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Production of the Microfiltration Membranes of Wide Range Porosity, High Mechanical, Thermal and Chemical Stability by „Green" Fabrication Method

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ABSTRACT

In the modern technological processes, the microfiltration membranes are used for removal of the suspended particles, colloids and microorganisms from the liquid solutions, as well as for use of the biologically active substances. Their demand at the world market rises day by day and if their production from the polymeric materials having wide range porosity, as well as high mechanical, thermal and chemical stability is provided the field of their use will expand even more. Proceeding from all above, fluoroplastic (F-4) was chosen, as the thermo- and chemically stable polymer used in medicine and food industry for production of the wide range porosity microfiltration membranes. The methods used for modification of membranes in the process of the research do not require any toxic solvents or complicated appliances or high power inputs. The thermo- and chemical stability of the produced membranes allows their multiple use in the process of filtration, which also allows implementing the principles of „green" technology. Pore sizes distribution of membranes was researched on Porometer by using the method of capillary flow porometry in compliance with the standards of ACTM F-316-03, which rules out using of the toxic (or hazardous substances), ex. mercury.

Keywords: fluoroplastic membrane; microfiltration; membrane fabrication**;** capillary flow porometry; pore sizes distribution.

INTRODUCTION

Nowadays, the membrane technologies occupy one of the important places among the world technologies and the sphere of their use expands day by day, which is caused with simplicity of instrumentation, low power intensity and high efficiency of technological processes. At the world market, microfiltration membranes are mostly used among the membranes employed in the membrane technologies, which is caused by the wide range of their use [Anis et al., 2019]. Demand of the microfiltration membranes will rise even more, if they are made of the ecological, thermo- and chemically stable polymers. Polymer polytetrafluoroethylene – fluoroplastic (mark F-4), the thermo-and chemically stable polymer used in medicine and food industry was chosen. Although it is expensive, its multiple use provides economy and ecological compatibility, which is important for filtration of liquid foodstuff [Nunes and Peinemann, 2006]. Their use will be especially important in pharmacy – for production of injection water [Mulder, 1996].

Mechanical, thermal and chemical stability is the extremely important index for operation of the membranes. Stability of membranes to components of mixtures under separation as well to the substances using for regeneration and conservation of membranes is the important feature. Corrosive media (acids, alkali, various oxidants and regenerants), as a rule, cause destruction of the macromolecule chain links, which causes change of the membrane qualities (decrease of mechanical stability and selectiveness). Mechanical, thermo- and chemical stability of membranes mainly depends on the chemical nature of polymers using for production of membranes [Catanese et al., 1999; Kang and Cao, 2014; Ohkubo, 2022].

The aim of the conducted work included extension of the range of pores of microfiltration membranes produced from fluoroplastic material, which can provide expansion or the field of their application, especially, filtration of liquid foodstuff. Production of the membranes with the wide range of the mean diameter pores, namely from 1000 to 10000 nm, was targeted. Production of membranes with the pores range under 2000 nm is important for water purification from microorganisms, i.e. it may allow production of injection water from distilled water, as a very important result for pharmacology and medicine. Apart from the above, production of the membranes with the wide range of the mean diameters pores will allow filtration of the various liquid foodstuff. Namely, synthesis of such varied membranes can allow their use for filtration of drinking water, wine, beer, soft and alcoholic beverages, cognac, mineral water, various water and alcoholic infusions, medicinal plants extracts [Otitoju et al., 2016; Kong et al., 2020; Mat Nawi et al., 2022].

Above all, the named polymer can allow multiple regenerations of the membranes foiled resulted filtration by any regenerants. Just this can allow their multiple use [Gotsiridze et al., 2009; Gotsiridze et al., 2017; Mkheidze et al., 2018].

