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## METHODS FOR REGENERATION AND STORAGE OF CERAMIC MEMBRANES

### Abstract

Ceramic membranes are among one of the most promising candidates for membrane applications, owing to their excellent resistance to mechanical, chemical, and thermal stresses. These advantages make them an attractive filter material. An additional benefit which is extremely important for the industry, is their possibility of continuous operation at high efficiency while maintaining constant transmembrane pressure. Due to the inorganic material from which they are made, ceramic membranes have the possibility of being cleaned by steam sterilization and are resistant to micro-organisms. Although, due to low production costs, ceramic membranes are one of the most cost-effective membrane filtration technologies they are prone to substantial fouling. When used, a layer of contaminants is formed on the active surface, often reducing or completely filling the membrane pores resulting in fouling and concentration polarization. These phenomena cause a decreased efficiency of the process, which leads to the need for the membrane to be replaced with a new one. However, ceramic membranes have the possibility of being regenerated through a series of activities and the use of various chemical agents. The use of regenerated membranes would provide the opportunity to reduce exploitation costs. Although membrane regeneration does not guarantee a return to the initial parameters, it does allow for the recovery of high permeation flow. The aim of the research was to compare operating parameters of the ceramic membranes after multiple use and longtime storage with different condition of storage.

### Key words

ceramic membranes, fouling, membranes regeneration

### Introduction

Development of separation techniques are extremely important for industries using membranes. Chemical engineering scientists are working on increasing the conditions and parameters of the process. These separation techniques are well adapted to the use of ceramic membranes, which thanks to its features fulfil current needs and are distinguished in the use of available methods. Ceramic membranes remain one of the most promising technologies among membrane processes. This technology has many applications such as specialized wastewater treatment processes or enriching uranium. Ceramic membranes have also been adopted in the food industry to separate proteins and fats in milk and to concentrate fruit juices. Other applications include biotechnology, the pharmaceutical industry, petrochemistry, metallurgy and energy generation [1,2]. Ceramic membranes have many advantages, including durability, chemical stability and resistance to high temperatures. They are used in processes with specific technological requirements where the use of polymeric membranes is impossible [3]. Ceramic membranes allow operations at high flow rates while maintaining constant transmembrane pressure. This technology makes it possible to carry out the process in a continuous mode with high separation ability. Ceramic membranes enable microfiltration (MF), ultrafiltration (UF) and nanofiltration (NF) with pores diameters from 0,5 nm to 5  $\mu\text{m}$  [4,5].

Ceramic membranes have a porous, asymmetrical structure. They usually consist of two or three layers of different porosity called the support and active layers. The active layer determines the separation properties of the membrane and is a selective barrier to mass transport. Usually, this layer is covered by aluminum oxide, titanium, zirconium and silicon. Each oxide has a different surface charge, which reacts with other substances. The active layer is applied by dipping the ceramic support elements in a suspension of the above compounds, which are then dried and fired [7]. The support layer is only a carrier that does not affect the separation capacity and is made of ceramic powder that can be pressed and then dried and baked. Ceramic membranes are produced in the form of flat plates or in a single or multi-channel tubular form with a circular or hexagonal cross-section, as shown in Fig. 1. In industry, multi-channel membranes are most commonly used due to their increased effective exchange surface.

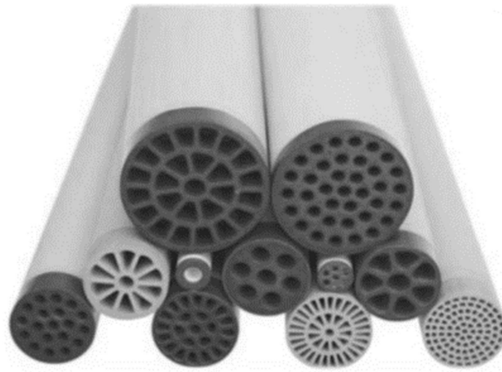


Fig. 1. Examples of single and multi-channel tubular ceramic membranes

Source: [6]

Most often, ceramic membranes are used for filtration in the cross flow mode. This technology is based on a parallel stream feed (tangent to the surface of the membrane), which is separated into a filtrate flowing through the pores (permeate) and a retentate consisting of retained particles. The driving force of the process is the differential pressure placed on the membrane (transmembrane pressure). Ceramic membranes can be used in high-temperature processes (up to 300°C) having extreme acidity or alkalinity. They are more stable than polymeric membranes and their lifetime is much longer [8]. However, their cost is much higher than polymeric membranes. Ceramic membranes are also characterized by high microbiological resistance. Their unquestionable advantage is the possibility of sterilization by means of steam and backwashing. There are two main ceramic membranes commercially available in Europe: Membralox GP (graded permeability) manufactured by Pall Corporation and Isoflux produced by Tami Industries [9].

