A new technique to purify biologically treated wastewater by reverse osmosis: utilization of concentrate

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ABSTRACT
Introduction. Possibilities to purify municipal wastewater using reverse osmosis membrane techniques are investigated aimed at production of quality water for industrial use or meeting regulations for surface water sources discharge. A new developed tools to utilize concentrate effluents by reducing its flow by a value that does not exceed 0.5–1.0 per cent of initial feed water flow and it’s withdrawal of all rejected impurities together with dewatered sludge as a sludge moisture. Objectives: development of reverse osmosis techniques to purify wastewater after biological treatment; evaluation of possibilities to radically reduce concentrate flow to withdraw it together with activated sludge as it’s moisture.

Materials and methods. Experimental research is conducted to develop membrane operational modes during wastewater treatment. Experimental procedure is developed and described to evaluate reduction of membrane rejection of dissolved impurities and product flow decrease during experimental wastewater treatment and concentrate utilization test run.

Results. The basic equations are derived that enable us to determine: the required concentrate flow value that corresponds to concentration values of COD and suspended solids values in the feed water; the required values of membrane recoveries that correspond to ammonia concentration in the feed water to meet required regulation values in the product water. The tools to evaluate membrane area and a number of membrane modules are developed and described. Optimum values of the working pressure are evaluated as well as other economic parameters are presented to compare the developed techniques with biological treatment.

Conclusions. To reach the required ammonia concentration in product water, double stage treatment of feed water with low-pressure reverse osmosis membranes is required. Influence of dissolved organics defined as COD, on membrane performance. The optimum value of working pressure is determined which is 7.5–8 Bars.

KEYWORDS: reverse osmosis, nanofiltration, recovery, wastewater treatment, biogenic elements removal, ammonia, wastewater sludge dewatering, sludge moisture

FOR CITATION: Pervov A.G., Tikhonov K.V. A new technique to purify biologically treated wastewater by reverse osmosis: utilization of concentrate. Vestnik MGSU [Monthly Journal on Construction and Architecture]. 2020; 15(5): 688-700. DOI: 10.22227/1997-0935.2020.5.688-700 (rus.).

Новая технология обработки сточных вод, прошедших биологическую очистку, методом обратного осмоса: утилизация концентрата

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АННОТАЦИЯ
Введение. Исследованы возможности обработки биологически очищенных сточных вод методом обратного осмоса с целью получения высококачественной воды, пригодной для технологических нужд различных производств или для сброса в водоемы рыбохозяйственного назначения. Представлено обоснование возможности утилизации концентрат- та установки обратного осмоса путем радикального сокращения его расхода до величины, не превышающей 0.5–1 % расхода воды, поступающей на очистку, и выведения его вместе с обезвоженным осадком избыточного активного ила в виде его влажности. Цель — разработка новой технологии обратного осмоса для обработки сточных вод, прошедших биологическую очистку; обоснование возможности радикального снижения расхода концентрата для его удаления вместе с обезвоженным осадком.

Материалы и методы. Проведены экспериментальные исследования режимов работы мембранных установок при очистке сточных вод. Описана экспериментальная методика, позволяющая определить изменения концентраций различных растворенных загрязнений в фильтрате мембранных аппаратов и падение производительности мембран в процессе обработки сточных вод и снижения расхода концентрата.
INTRODUCTION

Reverse osmosis is recognized as a very efficient tool to reject different impurities [1–3]. However, the wide application of this technique is reduced by disadvantage of membrane treatment technology — the existence of concentrate streams that are discharged in water sources [1–6]. At the world, biggest wastewater treatment reverse osmosis facilities concentrate is discharged in the sea [1]. But such an approach cannot be widely applied. Concentrates of reverse osmosis constitute about 15–20 per cent of the feed water that enters membrane treatment facilities [1, 11]. To reduce concentrate flows, different tools are developed [8–11].

In earlier publications [10, 11] the authors proposed a new technique to reduce and “utilise” concentrate flow of reverse osmosis (RO) facilities used to treat wastewater after biological treatment. According to the developed technique [10] reverse osmosis concentrate flow is reduced to a value not exceeding 0.5–1.0 per cent of feed water that enters RO facility. This amount is withdrawn from the water treatment facility together with the dewatered sludge as the sludge moisture.