RESEARCH OBJECTS AND METHODS

Materials

Fluoroplastic material produced from polytetrafluoroethylene polymer powder was used in the conducted research. As a result of its modification, the wide range porosity microfiltration membranes were produced. Fluoroplastic is known in the USA as Teflon, in England – as Fluon, in France – as Sareflon, in Italy – as Algoflon, in Germany – as Hostaflon, in Japan – as Polyflon, in Russia – as Ftuoroplast [Kislitcina et al., 2020]. Fluoroplastic is a high-molecular polymer and it has the unique properties:

- especially high chemical stability due to high shielding effect of negatively charged atoms of fluorine (it is stable even at boiling in aqua regia);
- stability to all mineral and organic acids and alkali, organic solvents, oxidants and other corrosive media;
- only alkaline metals alloys, elementary fluorine and trifluoro chlorine at high temperature can dissociate this polymer;
- it has a high ability not to become wet with water and not to succumb to the effects of water at the longest exposure;
- it is absolutely stable under tropical conditions, has antimycotic abilities;
- it is characterized by high thermostability (from – 269°C to +260°C).

Due to the physicochemical, mechanical and electric properties of fluoroplastic, it is used everywhere. The parts and products made of it are used in cosmos industry, medicine, shipbuilding, household goods, etc [Cui et al. 2014; Fomin et al. 2022]. Previously, a kind of microfiltration membrane having the mean diameter of pores 4000-45000 nm was produced by the authors from fluoroplastic material. Its initial rating was 500 $l/m^2 \cdot h$ at 1.5 atmospheric pressure [Gotsiridze et al., 2017; Mkheidze et al. 2018]. The resistance of the membrane made of fluoroplastic to microorganisms was studied and it was found that it is resistant to them [Mkheidze et al. 2019].

Methods of research of polymeric membranes

The main properties of polymeric membranes include porosity and pores dimensions. They are determined with the various parameters: general porosity, minimum, mean, maximum pore sizes dimensions and their distribution according to the dimensions. General porosity means total volume of pores in the material. Effective porosity means a part of the pores participating in transfer of liquid or gas through membrane. These pores are connected to each other and make the through canals. The other important parameters of the membranes include their superficial porosity, as just this parameter determines the membrane transmission ability in combination with the thickness of upper layer.

It is known that the microfiltration membranes have the pores of 0.1–1 um. Porosity of microfiltration membranes is determined in the Membrane Synthesis Laboratory of Agrarian and Technologies Research Institute, at porometer POROLUX™ 500 (Porometer NV) with so called capillary flow porometry method [ASTM F-316-03] is considered in our previous work [Mkheidze et al. 2020]. Also, the authors [Agarwal et al. 2012; Matveev et al. 2021; Tanis-Kanbur et al. 2021] applied the capillary porometry method for research of the polymeric membranes, nonwoven fabrics and the other porous materials. Porometer and wetting agent (porifil) were manufactured by POROMETER NV (Belgium). Concurrently, their porosity was determined with the traditional methods and calculated with the formula 1 [Mulder 1996]:

$$Porosity = (Ww\text{-}Wd)/(\rho w \times V) \quad (1)$$

where ρw – water density at room temperature (kg/m^3); V – membrane volume (m^3); W_w ,$W_{d\text{-}}$ – membrane mass in wetted and dry state, respectively.

Film thickness was measured with the digital micrometer. Membrane initial capacity was investigated in the pilot filtration plant. Indications were taken in 10 minutes from starting of filtration at 0.15 mPa. Membrane capacity is a quantitative property of membrane permeability. Capacity is a substance quantity passing through the membrane surface unit during the time unit. Membrane capacity Q was calculated with the formula 2 [Ripperger and Altmann, 2002].

$$Q = \frac{V}{S \times t} \quad (2)$$

where: Q – the *membrane* capacity (l/m^2h); V – is pure water volume (l); S – average space of *membrane* (m^2); t – is filtration time (h).

RESULTS AND DISCUSSION

Mechanic and thermal modification

The laboratorial microfiltration membranes were made from fluoroplastic material and their further modification was performed. Pore dimensions of the modified membranes produced with the various methods were measured by means of porometer (POROLUX-500). The results are shown in the Table N 1.