Unfortunately, as mentioned, the primary challenge for using ceramic membranes is fouling. This results in a decrease in the permeate flow and an increase in the process resistance dependent on the duration of filtration. Fouling consists in the deposition of colloids, salts and particles in the pores and on the surface of the membrane. This can be caused by several factors, including among others, blocking and narrowing the pores (filtration cake), adsorption of filtered substances and concentration polarization [10]. In order to prevent the negative effects of fouling of ceramic membranes, it is recommended maintaining high cross-flow velocities and low transmembrane pressures [11]. In addition, when the filtration flux drops significantly, it is necessary to clean the membrane so that as much as possible, it regains its original parameters. This can be done in many ways, e.g. backwashing or chemical washing.

Therefore, presented difficulties associated with the application of ceramic membranes it was decided to examine the effect of the method of storing. Membrane was use before and has some fouling load caused by previous process. Optimization of membranes storage parameters can increase their industry adaptation, and above all at low costs, improve the operational parameters of the operation. In the experiment, it was use a hydrogen peroxide solution as an oxidant for contaminants caused by foulants, added to the distilled water during storage.

#### **Ceramic membrane fouling and regeneration**

The performance of ceramic membranes decreases with the duration of the process after each filtration. This problem is most often associated with fouling, which significantly limits the permeability and increases flow resistance. As a result, it is necessary to either replace the membrane or regenerate it. In the case of ceramic membranes, it is possible to remove part of contamination from the pores by regeneration, however initial efficiency cannot be restored [12-14]. Fouling can cause adsorption of components on the surface of the membrane and inside the pores, deformation of the membrane, reaction of molecules with membrane material, reduction of the diffusivity of particles in the pores as well as bacterial growth. The symptoms of this phenomenon are a decrease of the permeate flux during the duration of the process while maintaining a constant pressure or an increase of pressure over time at constant filtration rate conditions. Initially, separated components of a size smaller than those of the membrane's pores become partially deposited on the pore walls leading to a reduction in pore size. This results in a significant reduction of the cross-section and the surface available for filtration. Also, larger particles are adsorbed at the pore entrances, causing significant

decrease of the flow and clogging of the pores. Both cases lead to the accumulation of retained particles on the surface of the membrane, increasing its resistance.

The phenomenon of fouling has been described by the classic blocking laws of filtration as first described by Hemans and Bredée in 1935 [15], and then modified by Grace (1956) [16], Shirato et al. (1979) [17] and by Hermia (1982) [18]. Initially, this referred to the traditional filtration of liquids under constant pressure, however, currently they are used to describe the phenomena of permeate flux decline in membrane microfiltration and ultrafiltration. The model of blocking consists of four different filtration blocking mechanisms: total blocking, indirect blocking, standard blocking and the filtration cake (Fig.2). The first two phenomena describe the surface clogging of pores and refer to fouling by particles larger than the membrane pores. Standard blocking refers to the accumulation of contaminants in the pore walls, which leads to a reduction in their diameter. The mechanism of creating a filter cake is focused on the increase of the thickness of film consisting of accumulated particles on the surface of the membrane. The models assume that the pores in the membrane are parallel and have the same geometry (diameter and length) [10].

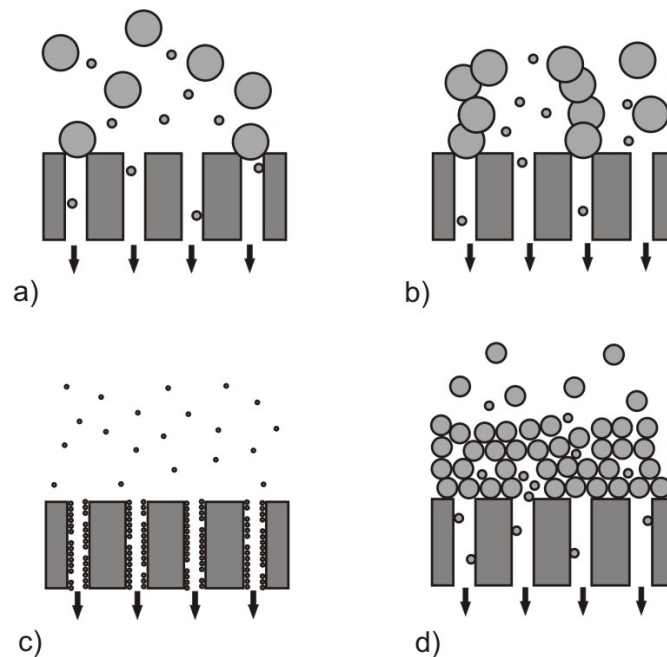


Fig. 2. Blocking filtration mechanisms: a) total blocking model, b) indirect blocking model, c) standard blocking model, d) filtration cake