Reverse osmosis design experience revealed main problem of membrane selection — reduction of ammonia concentration. Reverse osmosis membranes reduce ammonia concentration by 15–20 times. For the case, when ammonia concentration in wastewater is 50–60 ppm double stage reverse, osmosis process is required. Membrane flux decreases with recovery and feed water TDS growth. Therefore, concentrate flow can be reduced through the use of nanofiltration membranes. After wastewater is treated by reverse osmosis membrane, reverse osmosis concentrate is further treated with nanofiltration membranes at the additional stage.

Present article describes results of research to develop guidelines to utilize reverse osmosis concentrate flow, such as:

• concentrate flow and recovery values are determined to meet requirements to reach concentrate volume that equals the sludge moisture;

• relationships are elaborated to predict product water composition and determine membrane unit characteristics (pressure, recovery, membrane rejection etc.);

• a new technique is proposed, which allows to evaluate the membrane area and membrane modules amount to reach the required product water quality and product flow.

OBJECTIVES

According to developed new technique concentrate flow is reduced and withdrawn together with dewatered activated sludge [12–18]. Application of reverse osmosis can provide efficient solution to remove biogenic elements from wastewater that seems very competitive to membrane bioreactors and other membrane processes used for wastewater treatment and reuse [12, 13, 19–21]. The required concentrate flow and its chemical composition can be obtained from balance equations that are based on assumption that the amount of dissolved salts (impurities) that are discharged from membrane system together with the sludge and desalinated water are equal to amount of impurities in the feed wastewater that enters membrane system. Therefore, the amount of salts that enters the sedimentation tank with concentrate flow and the sludge flow is equal to amount of salts that are contained in dewatered sludge that is removed from the system.

The research was mainly aimed on development of tools to design RO unit that treats wastewater and provides recovery up to 99.5%.

• to determine optimum values of recovery of the first stage RO unit;

• to determine required value of recovery of the second stage unit;

• to determine characteristics of product and concentrate on the second stage of concentrate treatment with nanofiltration membranes;

• to determine the required amount of membrane modules on the first and on the second stage of wastewater concentrate treatment to reach recovery up to 99%.
MATERIALS AND METHODS

To evaluate membrane product flow and rejection characteristics during water treatment, experiments were conducted using wastewater after biological treatment. 70 liters of wastewater were sampled from the secondary sedimentation tank at the wastewater facilities in Narofominsk (Moscow vicinity).

The test procedure to reduce concentrate flow and investigate its influence on membrane performance was described in [9–11]. The test unit flow diagram is shown on Fig. 1. Wastewater after bioreactor was collected in the feed water tank 1. The pump 2 to the module 3 pumped wastewater. Concentrate was returned back to the tank 1 and product flow was forwarded to product tank 4. Pressure value was regulated using the pressure valve 5 and controlled by pressure gauge 6. Reverse osmosis membrane used were 1812 type elements manufactured by CSM Company (model 1812 BLN). For concentrate treatment, 90NE membranes were used (model 1812 90NE). The gear pump created pressure. Pressure value was 6 Bars.

Wastewater after biological treatment was placed in tank 1. From tank 1 feed water was fed by pump 2 to membrane module 3 where feed water was separated into product and concentrate (retentate) flows. Product flow is forwarded to product tank 4 and retentate is returned to tank 1. During test unit operation retentate volume in tank 1 constantly decreased while product volume in tank 4 constantly grew. The ratio of initial volume in tank 1 in the beginning of experiment to the volume in tank 1 at the certain moment is defined as Concentration coefficient $K$. Usually in water desalination practice $K$ value does not exceed 4–6, as dissolved salt concentrations in retentate grow and sparingly soluble salts scaling as well as organic coagulation and fouling can occur. Therefore on the first stage of experiment $K$ value was 10 that corresponds to reduction of feed water volume in tank 1 from 70 to 10 liters. Membrane element of 1812 type tailored with low pressure reverse osmosis BLN membranes (CSM production) that reject ammonia ions by 90–95 per cent was used. The product water volume obtained during the first stage of experiment was 63 liters. On the second stage product tank 4 was used as a feed water tank 1 and collected product water was secondly treated by RO membrane to reduce ammonia concentration to meet regulation requirements for water discharge (0.4–0.5 ppm of ammonia).