As shown in Table 1 outer surface of the membranes formed is an active layer and the inner pores are much larger, what means that the membranes formed are asymmetric, which are much better than symmetric ones, as selectiveness and capacity of asymmetric membranes are much higher. Besides, after mechanic and thermal modification of the formed membranes the pore dimensions became much smaller and above all, their asymmetricity did not upset, which allows continuing the research in this direction.

The curves received as a result of research of the initial and modified membranes with the porometer (please, find Curve 1 – dependence of flow on pressure, Fig. 1, and Curve 2 – dependence of flow on pores diameter, Fig. 2) show that the mechanical impact air flow decreases, also pore diameter reduces. Initial sample thickness – 0.960 mm, mechanically modified sample thickness 0.695 mm.

Modification with tea infusion

In the previous studies (Raul Gotsiridze, 1989) in the process of production of baromembranes with hydrophilic polymers the modification of the synthesized membranes with tea infusion was performed. The best results were received in the research of reverse osmosis membranes, namely, selectiveness of the produced reverse osmosis membranes to NaCl grew from 95% to 98.5%. The studies had not been performed for the membranes synthesized with the hydrophobic polymers; that is why, this time the membranes synthesized from hydrophobic membranes were used for research.

Table 1. Data of pore size of Fluoroplastic membranes after mechanical modification

	Fluoroplastic		Film thickness. mm	Pore size		
				Smallest pore size (um)	Main pore size (um)	Bubble point pore size (um)
1	Initial	upp side	0.96	0.351	2.38	6.05
		down side		0.602	2.67	7.78
2	Modified (mechanically processes)	upp side	0.65	0.268	0.69	0.79
		down side		0.458	0.7	0.947
3	Modified (mechanically/ thermally processes)	upp side	0.55	0.271	0.699	0.84
		down side		0.281	3.88	8.88

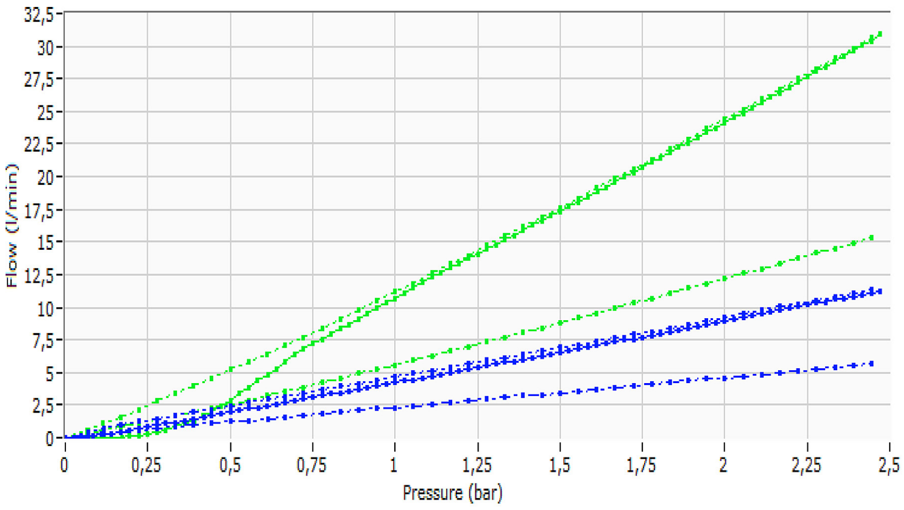

Fig. 1. Flow (ml/min) – pressure (Bar) curve. (Blue curve – virgin fluoroplastic; green curve – fluoroplastic after mechanical modification**)**