Source: [10]

Fouling is unavoidable during filtration and essentially limits the ability to purify the membrane system. It is necessary to develop an innovative method of fouling reduction in order to minimize operational costs related to the maintenance of membrane filtration [8]. So far, many methods of regeneration have been invented, however, research into the discovery of a process of the highest efficiency is still in progress.

Currently, back washing is the most popular method of fighting against fouling. This method involves washing the membrane in the opposite direction than the normal membrane permeate flux. Unfortunately, this technique brings with it a high risk of damaging the active surface and structures of the membrane [19]. Destruction of the membrane causes a significant increase in installation costs. Although ceramic membranes can be regenerated using various chemical agents. A short view of the substances used is presented in Table 3. Yin and Xing (2013) investigated the fouling and cleaning of ceramic membranes after wastewater desulphurization. They found that the most effective membrane cleaning solution was 1% NaOH mixed with 0.5% NaClO [20]. Li et al. (2018) led in the process of ultrafiltration of limed sugarcane juice by ceramic membranes. To oppose fouling, membranes are washed in distilled water, followed by a mixture of 1.0% NaOH and 0.5% NaClO (similar to above) and a 0.5% HNO<sub>3</sub> solution. This process produces a repetitive permeate flux recovery at a level of 96.6% [21].

One of the latest methods used in water treatment technology is the combination of filtration using ceramic membranes and aeration by Air nano Bubbles [10]. Air Nano Bubbles are spherical air bubbles, 10-20 nm high and 50-100 nm wide [22]. So far, they have been used in wastewater treatment processes: for coagulation and aeration aimed at increasing the activity of aerobic microorganisms and water disinfection [23]. The membrane filtration technology with nano air bubbles effectively prevents and reduces the blocking of membrane pores, breaking the film of contaminants accumulated on the surface. Air nano Bubbles exhibit degrading properties of organic matter, which makes them an ideal solution for filtration of humic acids and proteins, e.g. BSA (Bovine Serum Albumin). Nano air bubbles remove contaminants with minimal observed side effects. Using this technology allows extension of membrane's life and reduces process costs.

Table 1. Common chemical cleaning agents used for chemical cleaning of ceramic membranes

Family	Examples	General functions
Acids	Strong: HCl, HNO <sub>3</sub> Weak: H <sub>3</sub> PO <sub>4</sub>	pH regulation, dissolution of inorganic precipitates, acidic hydrolysis of certain macromolecules
Alkalis	Strong: NaOH, KOH Weak: Na <sub>2</sub> CO <sub>3</sub>	pH regulation, alteration of surface charges, alkaline hydrolysis of proteins, catalyzing saponification of fats
Oxidants	NaClO, H <sub>2</sub> O <sub>2</sub>	Oxidation of organics; disinfection
Surfactants	Anionic: SDS Cationic: CTAB Nonionic: Tween 20	Dispersion/suspension of deposits
Chelates	EDTA	Complexion with metals, removal of mineral deposits.
Enzymes	Proteases, lipases	Catalysis of specific substrates (e.g., proteins, lipids)

Source: [24]

### Materials and methods

The measurements were carried out in a laboratory-scale installation working in a cross flow mode with module for one membrane, made by Intermasz company. In industrial equipment is possibility of multi stream flow wit application of many membranes module to increase efficiency. Tubular, microfiltration ceramic membrane produced by Tami Industries was used in the research . The main parameters of the investigated membrane are shown in Table 2.