By the end of the second test run feed water volume in the tank 1 was 6 liters that corresponded to $K$ value of 10. At the third experimental stage concentrate collected in tank 1 during the first test run (7 liters) was secondly treated by mebrane module tailored with 90NE nanofiltration membrane to reach 0.5–0.7 liters volume. The selection of nanofiltration membrane with larger pore size enables us to treat and separate solutions with high TDS values. Nanofiltration membrane product water was collected in separate product tank 4. During all three test runs samples were regularly withdrawn from tanks 1 and 4, when $K$ values equaled 1, 2, 5 and 10.

![Fig. 1. The test membrane unit flow diagram: 1 — feed water tank; 2 — pump; 3 — spiral wound membrane module; 4 — permeate tank; 5 — heat exchanger; 6 — pressure gauge; 7 — feed water flow meter; 8 — permeate flow meter; 9 — concentrate flow meter; 10 — by-pass adjusting valve; 11 — feed water adjusting valve; 12 — concentrate adjusting valve; 13 — cooling water adjusting valve; 14 — sampler; 15 — chemical cleaning valves](image-url)
RESULTS. EXPERIMENTAL DATA PROCESSING

1. Determination of membrane product water quality.

Experimental data was consistently processed with the aim to obtain values of the main technical parameters of membrane unit. First of all, dependencies of various ions concentration values on the recovery and the product volume were determined. Fig. 2 shows dependencies of ionic concentrations of sulphates, chlorides and ammonia ions on concentration factor $K$ values, where $K$ is a ratio of the initial volume in tank 1 (Fig. 1) to the volume of product water in product tank (4) during the test run.

Fig. 2. Increase of different species concentration values in product water with $K$ growth ($K = V_f/V_{conc}$)

Fig. 2 shows dependencies of various species concentration values in tank 1 (Fig. 1) versus $K$ values during the first and the third test runs. As it can be seen, the growth rate of salt concentration with $K$ increase in the first test run is obviously than in the third test run. This can be explained by higher rejection of reverse osmosis BLN membrane than of nanofiltration membrane used in the third test run. During the first test run about 5–10 per cent of salts contained in the feed water penetrate in product through membrane. And during the third test run 15–20 per cent of salts penetrate in product water due to low rejection of nanofiltration membranes. In this case, the experimental dependence is nonlinear: the concentration of salts in retentate does not grow in proportion to the decrease in retentate volume. Fig. 3 shows dependencies of ammonia concentration values in product tank on the volume of produced product. At the beginning of experiment, initial volume was 70 liters. This volume was treated on the first stage of experiment by low pressure reverse osmosis BLN type membranes until the amount of water in feed tank 1 (Fig. 1) was 7 liters and the volume of product water collected in tank 4 was 63 liters. On the second stage of experiment the remained amount of concentrate (7 liters) was treated using nanofiltration 90-NE membranes that demonstrated higher product flow. Evaluation of the areas of shaded figures shown on Fig. 3 enable us to calculate amounts of salts in product water on different stages of experiment at different $K$ values.

Then, decrease of membrane product flow throughout the experiment was evaluated as a dependence on $K$ value.

Fig. 3. Concentrations of ammonia in product water versus product water volume

Fig. 4 shows dependencies of ammonia and sulphate ionic concentrations as well as COD (chemical oxygen demand) values in concentrate flow during experimental test run. At different stages (that corresponded to certain $K$ values, such as: 2, 4, 6, 10, 20, 40, 80) product water samples were withdrawn where concentrations of all impurities as well as TDS values were determined. Similar dependencies of different species’ concentration values versus amount of product water collected are shown on Fig. 5 and 6. Concentrations of chloride and calcium ions are not shown as these values are much higher, than concentrations of ammonia and sulphates. BLN membranes efficiently rejected phosphate ions and their concentration values in product water were far lower than regulation values for surface water discharge.
As it is shown on Fig. 5 and 6, ammonia concentration values in product are higher, than discharge regulation values. This can be explained by the low rejection of monovalent ions by low-pressure RO membranes in the pressure range of 6–12 Bars. Ammonia ion rejection in this pressure range varies between 85 and 90 per cent. Therefore, to arrange high rejection of ammonia, the second stage RO BLN membrane treatment should be applied.

Fig. 6, shows relationships of ammonia and \( K \) concentration values in the first stage product water and in the second stage product water when the first stage product water, after the test run was finished, was collected and used as a feed water for the second stage. Concentration values of ammonia and sulphate ions are presented versus \( K \) values on Fig. 6.

Concentration values of TOC, ammonia and sulphates versus volume of product water are presented on Fig. 7.

