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## Track-etched membranes back-flushing and regeneration during the natural water purification

### Промывка и регенерация трековых мембран при очистке природной воды

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**Ключевые слова:** трековая мембрана; водоподготовка; местное водоснабжение; промывка; регенерация; фильтрация; производительность; гражданское строительство; здания и сооружения

**Abstract.** Membrane filtration is one of the main methods of local water treatment. Track-etched membranes allow to obtain high-quality purified water due to their high selectivity. During the filtration, the productivity of process can decrease due to the adsorption of components in the pores, pore blockages, formation of sediment layer above the membrane. To restore the productivity, the membrane should be flushed periodically or regenerated chemically.

Comparative study of the back-flushing and chemical regeneration after natural water filtration using standard 12- $\mu\text{m}$ -thick track-etched membrane and new 20- $\mu\text{m}$ -thick irradiated on both sides (with argon ions with the mileage less than the film thickness) was performed. The research was conducted with natural water from the pond "Zenit" (St. Petersburg) and the Volhov River (Leningrad region). The filtration was conducted in the dead-end mode. Water samples were analyzed by spectrophotometry, spectroturbidimetry and dynamic light scattering.

The size distribution of impurity particles of studied natural water, the change in their sizes during coagulation with an aqua-aurate were determined in work. The mechanisms underlying the decrease of filtration productivity were identified.

The experimental data showed that both-sided irradiated 20- $\mu\text{m}$ -thick membrane has advantages over a standard 12- $\mu\text{m}$ -thick membrane in natural water filtration with impurities that block the pores both in the direct filtration process and in back-flushing and regeneration. The possibility of regeneration and back-flushing of the 20- $\mu\text{m}$ -thick membrane allows us to recommend it for natural water filtration.

**Аннотация.** Мембранная фильтрация является одним из основных методов локальной водоочистки. Трековые мембраны позволяют получать очищенную воду высокого качества благодаря их высокой селективности. Во время фильтрации производительность процесса может уменьшаться за счет адсорбции в порах, закупорки пор или формирования осадка над мембраной. Для восстановления производительности мембрану следует периодически промывать или регенерировать химическим путем.

В работе проведено сравнительное исследование процесса обратной промывки и химической регенерации трековой мембраны толщиной 12 мкм, и новой мембраны толщиной 20 мкм, облученной с обеих сторон (ионами аргона с пробегом меньше толщины пленки), после фильтрации природной воды. В экспериментах использовали природную воду из пруда «Зенит» (Санкт-Петербург) и реки Волхов (Ленинградская обл.). Фильтрацию проводили в тупиковом режиме. Образцы воды анализировали методами спектрофотометрии, спектротурбидиметрии и динамического светорассеяния.

В работе определено распределение по размерам примесных частиц используемых природных вод, изменение их размеров при коагуляции аква-ауратом. Определены механизмы, лежащие в основе снижения производительности фильтрации.

Полученные результаты показали преимущество новой, двусторонне облученной трековой мембраны толщиной 20 мкм в процессах фильтрации природной воды, содержащей примеси, способные задерживаться в порах мембран, обратной промывки и химической регенерации. Возможность промывки и регенерации позволили рекомендовать трековую мембрану толщиной 20 мкм для очистки природной воды в питьевых целях.

## 1. Introduction

The problem of providing the population with pure drinking water is still unresolved and in a number of countries it has reached crisis proportions [1, 2]. A particularly acute issue is the problem of providing water of appropriate quality to individual consumers living in villages, small towns, and housing estates [3–6]. Local water treatment technologies could vary greatly depending on the quality of water, the presence of accessible materials (sorbents, membranes and natural filtering materials [7, 8]), the possibility of material utilization, strength characteristics, and so on.

Microfiltration on the track-etched membrane was chosen as the primary method of water treatment because of its high-energy efficiency [9], high quality of water treatment, and due to its compactness [10–12]. Track-etched membranes are characterized by its increased flexibility, selectivity, resistance to most acids and organic solvents, alkalis and the possibility of regeneration [13–18].