Table 2. Characteristics of tubular ceramic membrane use in tests

Active layer	TiO <sub>2</sub>
Average pores [μm]	0,2
Diameter inside/outside [mm]	6/10
Length [mm]	600
Filtration area [m <sup>2</sup> ]	0,011
Number of channel	1
Operating pressure [MPa]	< 1 MPa
Operating temperature [°C]	< 300°C

Source: [25]

The experimental equipment consisted of a 10 dm<sup>3</sup> supply tank, a pressure circulating pump and a membrane module prepared for the used membrane. The installation is equipped with manometers from the feed and permeate to determine the transmembrane pressure. The stream temperature was thermostatically controlled by cooling water. The value of the flow rate depends on the transmembrane pressure. The membrane tested was already used for the microfiltration of milk and then for 2 months stored in distilled water. The membrane would be clogged by fats and large proteins. The aim of the study was to investigate operating parameters of the ceramic membranes after multiple use and longtime storage. The membrane hydrodynamics are determined by the following parameters:

- water permeate flux -  $J_v$

$$J_v = \frac{V}{t * A} \left[ \frac{m^3}{m^2 * h} \right] \quad (1)$$

V - measured permeate volume [ $m^3$ ] in time t [h]

A- active filtration area [ $m^2$ ]

- membrane resistance-  $R_m$

$$R_m = \frac{\Delta P}{J_v * \eta} [m^{-1}] \quad (2)$$

$\Delta P$  - transmembrane pressure [Pa]

$\eta$  - dynamic water viscosity [Pa·s]

The experiment was carried out on the hydrodynamic of the membrane stored for 2 months in distilled water. These tests showed low permeate flux. Therefore the storage in 0.5% V/V  $H_2O_2$  solution for 2 weeks was tested. The solution was prepared by mixing 80 ml of 30%  $H_2O_2$  from the POCH manufacturer with 5  $dm^3$  of distilled water. The idea of adding hydrogen peroxide to distilled water during storage has been taken from the membrane operating instructions [6,25]. It has been suggested there to use  $H_2O_2$  in the case of heavy contaminants on the membrane. Hydrogen peroxide is a popular oxidant, has a bactericidal effect and is cheap and easily available. The concentration proposed in the data sheet is 0.2% V/V. The experiment decided to try a higher concentration due to several tests without satisfactory results. The investigations of hydrodynamics were performed at temperatures of 25°C, 50°C and 80°C with transmembrane pressure equal to values between 0,2-0,5 MPa.

## Results

The tests began with rinsing with distilled water. The hydrodynamics (the permeate flux for different transmembrane pressure for water stream) of the processes were investigated. The results of membrane permeability of distilled water is presented in Table 3. The viscosity of water was taken in accordance with literature data [26].

Table 3. Membrane permeability for water after storage in distilled water

T [°C]	$\eta$ [Pa*s]	$\Delta p$ [Pa]	V [ $cm^3$ ]	$t_{sr}$ [s]	Jv [ $m^3/s*m^2$ ]	Jv [ $m^3/h*m^2$ ]	Rm [ $m^{-1}$ ]
25	8,91E-04	200000	10	50,01	1,82E-05	0,065	1,23E+13
		250000	10	37,21	2,44E-05	0,088	1,15E+13
		300000	10	29,72	3,06E-05	0,110	1,10E+13
		350000	10	25,26	3,60E-05	0,130	1,09E+13
		400000	10	20,14	4,51E-05	0,162	9,95E+12
		450000	10	17,74	5,12E-05	0,184	9,86E+12
		500000	10	16,03	5,67E-05	0,204	9,90E+12
50	5,52E-04	200000	20	42,66	4,26E-05	0,153	8,50E+12
		250000	20	32,61	5,58E-05	0,201	8,12E+12
		300000	20	27,85	6,53E-05	0,235	8,33E+12
		350000	20	23,66	7,68E-05	0,277	8,25E+12
		400000	20	20,21	9,00E-05	0,324	8,06E+12
		450000	20	17,54	1,04E-04	0,373	7,87E+12
		500000	20	15,29	1,19E-04	0,428	7,62E+12
80	3,56E-04	200000	20	28,80	6,31E-05	0,227	8,90E+12
		250000	20	23,22	7,83E-05	0,282	8,97E+12
		300000	20	20,09	9,05E-05	0,326	9,32E+12
		350000	20	18,02	1,01E-04	0,363	9,75E+12
		400000	20	16,74	1,09E-04	0,391	1,04E+13
		450000	20	14,69	1,24E-04	0,446	1,02E+13
		500000	20	13,33	1,36E-04	0,491	1,03E+13

Source: Author's

The driving force behind the microfiltration process is the pressure difference, therefore along with an increase in transmembrane pressure, the permeate flow also increases. Figure 3 shows that the hydrodynamics of the membrane exhibits a linear relationship, and the stream values increase with increased feed temperature which is connected with viscosity of the water and was included in the calculations.