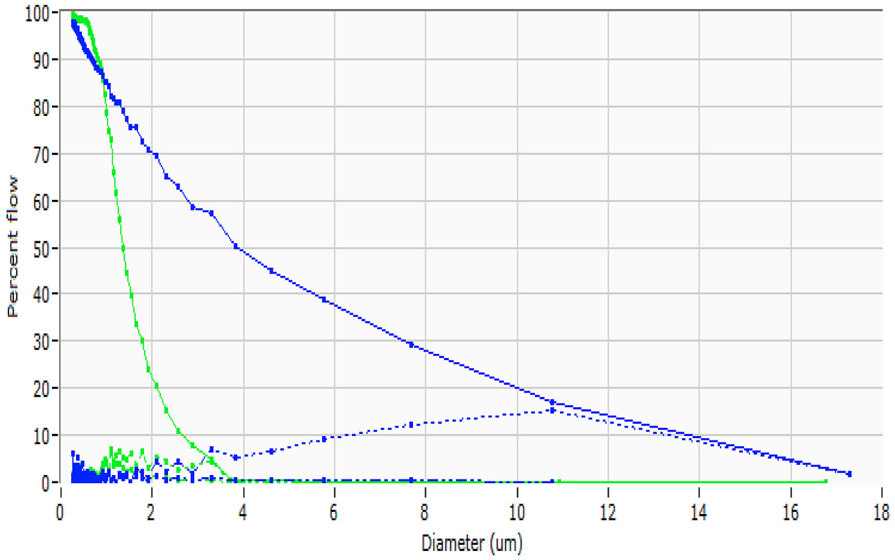

Fig. 2. Flow (%) – diameter (um) curve. (Blue curve – virgin fluoroplastic; green curve – fluoroplastic after mechanical modification)

For the purpose of the experiment, 6 kinds of tea infusion were produced (black tea, green tea, yellow tea, yellow blueberry tea, red blueberry tea and red tea). In each infusion 2 samples were placed. After 24 hours, one sample was remained unchanged, the second one was exposed to mechanical modification. Each sample pores dimensions were measured with the porometer. Porometer detects three indices of pore dimensions for each sample: minimum, mean and maximum ones. Also, the thickness of each sample was determined by means of micrometer. The results are shown in Table 2. As it is shown in Table 2, the results of keeping of the microfiltration membranes formed from fluoroplastic material in the various kinds of tea infusion prove that is it possible to raise the selectiveness of the membranes produced from the formed hydrophobic polymeric material without their mechanical modification, which – concurrently with improvement of selectiveness – will provide growth of capacity (see Fig. 3**)**.

The figures and diagrams show the results of changes in the dimensions of the pores of the virgin and mechanically modified fluoroplastic samples kept in the various kinds of tea infusions. Change of the membrane pores dimensions (minimum, mean, maximum pore sizes) after keeping in the green tea (green column) and after pressing

Table 2. Data of pore size of Fluoroplastic membranes after modification with tea infusion

	The sample formed from fluoroplastic polymer	Film thickness mm	Pore size		
			Smallest pore size (um)	Main pore size (um)	Bubble point pore size (um)
1	Sample 1 – kept in the black tea infusion	0.960	0.272	1.35	1.23
2	Sample 1 – kept in the black tea infusion (after mechanical modification)	0.695	0.278	0.869	1.38
3	Sample 2 – kept in the green tea infusion	0.96	0.328	1.98	4.36
4	Sample 2 – kept in the green tea infusion (after mechanical modification)	0.608	0.678	0.693	0.63
5	Sample 3 – kept in the yellow tea infusion	0.96	0.366	1.49	3.49
6	Sample 3 – kept in the yellow tea infusion (after mechanical modification)	0.637	0.268	0.935	1.30
7	Sample 4 – kept in the yellow blueberry infusion	0.96	0.334	2.18	5.12
8	Sample 4 – kept in the yellow blueberry infusion (after mechanical modification)	0.665	0.307	0.79	1.50
9	Sample 5 – kept in the red blueberry infusion	0.96	0.542	2.26	5.42
10	Sample 5 – kept in the red blueberry infusion (after mechanical modification)	0.664	0.308	0.769	1.37
11	Sample 6 – kept in the red tea infusion	0.96	0.578	4.94	13.7

