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Some issues in membrane pore size research

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Abstract

The paper presents a comparative analysis of the pore sizes membranes obtained by the bubble point method and scanning probe microscopy. The dependence of the membranes filtration characteristics on its pores size has been revealed. It has been established that the membrane pore sizes by the bubble point method, somewhat correspond to the pore sizes obtained by scanning probe microscopy.

Keywords: Microfiltration, Membrane, Pore size, Specific efficiency

The basic filtration characteristics of micro- and ultrafiltration membranes can be assessed by pore size, porosity, and specific efficiency of the membranes. Membrane pore size can be studied by direct methods such as electron microscopy, scanning probe microscopy, and by indirect methods such as the bubble point method [1-2].

The aim of the work was to determine the size of membrane pores the bubble point method and to perform a comparative analysis of the pore sizes obtained during the study with a scanning probe microscope.

The determination of membrane pore size was performed on a laboratory device MTSI-BP-3 developed and created at the Membrane Technology Engineering Institute, which provides the study of disc-type micro- and ultrafiltration membranes of four different sizes (Diameters 10, 16, 26, 34 mm). Its main parts were manufactured using a ULTIMAKER 2 3D printer and calibrated in accordance with five foreign-made membrane standards, whose pore sizes and corresponding pressure ratings are shown in Table 1.

Table 1. Pressure at the moment of first bubble formation and membrane pore size

Membrane	Pressure at the moment of bubble formation, P, bar	Membrane pore size, μm
M1	0.06	5.00
M2	0.25	1.20
M3	0.65	0.45
M4	1.48	0.20
M5	2.95	0.10

The working principle of the device is based on the method of creating a bubble point based on the capillary effect, according to which the pores of the membrane are equivalent to capillaries and the liquid is retained in the pores by capillary forces. The pore size of the membrane is calculated by the formula:

$$D_{max} = 0.81/P$$

where D_{max} –The diameter of the membrane pore (μm) which has the shape of a capillary;

P –Pressure (bar) at which the first bubble forms.

Deionized water was chosen as the standard solution, and air was chosen as the gas [3-7].

The study of the membrane surface structure was carried out on a CERTUS STANDARD V scanning probe microscope, the 2D and 3D images of which visually show the structure and dimensions of the membrane pores. Scanning was performed in non-contact mode, with an NSG20 type cantilever (probe radius of curvature is 10 nm) [8-10]. Turbidity of natural water before and after filtration was determined on a turbidimeter TURB 555, in formazin units. [11-13]. The specific productivity of the membrane obtained by phase inversion was determined using the MTSI-JM-5 laboratory device [14-17]. The results obtained from the experimental study are presented in Table 2.

Table 2. Pressure at the moment of first bubble formation, membrane pore size, water turbidity, and membrane specific productivity

Membrane	Pressure at the moment of bubble formation, P, bar	Membrane pore size, μm	Turbidity of natural water, FTU		Specific productivity of the membrane J, l/m²h
			Before filtering	After filtration	
M1	3.0	4.500	1.90	0.03	5200
M2	3.5	3.850	1.90	0.03	4000
M3	4.0	3.370	1.90	0.03	3400
M4	4.5	3.000	1.90	0.03	3100
M5	5.0	2.700	1.03	0.03	2800
M6	5.5	2.450	1.91	0.03	2710
M7	6.0	2.250	1.87	0.03	2530
M8	6.5	2.070	1.89	0.02	2370
M9	7.0	1.920	1.90	0.02	2215
M10	7.5	1.800	1.89	0.02	2065
M11	8.0	1.680	1.90	0.01	1920
M12	8.5	1.580	1.91	0.01	1780
M13	9.0	1.500	1.90	0.01	1645
M14	9.5	1.420	1.90	0.01	1515
M15	10.0	1.350	1.90	0.01	1390
M16	10.5	1.280	1.90	0.01	1270
M17	11.0	1.220	1.90	0.01	1155
M18	11.5	1.170	1.90	0.01	1045
M19	12.0	1.120	1.90	0.01	940
M20	12.5	1.080	1.90	0.01	840

