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Study of the Effect of Lithium Chloride on Polymer Solubility by Optical Microscopy and Photon-Correlative Spectroscopy

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Abstract – : In the work the research of the process of drying of the polymer and low-molecular electrolytic additive – lithium chloride for obtaining of membrane film on the basis of Poly [N, N′–(1.3-phenylene) isophthalamide] is considered. The study of the process of solving of polymeric composition was carried out in N, N′-dimethilformamide at various temperatures by the investigation of the effect of dried lithium chloride and polymer on solubility degree for polymeric composition. Monitoring of solubility degree for polymeric composition was carried out by the methods of optical microscopy and photon-correlative microscopy.

In response to the research it was revealed that in the polymeric composition, obtained by the components dried to 105^0C, after monitoring by optical polarization – interference microscope and analyses for determining of the nanoparticles, the particles with the size more than 10000nm in polymeric mass are observed. Along with it, the particles of 10nm-30nm size were found in polymeric mass after monitoring by the analyses for determination of nanoparticles and by optical polarization – interference microscope. Qualitative decomposition of the particles in polymeric composition allows the regulation of the surface, required for formation of membrane pores and the prediction of specific productivity of membrane film.

As a result of the research in has been established that, at the temperatures in the range from 105^0C to 135^0C, the components of dried polymer and lithium chloride determine an obtaining of the particles of multiply different size in polymeric mass by maximum value of solving – 10nm.

Monitoring of the particles in solving process was carried out at polarization – interference optical microscope and the sizes of polymer particles and were established at the analyses for determination of the nanoparticles.

Keywords – Polymer, lyotropic additive, Poly [N, N′–(1.3-phenylene) isophthalamide], N, N′-dimethilformamide, lithium chloride.

I. INTRODUCTION

By the data of World health defense organization, 80% of the diseases are caused by the deficit of pure fresh water and qualitative products. Therefore, the demand is increased for the membranes for baromembrane processes, which provide the sterile-finishing filtration of fresh water and wine implying purification – sterilization of the liquids, regulation of rigidity, chemical elements removal of bacteria and yeasts by 100%, conditioning to permissible norm and impartment of desirable organic-leptic properties. For optimization of sterile-finishing filtration of fresh water and wine the creation of the membranes is topical with the properties which will be different by corresponding sizes of the pores, by high porosity per unit area, by low roughness of surface relief and by high specific productivity. Their use will be favorable for simplification of the process of sterile-finishing filtration of the product, providing the significant saving power inputs [1-6].

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The problems of preliminary treatment of the components of polymeric composition and of their further, solving were considered in the works of various authors, such as:

The paper of Ali Reza Kamali, Derek J. Fray, Carsten Schwandt, in which the ionic compounds of lithium chloride are characterized. The processes of absorbion of the water from the atmosphere by waterless salt lithium chloride was studied thermokinetically [7].

S.Joshi, A.Rao.– Synthesis of poly [N, N′–(1.3-phenylene) isophthalamide] and estimation of reverse osmosis membrane prepared from it. In the paper the preparation of poly [N, N′–(1.3-phenylene) isophthalamide], its physical-chemical properties and estimation of reverse osmosis, produced on the basis of poly [N, N′–(1.3-phenylene) isophthalamide], are presented [8].

The paper of M.Chen, Ch.Xiao, Ch.Wang, in which the preparation of poly-reinforced membranes and effect of polymer concentration on membrane operational characteristics are described. The properties of the membranes are estimated in accordance with morphology and penetrability [9].

In the paper of K.Kulikov, T.Koshlan the determination of the size of colloidal particles by the method of the light dynamical scattering is considered. The functions of distribution of particles sizes are described and the agreement between theoretical and experimental analyses are noted [10].

Polysulfones, polyamides, polycarbonates and cellulose esters are among polymeric membrane materials. The main characteristics, which distinguish the synthetic polymers from other chemical compounds, are there mechanical, thermal and chemical properties.

From the wide choice of synthetic polymers the poly [N, N′–(1.3-phenylene) isophthalamide] is the best material for fabrication of micro-, ultra- and nanofiltrational membranes. Apart from the membrane production, poly [N, N′–(1,3-phenylene) isophthalamide] is used for fabrication of thermal-and fire-resistant clothes, for filtration of the gases and for thermal isolation.