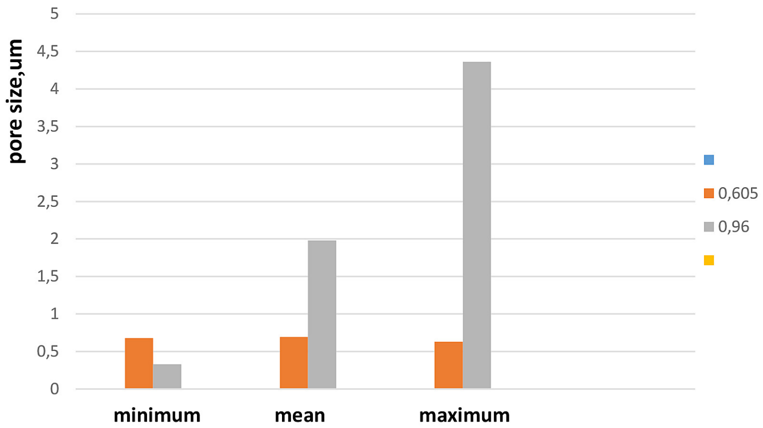

Fig. 3. Samples pore size after mechanical modification and after modification with green tea infusion

(brown column). Virgin sample thickness – 0,960 mm, mechanically modified sample thickness 0.695 mm. On the ground of the research results, black tea infusion was selected and further studies were performed with it only.

Modification with exposure to temperature

For the purpose of the experiment the black tea 5% and 10% solutions were taken. Two samples were placed in each solution of the various temperature (40, 60, 80, 100°C). After 5 hours, one sample remained unchanged, but the second sample was mechanically modified. The pores of each sample were measured by means of a porometer. The results are given in Table 3. On the ground of the research results,10% infusion of black tea was selected. The curves (Fig. 4–6) of pore size flow distribution received as a result of research of the membranes with porometer show that under impact of temperature the membrane pores have a wide spread, which affects the membrane selectiveness.

After keeping in 10% infusion of black tea for the various periods (15, 30, 60 and 180 minutes) the membrane pores were measured with a porometer (2 initial samples and 2 mechanically modified ones for each period). The results are given in Table 4 and in Figure 7.

Aiming to increase the range of the membrane porosity, a part of the samples was exposed to boiling water for 10 min.

The thickness of the samples exposed to boiling water reduced to the concrete size under the mechanic impact. The pores were measured with

Table 3. Data of pore size of Fluoroplastic membranes after termal modification(with the various temperature)

N	Membrane simples	Film thickness mm	Pore size			Remark
			Smallest pore size (um)	Main pore size (um)	Bubble point pore size (um)	
1	Sample 1 Sample 1 Mechanically modified	0.97 0.658	0.505 0.312	2.24 0.933	5.48 2.28	5%. 40 °C
	Sample 2 Sample 2 Mechanically modified	0.975 0.621	0.499 0.686	3.01 0.699	9.13 0.885	10%. 40 °C
2	Sample 3 Sample 3 Mechanically modified	0.974 0.646	0.319 0.442	2.89 1.01	8.95 1.83	5%. 60 °C
	Sample 4 Sample 4 Mechanically modified	0.964 0.613	0.394 0.7412	2.123 0.7826	5.477 1.246	10%. 60 °C
3	Sample 5 Sample 5.Mechanically modified	0.969 0.654	0.3345 0.308	2.34 0.986	5.48 2.49	5%. 80 °C
	Sample 6 Sample 6. Mechanically modified	0.967 0.646	0.356 0.308	2.18 0.869	6.85 2.286	10%. 80 °C
4	Sample 7 Sample 7. Mechanically modified	0.96 0.662	0.621 0.315	2. 24 1.2	4.57 2.28	5%. 100 °C
	Sample 8 Sample 8 Mechanically modified	0.96 0.648	0.315 3.66	2.53 1.64	8.11 4.57	10%. 100 °C