During the microfiltration, the productivity of process decreases due to the following reasons:

- adsorption of components in the pores (the components are substantially smaller than the pore sizes, but they have time to be adsorbed on the walls of membrane pores);
- pore blockages (components are commensurate with pores);
- formation of sediment layer above the membrane (components larger than pores, a dynamic membrane is forming) [19].

To increase the productivity of membranes conduct:

- direct flushing at tangential mode (destroying the dynamic membrane formed on the surface of the original membrane);
- backwashing (from the reverse side of the membrane with a filtrate or pure solvent);
- regeneration with reagents (with stopping the filtration process for contact of membrane with reagents and subsequent washing the membrane off the reagent) [20–23].

Hydraulic washes are using to increase the lifetime of the membrane, but the greatest effect is achieving by using chemical washes. Correct selection of reagents, whose purpose is to transfer the deposits into a soluble form, effects on the efficiency of chemical purification. In order to choose the reagents correctly it is necessary to know the composition and structure of pollutants [24]. The most unfavorable variant of dropping the productivity of the filtration process is clogging of the pores.

The research objects are new, both-sided irradiated 20- $\mu$ m-thick track-etched membrane [25] and a standard 12- $\mu$ m-thick membrane. Productivity and efficiency of water purification process using 20- $\mu$ m-thick track-etched membrane was studied in [13].

The aim of this study is comparing the effectiveness of back-flushing and regeneration of these membranes during the filtration of natural water containing impurities, capable to be retained by the pores of membranes.

To achieve this goal, it was necessary to solve the following tasks:

1. Determine the distribution of impurity particles over size in the samples of studied water sources;
2. Carry out experiments of water samples filtering on the compared membranes in a dead-end mode; identify the mechanisms underlying the decrease of filtration productivity;
3. Compare the possibility of recovering the filtration productivity on both membranes by backwashing with distilled water and chemical regeneration;
4. Check the possibility of coagulation by aqua-aurate of colloidal impurities of studied natural waters to a size exceeding membranes pore size.

## 2. Methods and Materials

Experiments on backwashing and regeneration were carried out on two track-etched membranes. On the new both-sided irradiated track-etched membrane based on 20- $\mu\text{m}$ -thick polyethylene terephthalate film received by irradiation of argon ions with the mileage less than the film thickness, with a pore diameter 0.22  $\mu\text{m}$ , a pore density  $1.5 \times 10^8 \text{ cm}^{-2}$ . And on a standard 12- $\mu\text{m}$  thick track-etched membrane with a pore diameter 0.205  $\mu\text{m}$ , a pore density of  $2.3 \times 10^8 \text{ cm}^{-2}$ .

The filtration was carried out on water samples from pond "Zenit" near "Zenit Sports Games Palace" (St. Petersburg, Butlerova street, 9) and the Volhov River near the Podol village (Volhov district).

Before carrying out experiments on natural water, an experiment on the possibility of flushing the membrane by distilled water filtration was carried out. In this experiment, distilled water was first conducted in the forward direction, and then the membrane was washed, by filtering in the opposite direction.

The studies were conducted in a dead-end mode, because: it allows to determine the reason of productivity dropping and to ensure the clogging of pores with a smaller volume of sample faster than in the tangential mode. The pressure was maintained constant at 0.3 atm.

For flushing and regeneration, a dead-end model in a cell with filtration area 25.5  $\text{cm}^2$  and a volume 200  $\text{cm}^3$  was used. In the experiments of raw natural water, filtrates, and samples after flushing and regeneration, the following methods and instruments were used:

- Spectrophotometry and spectroturbidimetry methods (for determination concentrations of individual components) on KFK-3.01 photoelectrocolorimeter, SF-56 spectrophotometer;
- Dynamic light scattering - a dispersion analysis of nature water was performed on a Zetatrac laser analyser [26];

Filtration productivity  $G$ ,  $\text{cm}/(\text{c} \cdot \text{bar})$ , was determined by the formula (1):

$$G = \frac{V}{t \cdot P \cdot S}, \quad (1)$$

where  $V$  – volume of the sample,  $\text{cm}^3$ ;  $t$  – sampling time, s;  $P$  – pressure, bar;  $S$  – filtration area,  $\text{cm}^2$ .