Fig. 7 demonstrates reduction of specific product flow values of BLN membranes with the increase of product volume (from 0 to 63 liters) and of 90NE membranes with the increase of product volume from 63 to 69.3 liters. As it is shown, membrane product flow decreased by more than 10 times. Fig. 9 shows reduction of specific product flow versus \( K \) on both stages of concentrate treatment. Product flow was measured under pressure value of 6 Bars.

The next step was devoted to determination of technical parameters of membrane unit. Experimental data processing was aimed at determination of technical characteristics of membrane unit, such as membrane area required and minimal concentrate amount. To predict influence of membrane unit recovery on product water quality, various relationships were examined. Fig. 9 shows the growth of TDS in BLN and 90NE products. TDS values are obtained at pressure values of 6 and 12 Bars.
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Fig. 8. Reduction of specific membrane product flow rate, versus volume of product water

Fig. 9. Reduction of specific product water rate versus $K$ on the first stage (BLN membranes) and on the second stage (70 NE membranes) of membrane treatment

Fig. 10. The increase of TDS in product with $K$ growth

Fig. 11 shows decrease of BLN membrane product flow with time during experimental test run. To predict increase of concentration values of different dissolved species in product with recovery growth, the graph shown on Fig. 11 is conditionally devided into sections. Each section corresponds to different range of $K$ values: from 1 to 1.6; from 1.6 to 3; etc. Area of shaded figure demonstrates the amount of product water produced in each range of $K$ (when $K$ changes from 1 to 1.6 etc.). The amount of product water obtained in each range of $K$ variation is presented as a percentage of the total amount of product water produced by BLN during first stage of experiment when $K$ increased from 1 to 10.

The graph of 90NE product flow reduction with time during concentrate treatment with time, when $K$ value changed from 1 to 15 was similarly obtained and is shown on Fig. 12. For each range of $K$ amounts of product water are obtained as well as the TDS values. These amounts were obtained by multiplying the average concentration value by volume. The average concentration value was calculated as the arithmetic mean of the of TDS values in water samples withdrawn in the beginning and in the end of each range of $K$, for example: at $K = 1$ and $K = 1.6$, and at $K = 1.6$ and $K = 3$, etc. For obtained values of TDS and product volumes, the plots are built that demonstrate TDS values and different species concentration values in total product amounts that correspond to certain $K$ and product volume values (Fig. 13). To determine concentration values, product volumes produced in each range and total salts amounts contained in these volumes were summarized and then the total volume then divided total salt amount.
Fig. 12. Prognosis of total concentrations of impurities in product water of 90NE membranes on the second stage. Step 1: reduction of specific product flow with time

Fig. 13. Processing of experimental data. Prognosis of total concentrations of impurities in product water of BLN membranes. Step 2: concentration versus $K$

Fig. 14. Processing of experimental data. Prognosis of total concentrations of impurities in product water of 90NE membranes. Step 2: concentration versus $K$

of value 7 is explained by the low product water quality obtained with $K$ values from 7 to 10. Fig. 15, b presents results of determination of membrane product flow rates in different $K$ ranges. The calculations are made for the case, when the required product flow of membrane facility is 1000 liter per hour and concentrate flow is 140 liters per hour. For each range of change of $K$ value product flux was determined (Fig. 15, a) and the arithmetic average value of membrane specific flux was determined, expressed in: liters/hour x square meter (Fig. 11).

Using calculated values of product water produced during one hour in the selected range of $K$ and average specific value of membrane flux within this range, we can calculate the required membrane area to provide the required amount of product water during one hour. Fig. 15, c shows results of membrane area determination within the selected ranges of $K$ variation. Calculations are made for the case, when BLN membranes treat wastewater and $K$ value changes from 1 to 7, and the pressure was 6 Bars. The required number of membrane elements can be determined assuming that membrane area in a standard 4040 element (BLN 4040) manufactured by CSM Company (Korea) equals 10 square meters. Thus, to decrease wastewater flow by 7 times and to produce 1000 liters of quality product water per hour we need to use 12 elements of 4040 standard. The $K$ value grows “step by step”, therefore elements can be connected in series: 5 in parallel, then 4 and 3.