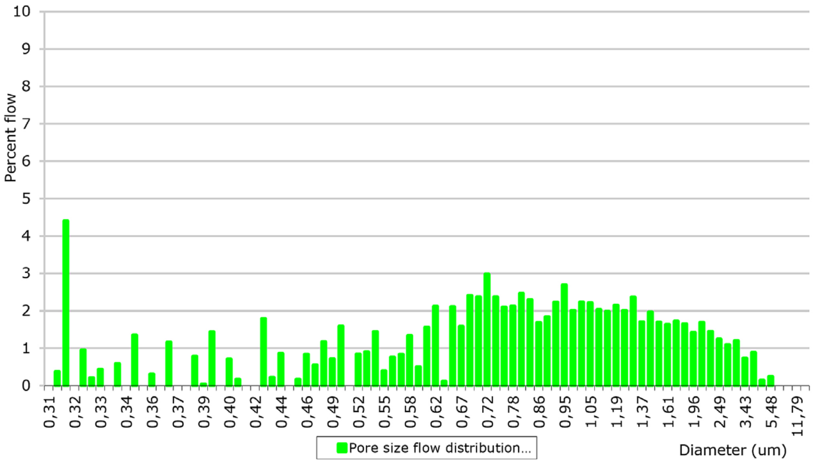

Fig. 4. Percent flow – diameter (um) curve (simple -5% black tea, 40℃)

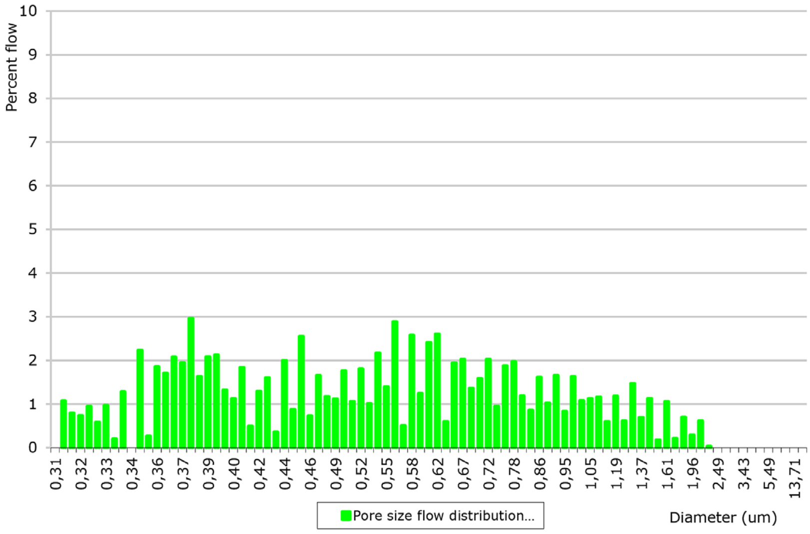

Fig. 5. Percent flow – diameter (um) curve (simple -5% black tea, mechanically modified, 40℃)