## 3. Results and Discussion

Experiments on determination of particle size distribution are represented in Figures 1–4.

The obtained results show that the most of suspended impurities in the water samples from Volhov River has a size from 60 to 250  $\mu\text{m}$  (Fig. 2), and in the Zenit pond – from 45 to 150  $\mu\text{m}$  (Fig. 4). Water samples from both water bodies contains an insignificant fraction of large particles (noticeable only in the analysis of the intensity distributions (Figs. 1 and 3).

These results show that water samples from both water bodies mostly contains suspended impurities smaller than the pore size, which could clog the membranes pores. The smallest impurities could be adsorbed in the pores of track-etched membranes.

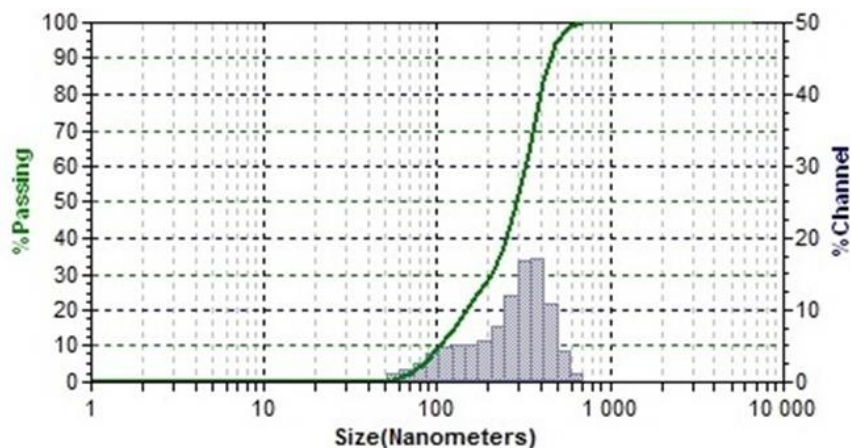


Figure 1. Intensity distributions over particle sizes for water sample from Volhov River

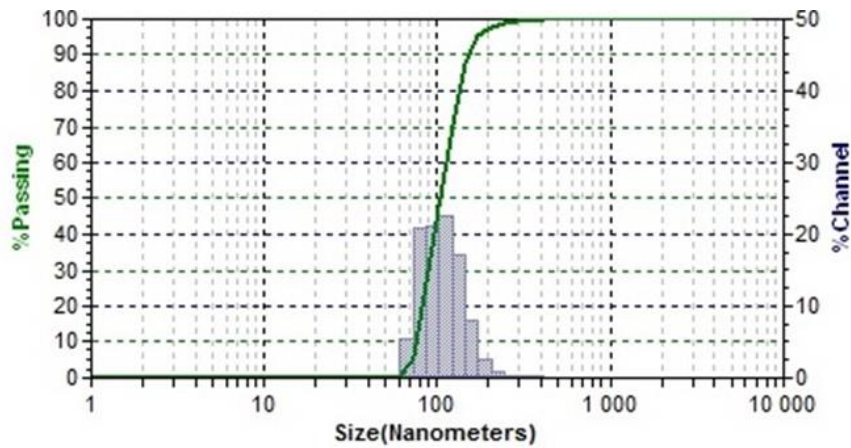


Figure 2. Number distributions over particle sizes for water sample from Volhov River