Fig. 16, a, b demonstrates similarly obtained results of a number of membrane elements calculations required to further reduce concentrate flow from 140 to 10 liters per hour on the second stage of concentrate treatment. For the second stage, 90NE nanofiltration membranes were used. As it is shown on Fig. 15, c, to reduce concentrate flow on the second stage, 5 membrane elements 90NE 4040 are needed connected in series: 3 in parallel and 2 in parallel.
Fig. 15. Processing of experimental data. Determination of the required BLN membrane area (product flow is 1000 liter per hour; concentrate flow is 140 liter per hour; pressure is 6 Bars): a — percentage of product flux for different ranges of $K$; b — the arithmetic average of specific membrane flow versus $K$; c — membrane area within the selected ranges of $K$ variation.

Fig. 16. Results of a required number of 90 NE membrane elements evaluation: a — percentage of product flux for different $K$ range; b — the arithmetic average of specific membrane flow versus $K$; c — membrane area within the selected ranges of $K$. 
It seems very important to investigate the influence of dissolved organics on membrane flux decrease [22–25]. COD values in wastewater tested in our experiments varied from 50 to 200 ppm. During wastewater treatment, when concentrate flow decreases by 70–100 times organic concentrations can reach high values that sufficiently increase osmotic pressure and reduce membrane flux. Therefore, to predict membrane behavior, we have to account for not only dissolved salts concentrations but dissolved organics (COD) as well. Fig. 16, a shows experimentally obtained dependencies of membrane BLN 1812 element flux on \( K \) during treatment of wastewater with different COD values. Fig. 17, b shows results of observed membrane flux reduction as dependencies on feed wastewater TDS (total dissolved salts) concentrations. It can be seen that membrane flux decreases not only due to increase of TDS, but also due to BOD growth.

Concentration values of different species in product water produced throughout the cycle were determined basing on results shown on Fig. 16 and 17. Product water quality prognosis is very important for membrane system design. One of the main parameters of membrane performance is Rejection \((R)\), which represents the amount of salts, penetrated through membrane and is defined as the ratio of the difference between salt concentration in the feed water (concentrate) \( C_f \) and in the product water \( C_p \) to concentration of salts in the feed water, expressed in percentage: 
\[
R = \frac{(C_f - C_p)}{C_f}, \%
\]
Membrane rejection is the main characteristic that demonstrates their efficiency. In our experiments membrane rejection (as compared to concentration of the feed water) constantly decreased, as salt concentration in circulating concentrate constantly increased with growth of \( K \) value. It is convenient to present rejection values as a function of Recovery, defined as the ratio of product flow rate \((Q_p)\) to the feed flow rate \((Q_f)\) expressed in percentage: 
\[
Rec = \frac{Q_p}{Q_f} \quad \text{(where } Q_f \text{ is the feed water flow and } Q_h \text{ is product water flow).}
\]
Fig. 18 (a) presents results of ammonia, sulphates, TDS and COD rejection determination during treatment of wastewater with BLN mem-
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Pressure value influences the value of low pressure membrane recovery: the higher pressure is, the higher is rejection. This is attributed to compaction and compression of membrane selective layer structure. Therefore rejection efficiency grows with the pressure increase. Fig. 20 shows BLN membrane rejection of ammonia ions versus recovery values at different pressure values. The derived formulas for calculation of recovery values also account for different pressure values.

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the value of the “current” working pressure (difference between working pressure and osmotic pressure).

Fig. 23 describes an example to calculate recovery values of the first and the second stages for different ammonia concentrations in the feed water using the derived formula. For the beginning, for maximum recovery value of 90% (Fig. 23, a) we can calculate required ammonia rejection on the second stage. Basing on the required ammonia concentration in the product water, and membrane rejection value, we can calculate ammonia concentration in the product water that enters the second stage. Further, knowing ammonia concentration in the first stage product water, and knowing ammonia concentration in the feed wastewater, we calculate the required recovery value on the first stage.

**CONCLUSIONS**

To reach the required ammonia concentration in product water, double stage treatment of feed with low pressure reverse osmosis membrane treatment is required.

Ammonia rejection efficiency depends on working pressure values. Higher-pressure values provide higher recovery values to provide required efficient ammonia rejection values.

Influence of dissolved organics defined as COD on membrane performance is investigated. Wastewater COD decreases membrane flux and should be accounted, when membrane area is determined.

Application of reverse osmosis and nanofiltration membranes enables us to produce quality water that meets discharge regulation standards and concentrate flow that does not exceed 0.3–0.5% of the total feed water that enters water treatment plant.

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Received December 2, 2019.
Adopted in a revised form on January 23, 2020.
Approved for publication April 28, 2020.

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