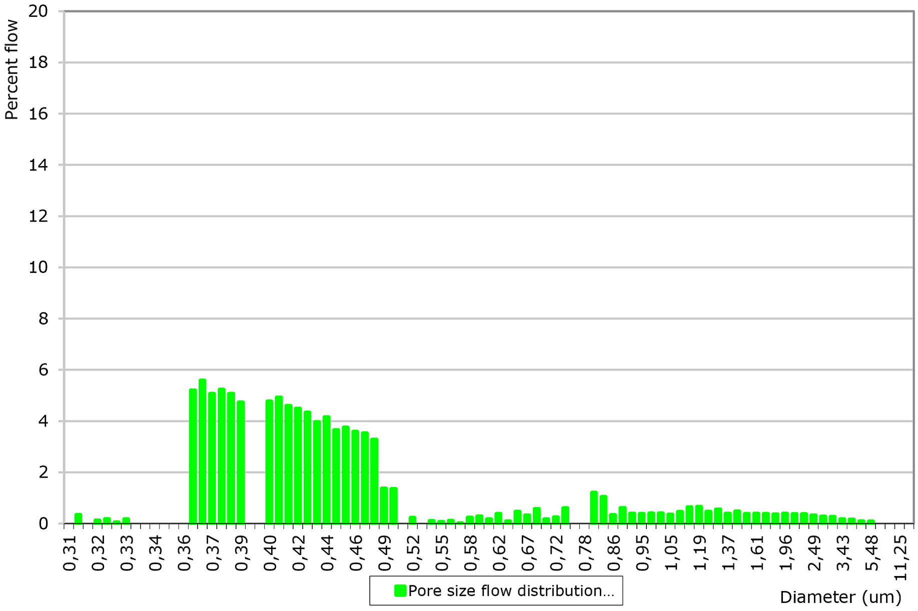

Fig. 6. Percent flow – diameter (um) curve (Simple -10% black tea, 40℃)

Table 4. Data of pore size of Fluoroplastic membranes after exposed to infusion black tea for the various periods

N	Period of keeping of membranes, minutes	Sample	Film thickness, mm	Pore size		
				Smallest pore size (um)	Main pore size (um)	Bubble point pore size (um)
1	15 minute	Initial sample	0.96	0.419	2.25	8.52
2	15 minute	Mechanically modified	0.681	0.343	1.03	2.4
3	30 minute	Initial sample	0.962	0.873	0.896	1.2
4	30 minute	Mechanically modified	0.589	0.356	2.05	4.79
5	60 minute	Initial sample	0.963	0.343	2.42	4.79
6	60 minute	Mechanically modified	0.646	0.8	0.846	1.07
7	180 minute	Initial sample	0.963	0.436	2.25	4.79
8	180 minute	Mechanically modified	0.602	0.98	1.0	1.37

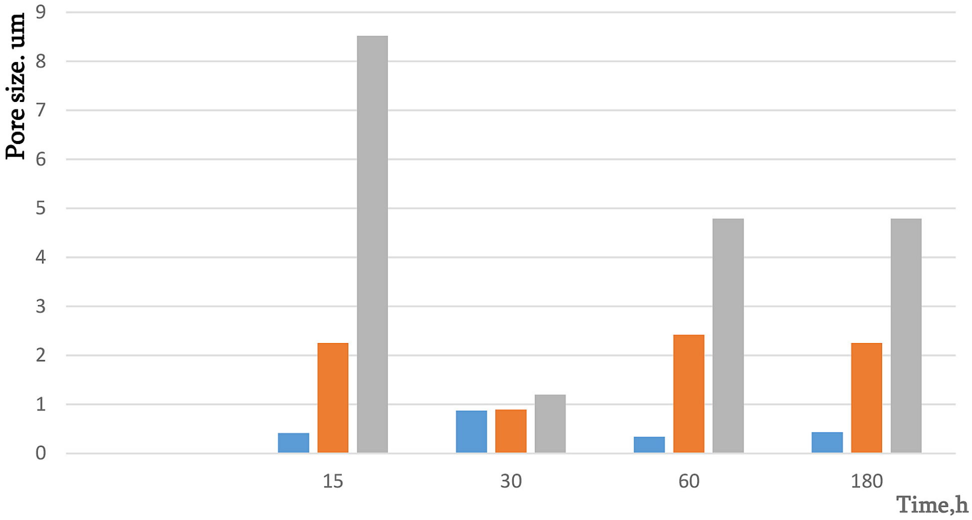

Fig.7. Dependence of pores dimensions on keeping time (initial membrane)

Table 5. Data of pore size of Fluoroplastic membranes after exposed to boiling water