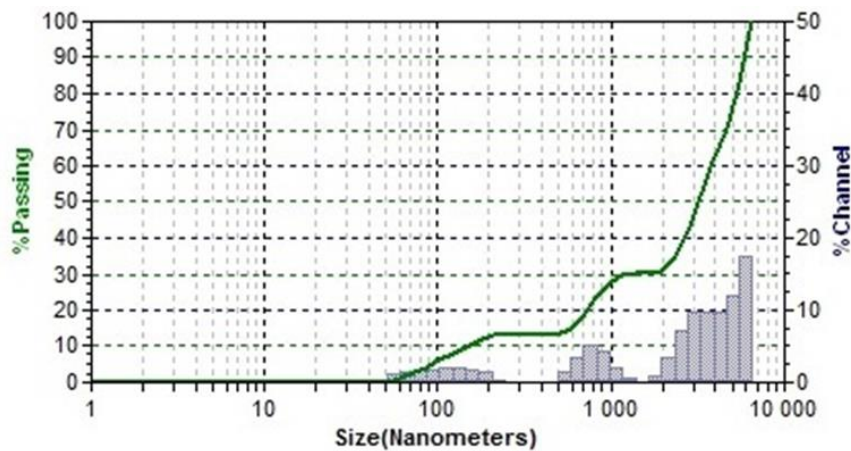


Figure 3. Intensity distributions over particle sizes for water sample from a pond "Zenit"

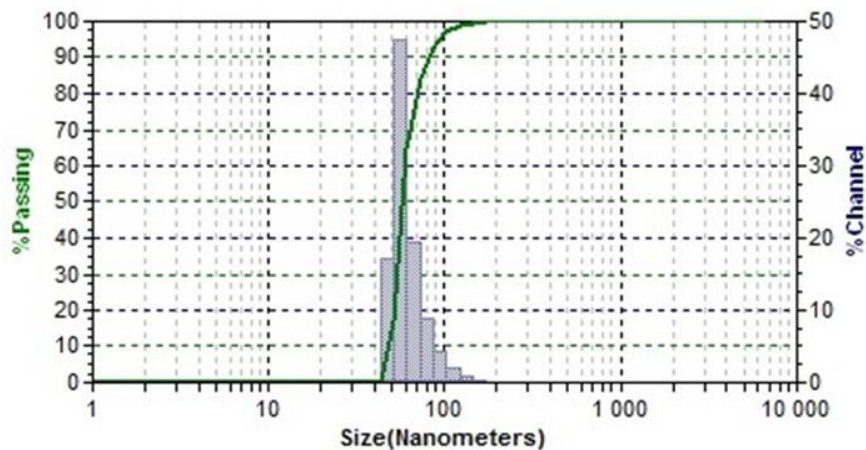
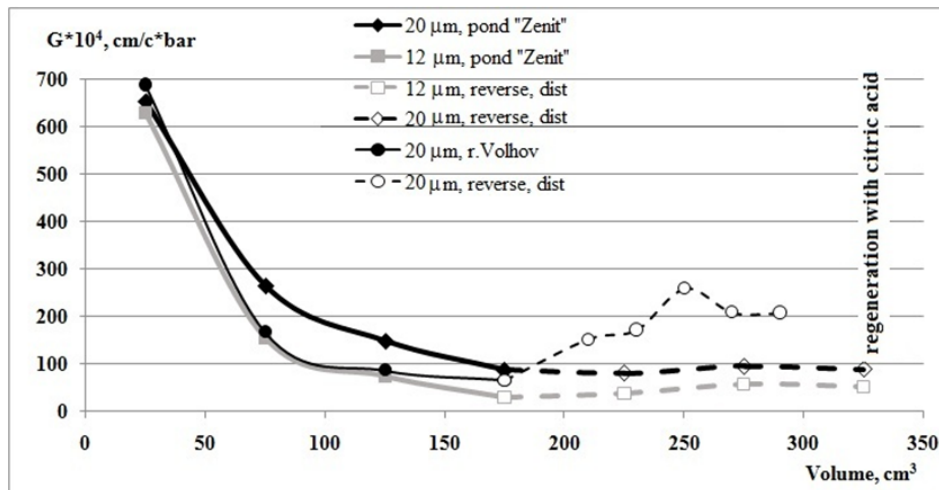


Figure 4. Number distributions over particle sizes for water sample from a pond "Zenit"