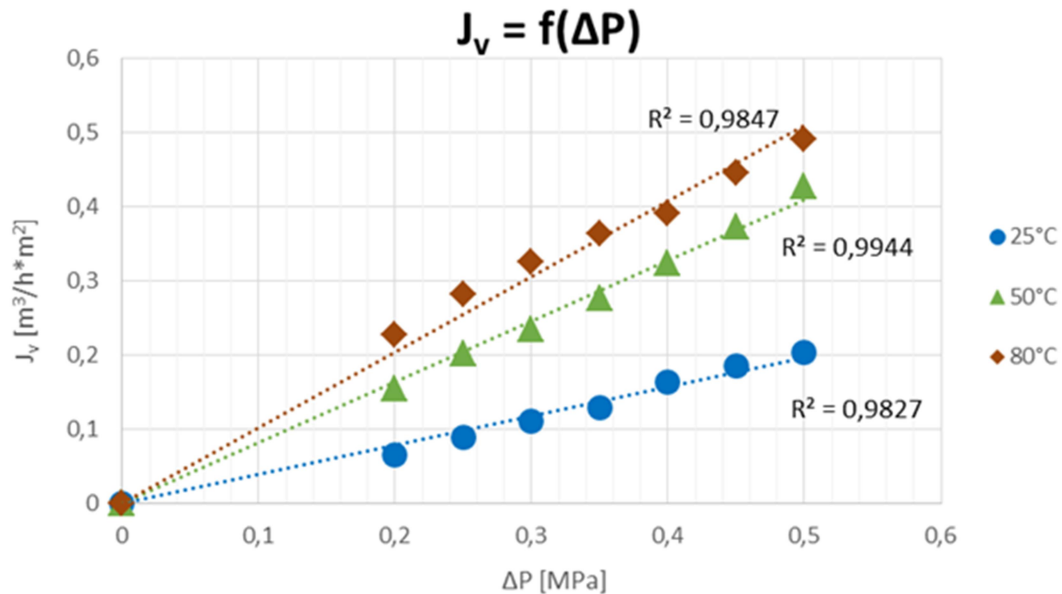


Fig. 3. Effect of transmembrane pressure on permeate volume flux for a membrane stored in distilled water  
Source: Author's

Figure 4 shows the tendency of membrane resistance measured for different values of transmembrane pressure. It can be noticed that as the temperature of the media increases the membrane resistance decreases. However, at a temperature of 80 °C, the trend of membrane resistance increases. The tests of permeate flux and membrane resistance were made during one session. There is a possibility that previous experiment made elimination of the foulants at membrane surface which could expose pores of membrane and the particles from the surface could clog and decrease pores of membrane. As a result, the membrane pores have been reduced and resistance would increase.

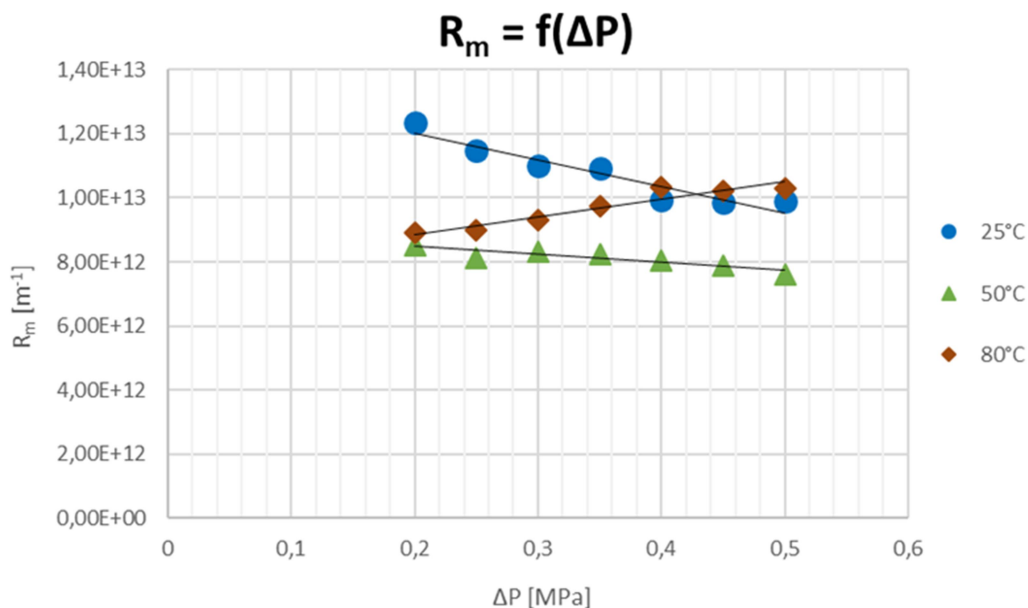


Fig. 4. Effect of transmembrane pressure on membrane resistance for a membrane stored in distilled water  
Source: Author's

Despite multiple regeneration performed in accordance with recommendations (Table 4) the ceramic membrane showed results below expectation. The values of the permeate flux were too low, therefore the

storage method was changed. The membrane was stored in a 0.5% solution of hydrogen peroxide for 2 weeks, after which tests were repeated. The results are shown in Table 5.