M21	13.0	1.030	1.90	0.01	745
M22	13.5	1.000	1.90	0.01	720
M23	14.0	0.960	1.90	0.01	700
M24	14.5	0.930	1.90	0.01	680
M25	15.0	0.900	1.90	0.01	660
M26	15.5	0.870	1.90	0.01	645
M27	16.0	0.840	1.90	0.01	630
M28	16.5	0.820	1.90	0.01	620
M29	17.0	0.790	1.90	0.01	610
M30	35.5	0.380	1.90	0.01	490
M31	36.0	0.375	1.90	0.01	480
M32	36.5	0.369	1.90	0.01	475
M33	37.0	0.364	1.90	0.01	470
M34	37.5	0.360	1.90	0.01	465
M35	38.0	0.355	1.90	0.01	460
M36	38.5	0.350	1.90	0.01	450
M37	39.0	0.346	1.90	0.01	445
M38	39.5	0.341	1.90	0.01	435
M39	40.0	0.337	1.90	0.01	430
M40	41.5	0.324	1.90	0.01	420
M41	42.0	0.320	1.90	0.01	415
M42	42.5	0.316	1.90	0.01	410
M43	43.0	0.312	1.90	0.01	405
M44	43.5	0.310	1.90	0.01	400

The dependence of the specific productivity of the membrane on its pore size was revealed in the study using the bubble point formation method. From the data presented in Table 2, it can be seen that the M1 membrane has the maximum specific productivity membrane (5200 l/m^2h) and the largest pore size (4.500 μm), for which the lowest indicator minimum pressure for bubble formation is 3.0 bar.

The results obtained from the scanning probe microscopy study are presented in Table 3 and Figure 3.a.,b.,c. The results show that the geometric parameters (average length, width, and depth of the 2D image of the membrane) of the smallest membrane pore size were determined.

Table 3. Geometric parameters (the average length, width and depth of the 2D image) of the smallest membrane pore, μm

Membrane	Geometric parameters (the average length, width and depth of the 2D image) of the smallest membrane pore, μm		
	length	width	depth
M1	4.4384	4.7937	0.036628
M28	0.82777	0.24944	0.017666
M43	0.32818	0.27646	0.10132

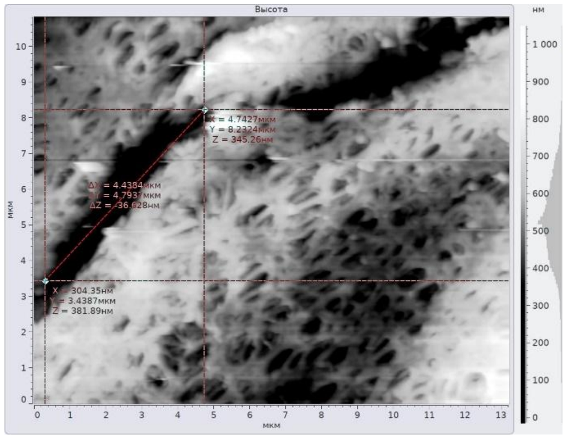

Figure 3. a. M1 2D Photomicrograph of the membrane surface

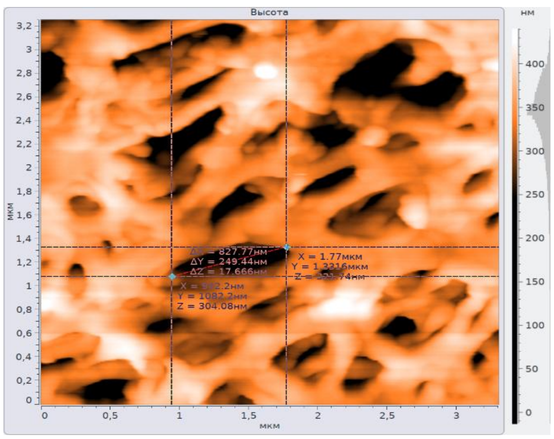

Figure 3. b. M8 Photomicrograph of the membrane surface

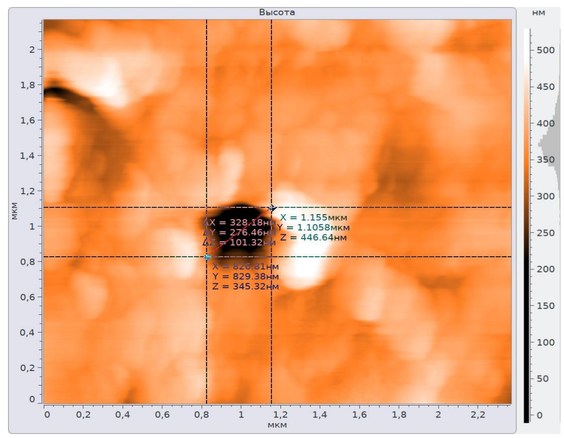

Figure 3. c. M12 Photomicrograph of the membrane surface

As a result of a comparative analysis of the filtration characteristics of membranes obtained from various polymers, it was established that the membrane pore size determined by the bubble point generation method is in some agreement with the pore size values obtained by scanning probe microscopy. Therefore, based on the filtration characteristics and the results of the analysis of the filtered water, the membranes manufactured at the institute can be used in water microfiltration.

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