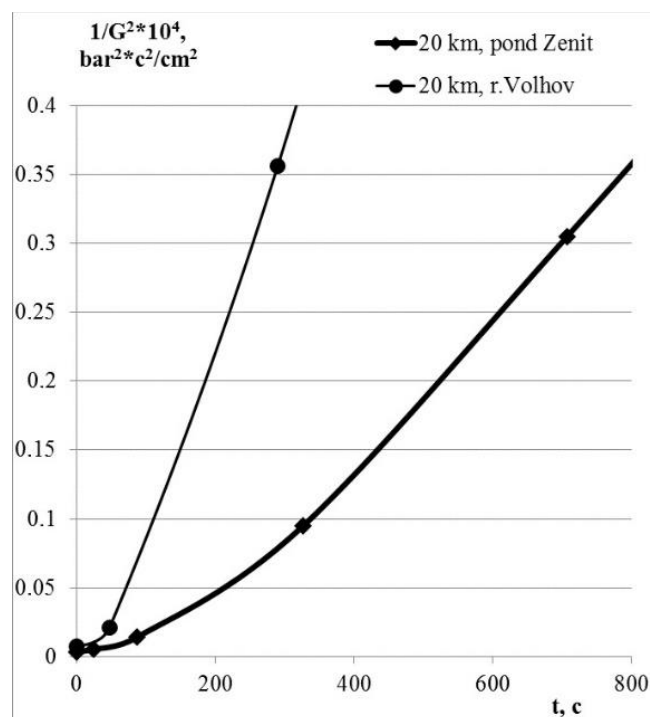
The results of filtration the water samples from both sources through the track-etched membranes are shown in Figure 5. It could be seen that the productivity of filtration falls quickly while filtering water samples with small impurity particles. After productivity dropped by 10–15 times, the membranes were flushed with distilled water in reverse direction. After filtration of water samples from the pond Zenit, the productivity has not recovered, but after filtration of water from the Volhov River – the membrane's productivity was restored to 1/3 of the initial value.



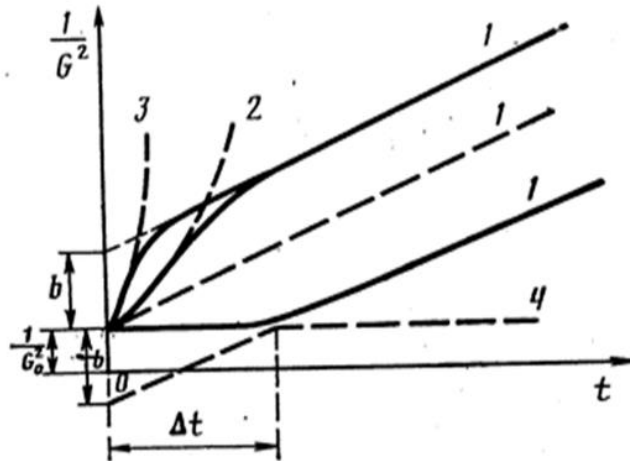
**Figure 5. Dependences of the productivity of dead-end filtration water from pond "Zenit" and river Volhov through 12- $\mu\text{m}$ -thick and 20- $\mu\text{m}$ -thick membranes, and reverse washing of these membranes with distilled water**

To explain the different results of reverse flushing (of both-sided irradiated membrane) after filtration of the water samples from the pond Zenit and r. Volhov the dependencies of the square of the reverse productivity on the time of filtration were built and analyzed (Fig. 6).

The obtained graphs (Fig. 6) were compared with the theoretical ones (Fig. 7) [19]. It was concluded that in the water samples from the Volhov River the pore blocking mechanism dominates, and for water samples from the Zenit pond adsorption in the pores dominates. These conclusions correspond to the particle size distribution in these waters and explain the greater efficiency of reverse washing for water samples from the Volhov River.



