



(a)

(b)

(c)

Fig. 6. Principles and results of evaluation of total membrane area required to reduce feed water flow by 100 times: (a) amounts of product flow in the different ranges of *K* changes, (b) reduction of specific product flow with *K* and (c) dependencies of calculated number of 8040 elements on *K*; 1 - Option 1, stages 1 and 3; 2 - Option 2, stages 1 and 3; 3 - Option 2, stages 4 and 5; 4 - Option 2, stage 2.

concentrate, second stage permeate, in the mixture of the third stage concentrate and second stage permeate, as well as in the fourth stage permeate (product water), produced in Option 2 (Fig. 1b) are presented in Table 3.

Results of treatment of the blend solution after mixing of the first stage concentrate and the second stage permeate are shown in Fig. 5. As it can be seen, TDS and calcium concentration values after treatment of the blend solution with 70 NE membranes reach 0.5 and 100 mg/L, respectively

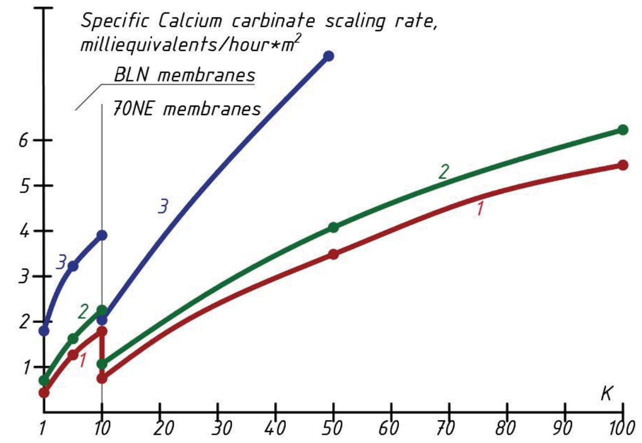


Fig. 7. Dependencies of calcium carbonate scaling rates on *K* at different stages of water treatment scheme (Fig. 1): 1 - Option 2 (Fig. 1b), stages 4 and 5, 70 NE membranes; 2 - Option 2 (Fig. 1b) stages 1 and 3, 70 NE membranes; 3 - Option 1 (Fig. 1a), stages 1 and 3, BLN membranes.

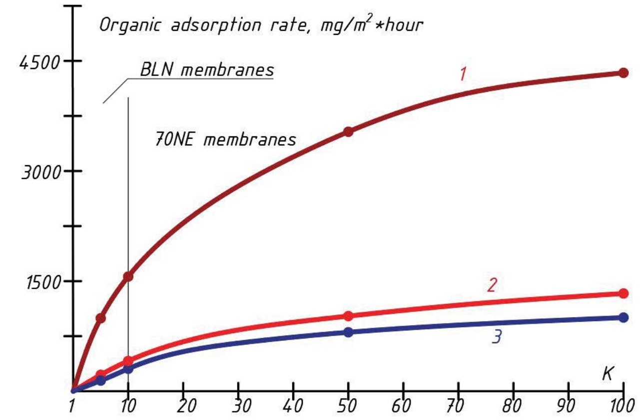


Fig. 8. Dependencies of organic fouling rates on *K* at different stages of water treatment scheme (Fig. 1): 1 - Option 1 (Fig. 1a), stages 1 and 3; 2 - Option 2 (Fig. 1b), stages 4 and 5; 3 - Option 2 (Fig. 1 b), stages 1 and 3.

(Fig. 5b). After concentration of ammonia reaches the regulation value of 0.2, permeate at the corresponding recovery value is collected for further reuse or discharge in natural water bodies. After the designed permeate quality is reached, concentrate is subjected to further treatment to reduce its volume by 100 times. It can also be seen in Fig. 5b that the concentrate TDS value that corresponds to *K* = 100 is substantially lower than concentrate value achieved using the technological scheme shown in Fig. 1a, and required membrane area is substantially lower. Fig. 6 shows comparison of techniques shown in Fig. 1a and b: TDS values in the first stage concentrate (a) and the fourth stage concentrate; membrane flux (b), required membrane area (c). As it can be seen in Table 2, total membrane area, number of elements and therefore membrane replacement costs are similar for both options. But the required amount of cleaning solution, antiscalant and total reagent consumption are substantially lower for the Option 2 (Fig. 1b).