N	Film thickness mm	Pore size					
		Smallest pore size (um)		Main pore size (um)		Bubble point pore size (um)	
		cold	hot	cold	hot	cold	hot
1.	0.96 mm (initial)	0.481	0.604	2.29	2.39	8.65	8.41
2.	0.65 mm	0.356	0.356	0.924	1.11	1.92	2.4
3.	0.50 mm	0.356	0.356	0.94	0.84	1.92	1.92
4.	0.45 mm	0.365	0.96	0.743	1.05	1.2	1.37
5	0.40 mm	0.356	0.36	1.05	0.821	2.4	1.6

Table 6. Data of pore size of Fluoroplastic membranes after exposed to infusion of black tea for the various temperature

	Film thickness, mm	Pore size					
		Smallest pore size (um)		Main pore size (um)		Bubble point pore size (um)	
		cold	hot	cold	hot	cold	hot
1	0.96 mm (initial)	0.695	0.356	2.06	2.38	4.77	8.37
2	0.65 mm	0.343	0.356	0.977	1.05	2.4	2.4
3	0.50 mm	0.96	0.343	1.01	1.35	1.6	3.2
4	0.45 mm	0.356	0.416	1.06	2.85	1.92	8.34
5	0.40 mm	0.356	0.457	1.71	1.85	4.8	4.79

Table 7. Thickness and density of membrane samples

	Film thickness, mm	Simple density, kg/m^3	
		Initial simple	Simple after 10% black tea infusion
1	0.96 mm	1528.26	1535.47
2	0.65 mm	1863.0	1811.52
3	0.50 mm	1976.56	1847.75
4	0.45 mm	2007.0	2024.4
5	0.40 mm	2389.19	2209.4

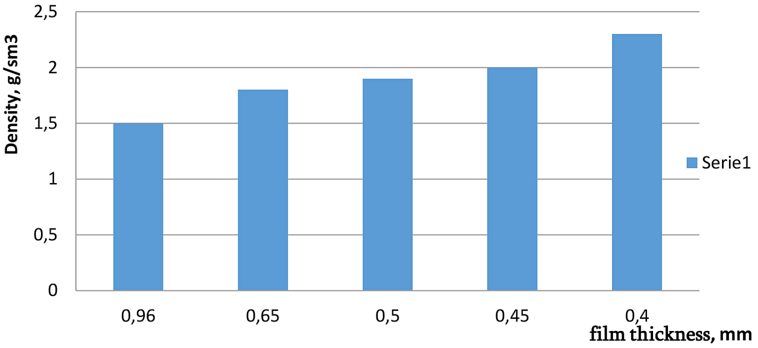

Fig. 8. Dependence of membrane density (g/sm^3) – membrane thickness, mm

a porometer. The results are given in Table 5. Part of the membrane samples were placed in the cold 10% black tea infusion and another part – in the boiling 10% black tea infusion for 10 minutes. The thickness of the initial samples and those exposed to the boiling infusion reduced to the concrete size under the mechanic impact. The pore dimensions were measured with porometer. The results are given in Table 6. Density was measured for the membranes of the various thickness samples, initial ones and those exposed to 10% black tea infusion are given in Table 7. Figure 8 show that density grows concurrently with change of the membrane thickness.

The results prove production of the microfiltration membranes of the wide range porosity (1000–10000 nm). The research of possibility of their use for separation, concentration, disinfection of the various liquid mixtures and extraction of bioactive compounds will continue.

CONCLUSIONS

The wide range porosity microfiltration membranes (1000–10000 nm) are produced under laboratory conditions, in various technological modes. The membrane pore dimensions (minimum, mean, maximum) are measured by means of the capillary flow porometry method (CFP) with POROLUX™ 500 (Porometer NV). Five microfiltration plants will be produced with the received microfiltration membranes. Application of the fluoroplastic membranes having high thermo- and chemical stability allows using such plants for: production of injection water; filtration of alcoholic and soft beverages; filtration of drink water, filtration of juices and vegetative infusions of various vegetables and fruits. The filters will be used multiple times, as the special regenerants will be matched for regeneration of the membranes.

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