**Figure 6. The initial section of the dependences of the reverse productivity on time through the both-sided irradiated membrane**

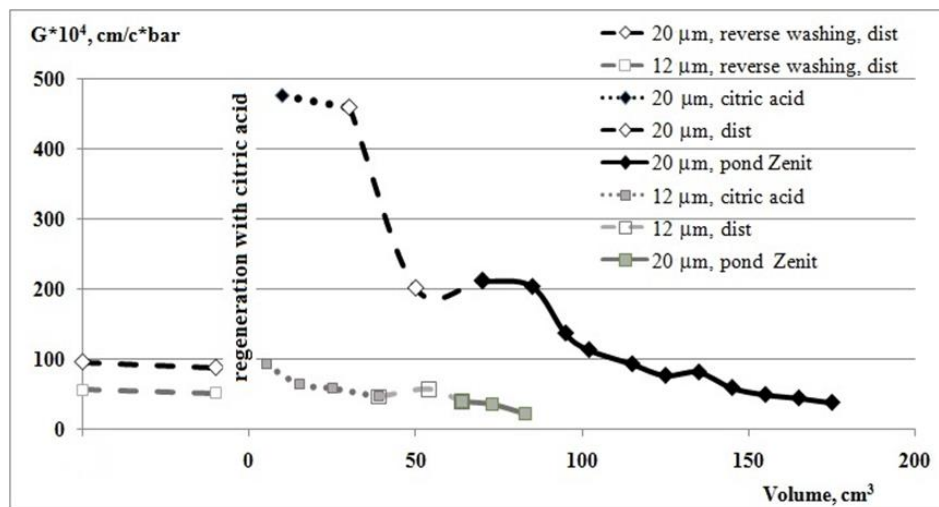


**Figure 7. Dependences of the square of the reverse productivity on time, corresponding to:**  
**1 – formation of a precipitate on the membrane surface; 2 – adsorption in pores;**  
**3 – blockage of pores; 4 – pregel polarization [19]**

For 12 and 20- $\mu\text{m}$ -thick track-etched membranes after filtration of water samples from the Zenit pond and ineffective backwashing with distilled water, regeneration with citric acid was carried out (Fig. 8).

It could be seen (Fig. 8) that for a both-sided irradiated track-etched membrane, the productivity of filtration increases in 5 times during the regeneration, while for a one-side irradiated standard 12- $\mu\text{m}$ -thick membrane productivity increase less than 2 times.

At the same time, when filtering the source water (from the Zenit pond) through the regenerated membranes, the productivity of the filtration process decreases quickly again.



**Figure 8. Change in productivity of dead-end filtration after regeneration with citric acid**

Hence, for natural waters containing suspended impurities, which size is much smaller than the size of the pores of the track-etched membrane, backwashing and regeneration do not provide a constant productivity. In this case, it is possible to offer preliminary coagulation of suspended impurities [11].

It was shown for track-etched membranes [11], that when cleaning waters with high turbidity and color, an effective method for prevent a drop in filtration productivity and increasing the efficiency of backwashing is to combine the coagulation and microfiltration processes. We tested the effectiveness of preliminary coagulation of colloidal impurities for low-turbidity waters to dimensions exceeding the membrane pore size.

A modern coagulant aqua-aurate based on aluminum oxychloride was used in our experiments. The analysis of the particle size distribution (Figs. 9, 10) showed that within 5–10 minutes after the aqua-aurate addition (concentration of  $\text{Al}_2\text{O}_3$  was 5 mg/L) a noticeable particles coagulation takes place. In subsequent studies, it is necessary to select the concentration of the coagulant and the time it takes to

enlarge the floccula to a size exceeding  $0.2 \mu\text{m}$ , not only in terms of "intensity distributions", but also in "number distributions". It allows to receive more efficient process of membrane filtration (of such waters).