Table 2
Evaluation of membrane area and required number of 8040 standard membrane elements to achieve the designed recovery

No.	*K*	Q_{perm}, % of Q_{feed}	First stage, scheme 1 (Fig. 1a) (Q_{feed} = 61.4 m^3/h)				Scheme 2 (Fig. 1b) stages 1, 3 (Q_{feed} = 68.4 m^3/h)				Scheme 2 (Fig. 1b) stages 4, 5 (Q_{feed} = 54.4 m^3/h)				Scheme 2, Fig. 1b stage 2 (Q_{feed} = 61.4 m^3/h)			
			Q_{perm}	SF	*F*	*N*	Q_{perm}	SF	*F*	*N*	*Q*	SF	*F*	*N*	*Q*	SF	*F*	*N*
		%	m^3/h	L/m^2·h	m^2	pcs	m^3/h	L/m^2·h	c	pcs	m^3/h	L/m^2·h	m^2	pcs	m^3/h	L/m^2·h	m^2	pcs
1	1–5	80	49	12	4,500	113	53.4	25	2,000	50	49	30	1,630	40	43	35	1240	31
2	5–10	10	6	9	700	18	6.3	20	310	8	6	32	130	5	5.4	32	170	5
3	10–20	5	3	14	180	5	3.1	15	160	4	3	22	140	3	–	–	–	–
4	20–30	8	1.2	8	150	4	1.3	9	148	4	1.2	15	80	2	–	–	–	–
5	30–40	0.8	0.40	6	95	3	0.49	–	–	–	0.48	10	48	1	–	–	–	–
6	40–50	0.5	0.30	4	80	2	0.32	–	–	–	0.30	8	36	1	–	–	–	–
7	50–60	0.4	0.2	2.5	75	2	0.22	–	–	–	0.2	6	33	1	–	–	–	–
8	60–80	0.3	0.15	1.5	60	2	0.16	–	–	–	0.15	5	30	1	–	–	–	–
9	80–100	0.2	0.12	1	40	1	0.13	–	–	–	0.12	4	30	1	–	–	–	–
Total		100	61.4			150	68.4			66	61.1			55	–	–	–	36

Q_{perm} – permeate flow rate, m^3/h;
SF – specific membrane flow, L/m^2·h;
F – membrane area, m^2;
N – number of membrane 8040modules, pieces.

Table 3
Results of COD, ammonia and TDS evaluation in permeate and concentrate at different stages of the technological scheme (Fig. 1b)

No.	Ingredients	Feed	Scheme 1		Scheme 2 (Fig. 1b), first stage, 70 NE membranes		Scheme 2 (Fig. 1b), third stage, 70 NE membranes		Scheme 2 (Fig. 1b), fourth stage
			RO × 5 Concentrate/Perm	70 NE × 30	Concentrate/Perm × 5	Concentrate/Perm × 30	Concentrate × 5	Mixture	Permeate × 5
1	COD	190/32	310/32	420/400	300/1,600	420/9,000	1,600	31	9.5
2	NH_4	1.27/0.5	5.8/0.2	25/5.1	8/0.5	12/3.5	0.2	0.5	0.2
3	TDS	465/50	2,000/50	7,000/1,500	3,500/160	7,000/1200	800/80	310	120

Table 4
Results of calculation of main technical and economical parameters of wastewater treatment plant, 50 m^3/h capacity

No.	Parameters	Scheme 1 (Fig. 1a)	Scheme 2 (Fig. 1b)
1	Number of membrane elements (8040 type), pcs	Stage 1: 151 Total: 150	Stage 1: 66 Stage 2: 36 Stage 3: 55 Total: 157
2	Annual antiscalant, kg/y	2,100	420
3	Annual cleaning agents consumption, kg	3,600	396 + 110 = 506
4	Total power of the pumps, kW	60	40 + 40 + 35 = 115
5	Total energy annual consumption kWh/y	420,000	805,000

4. Conclusions

- To post-treat wastewater after biological treatment RO techniques are efficiently applied. The need to reach regulation standards and low concentration of ammonia prompts to use reverse osmosis membranes with high rejection characteristics. This requires high costs and produces low TDS product water.
- To utilize concentrate, a concentration technique is developed to reduce concentrate volume by 100–200 times. The procedure includes using a cascade of low rejection nanofiltration membranes. A special method is developed to calculate the required membrane area.
- Low TDS of the product water complicates reduction of concentrate volume. A new technique is developed to increase of TDS and multivalent ions concentrations in

permeate. This provides higher TDS values in permeate and simplifies reduction of concentrate volume as well reduces membrane area and operational costs.

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