Table 4. The sequence of membrane installation rinsing according to instructions.

cleaning agent	temperature [°C]	rinsing time [min]
H <sub>2</sub> O	25	20
NaOH 0,1M	80	40
H <sub>2</sub> O	25	10
H <sub>3</sub> PO <sub>4</sub> 1%	50	20
H <sub>2</sub> O	25	20

Source: [27]

Table 5. Membrane permeability of water after storage in 0,5% H<sub>2</sub>O<sub>2</sub> solution.

T [°C]	$\eta$ [Pas]	$\Delta P$ [Pa]	V [cm <sup>3</sup> ]	t <sub>sr</sub> [s]	J <sub>v</sub> [m <sup>3</sup> /s*m <sup>2</sup> ]	J <sub>v</sub> [m <sup>3</sup> /h*m <sup>2</sup> ]	R <sub>m</sub> [m <sup>-1</sup> ]
25	8,91E-04	100000	10	4,53	2,01E-04	0,723	5,59E+11
		150000	10	2,78	3,28E-04	1,179	5,14E+11
		200000	10	2,18	4,18E-04	1,505	5,37E+11
		250000	20	3,40	5,35E-04	1,925	5,25E+11
		300000	20	2,93	6,22E-04	2,238	5,42E+11
		350000	30	3,73	7,32E-04	2,636	5,37E+11
		400000	30	3,33	8,20E-04	2,953	5,47E+11
		500000	50	4,15	1,10E-03	3,943	5,12E+11
50	5,52E-04	100000	20	5,98	3,04E-04	1,095	5,95E+11
		150000	20	3,83	4,75E-04	1,711	5,72E+11
		200000	20	2,75	6,61E-04	2,380	5,48E+11
		250000	50	5,50	8,26E-04	2,975	5,48E+11
		300000	50	3,94	1,15E-03	4,153	4,71E+11
		350000	50	3,69	1,23E-03	4,435	5,15E+11
		400000	50	3,06	1,49E-03	5,348	4,88E+11
80	3,56E-04	100000	50	8,41	5,40E-04	1,946	5,20E+11
		150000	50	6,08	7,48E-04	2,691	5,64E+11
		200000	50	4,65	9,78E-04	3,519	5,75E+11
		250000	50	3,79	1,20E-03	4,318	5,86E+11
		300000	50	3,14	1,45E-03	5,211	5,82E+11
		350000	50	2,74	1,66E-03	5,972	5,93E+11
		400000	50	2,40	1,89E-03	6,818	5,94E+11

Source: Author's

Figure 5 shows the hydrodynamics of the examined membrane stored in 0.5% H<sub>2</sub>O<sub>2</sub> solutions. The permeate fluxes presented similar relationships as membranes stored in distilled water. Furthermore, Figure 6 shows the membrane resistance for various transmembrane pressure. The values at each temperature oscillated at a similar level.