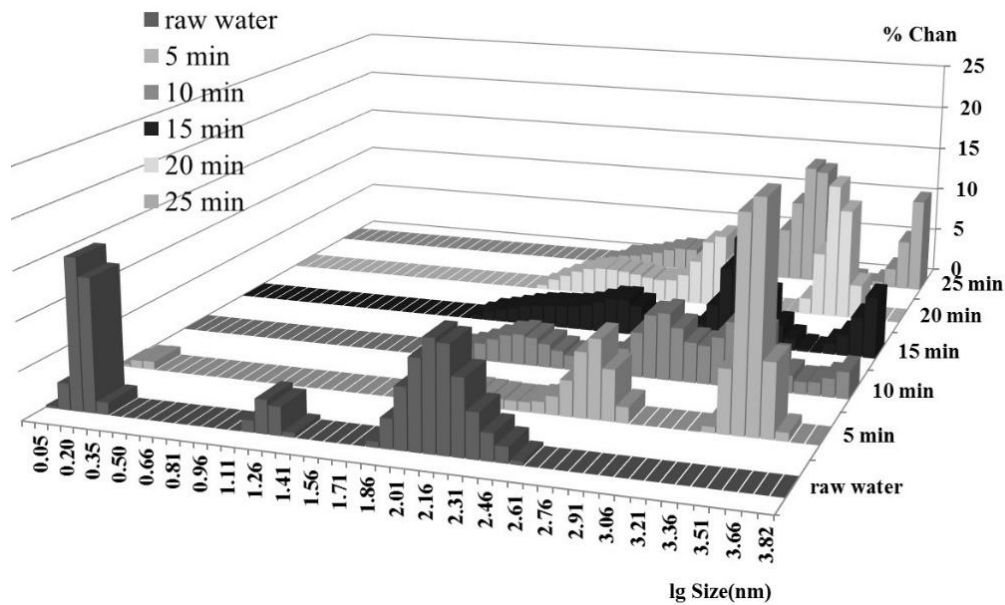


Figure 9. Intensity distributions over particle sizes for water sample from a pond "Zenit"

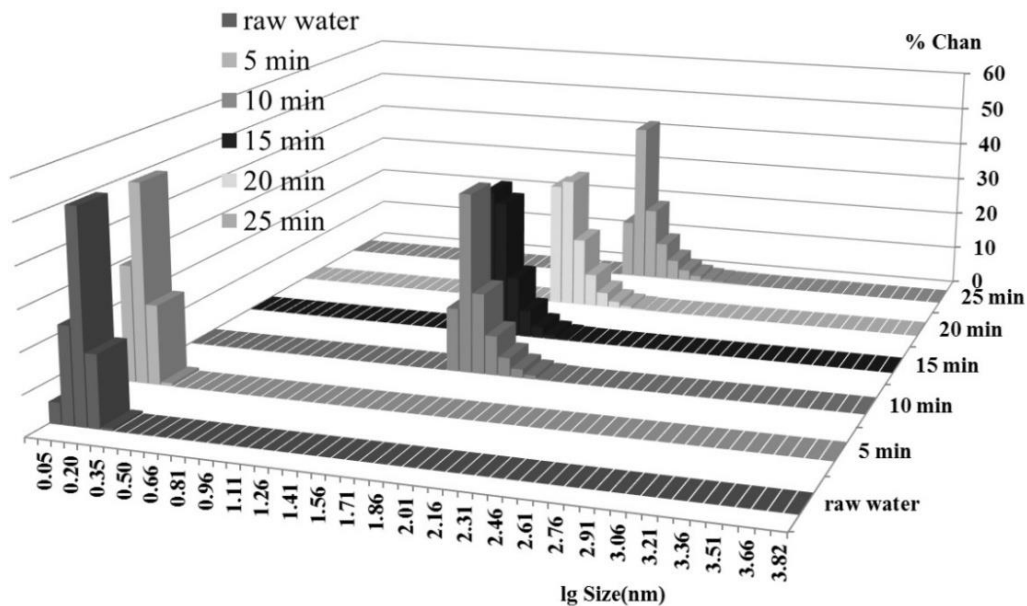


Figure 10. Number distributions over particle sizes for water sample from a pond "Zenit"

#### 4. Conclusions

1. Both-sided irradiated 20- $\mu\text{m}$ -thick membrane with a pore diameter  $0.20\text{--}0.205 \mu\text{m}$  has shown advantages over a standard 12- $\mu\text{m}$ -thick membrane in natural water filtration with impurities that block the pores both in the direct filtration process and in flushing and regeneration (with other equal parameters).

2. To prevent pore clogging, preliminary coagulation of impurities can be used.

3. The possibility of regeneration and flushing of the 20- $\mu\text{m}$ -thick membrane allows us to recommend this membrane for natural water purification for local water supply in such objects of civil engineering as individual buildings.

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