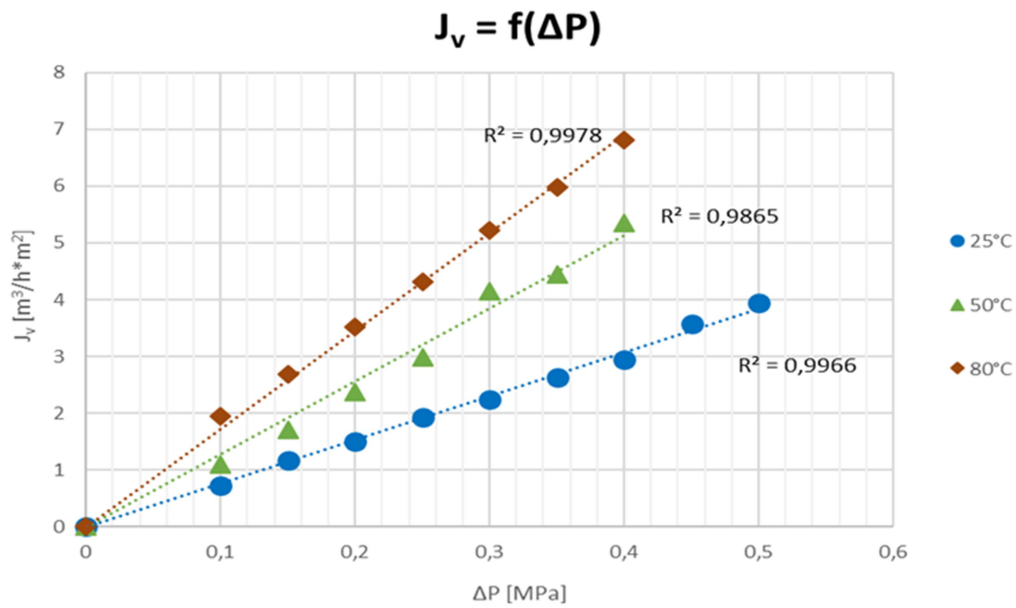


Fig. 5. Effect of transmembrane pressure on permeate volume flux for a membrane stored in 0.5%  $H_2O_2$  solution  
Source: Author's

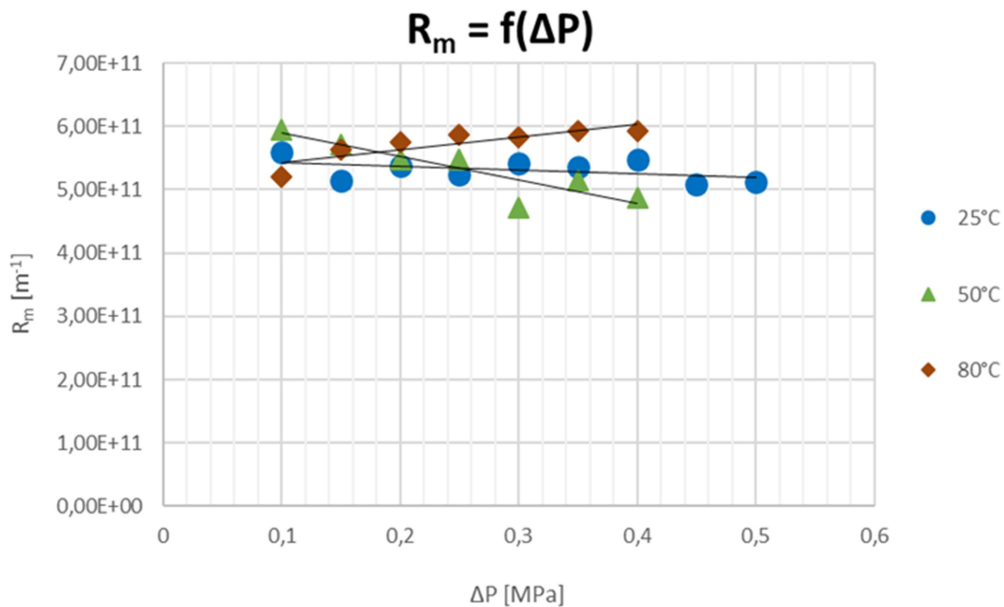


Fig. 6. Effect of transmembrane pressure on membrane resistance for a membrane stored in 0.5%  $H_2O_2$  solution  
Source: Author's

As expected, the permeate flux values for membranes stored in 0.5%  $H_2O_2$  solutions were higher than for membranes stored in distilled water. Corresponding values of membrane resistance were lower than before. The comparison of values at a temperature of 25°C is shown in Figures 7 and 8.



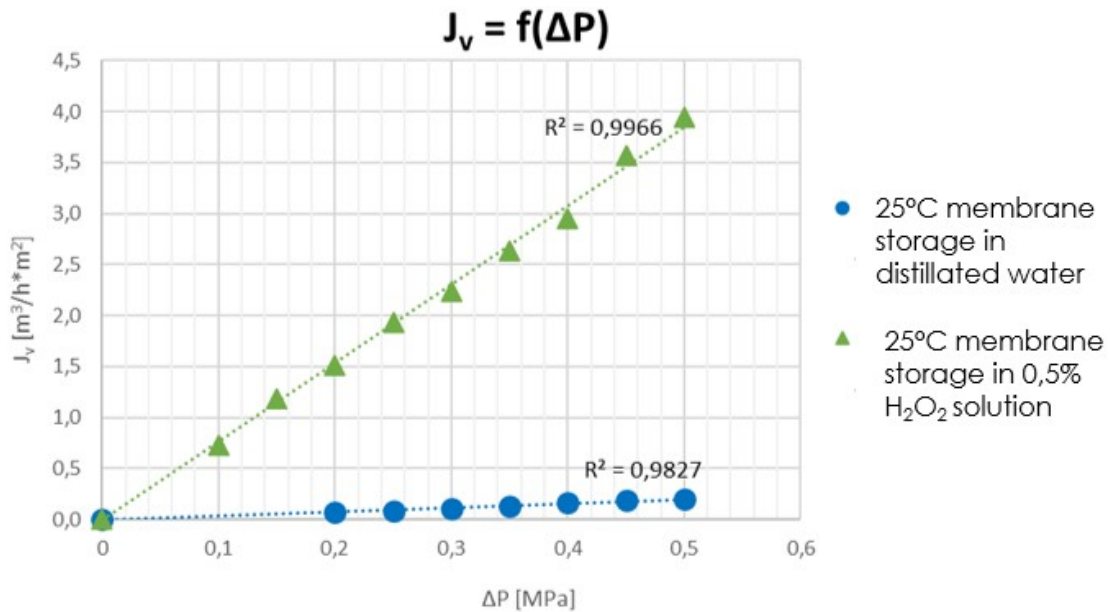


Fig. 7. Comparison of effect of transmembrane pressure on permeate volume fluxes  
Source: Author's

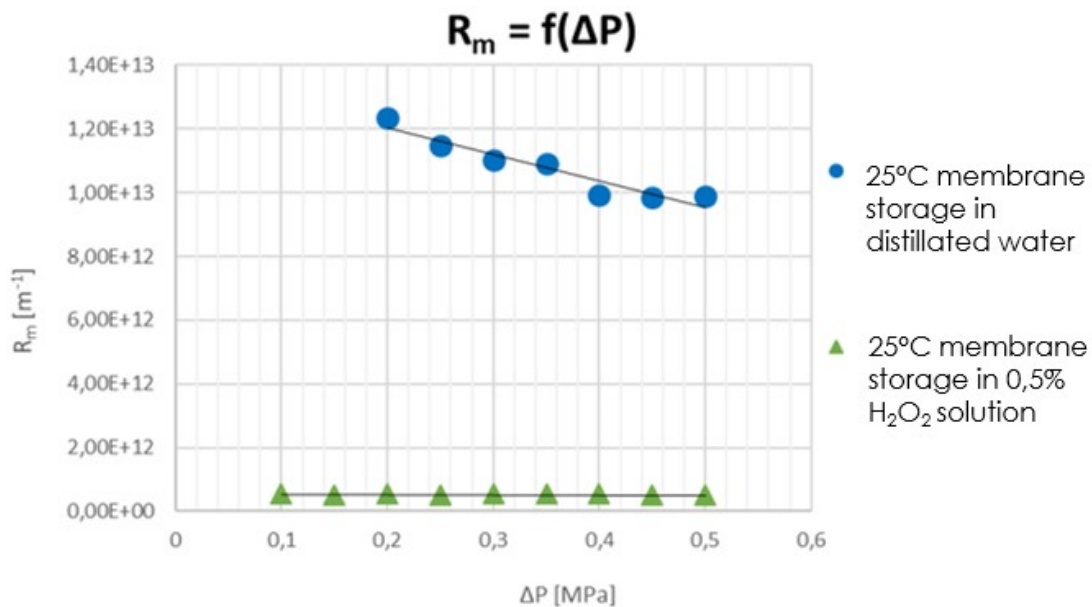


Fig. 8. Comparison of effect of transmembrane pressure on membrane resistance  
Source: Author's

The research has shown that the value of the permeate flux increased about twenty times, also the resistance of the membrane decreased significantly.

### Summary and conclusions

Ceramic membranes are a technology increasingly being used in industries. However, fouling remains an inherent problem, as it significantly reduces the efficiency of the process that requires the fouled membrane to be replaced with a new one. An undoubted advantage of ceramic membranes is the possibility of regeneration. Depending on the medium-filtered, the regeneration can be realized in many ways to recover the highest possible permeate flow. This study investigated the effect of membrane storage on its regeneration. It was shown that the hydrogen peroxide solution positively influenced the renewal of the membrane filtration capacity, when the fouled membranes were stored in 0.5% V/V solution for a period of two weeks which released a significant increase in the permeate flux (about 20 times), with the decrease in flow resistance.

Hydrogen peroxide is an oxidant, which removes mainly organic pollutants and was found to be more effective in this study, as compare to treatments with NaOH and ortho-phosphoric acid solutions. As the regeneration of the fouled membranes was found to be better by using hydrogen peroxide solution the same could be used for similar applications involving other processes.

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