Poly [N, N′–(1,3-phenylene) isophthalamide] is characterized by good transport properties, by high temperature of vitrification, by rigid structure and hydrophilic nature. Non-amide and hydrogen bonds determine the high thermal-and chemical stability of the mentioned polymers a which allows the use of the membranes, prepared from it, in the wide range of temperatures and in aggressive areas.

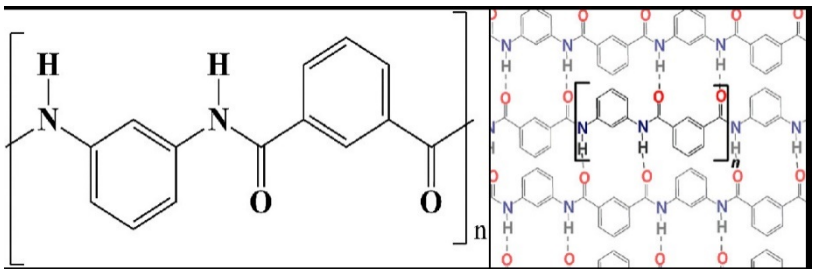


Fig. 1. Chemical structure Poly [N, N′-(1.3-phenylene isophthalamide]

Poly [N, N′– (1,3-phenylene) isophthalamide] is characterized by low solubility. To increase it solubility the chlorides of magnium, calcium and lithium are used together with the polar solvent [11-12].

Within recent years, for the purpose of more and more improvement of physical-chemical characteristics of the polymer, the investigation were carried out in the direction of the use of mineral salts in polymeric solutions. Monitoring of the solving process of the polymer and lyotropic additives in polymeric composition was carried out by various methods, such as optical microscopy and photon-correlative spectroscopy.

Research goal involved:

- Research of the process of drying of the polymer and low-molecular electrolytic additive – lithium chloride for preparation of membrane film on the basis of poly [N, N′–(1.3-phenylene) isophthalamide];
- Study of the effect of dried polymer and liotropic additive – lithium chloride at various temperatures in N, N′-dimethilformamide;
- Monitoring of solubility degree for polymeric composition by the methods of optical microscopy and photon-correlative spectroscopy;
- Regulation of particles sizes in polymeric compositions and determination of the degree of polydispersity.

The method of drying of poly [N, N′– (1.3-phenylene) isophthalamide] and lithium chloride is used as well as the method of the light dynamical scattering for determination of particles sizes in polymeric compositions (Dynamic light scattering – DSL) [13-15]. Drying process was carried out in the thermostat POL-EKO, model ST. Observation of the particles, solved in polymeric compositions, was carried out at polarization-interference optical microscope – Biolar, by 350-400 fold magnification of the objective by digital chamber of 10.7 MP clearness.

The sizes of polymer particles and polydispersity index were determined at the analyzer of the nanoparticles - Zetasizer Nano Zen 3690 – Malvern Instruments (England).

The morphology of membrane surface was studied at scanning probe microscope – SPM, Certus Standard V. Nano Can Technologies Ltd (Russia).

The productivity of membrane samples was established at laboratory device-plant, created in the Engineering Institute of Membrane Technologies of Georgian Technical University. The plant involves the laboratory membrane apparatus, manufactured by means of 3D printer.

Among synthetic polymeric membranes the micro-, ultra- and nanofiltration ones, prepared on the basis of aromatic polyamides, occupy a highly important place. They are used successfully in the filtration plants of water and wine. Membrane specificity implies the dependence of pores sizes, porosity per unit area and of the roughness of surface relief on the constitution of polymeric compositions as well as on the regime parameters of phase inversion and on the variation of chemical-composition of coagulation-liquid.

For optimization of the sterile-finishing filtration of fresh water and wine the obtaining of the new membranes required the researches in various direction**.** Before beginning of the researches the information was searched from the scientific literature about membrane polymeric materials, on the improvement of membranes filtration al characteristics and on their morphology. From this viewpoint the complex investigations of polymer, solvent and additives were of interest.

Drying of the components of polymeric compositions was carried at various temperatures. Effect of lyotropic additive – lithium chloride and polymer, dried at various temperatures, in N, N′-dimethilformamide was studied. Monitoring of solubility degree for polymeric composition was realized by the methods of optical microscopy and photon-correlative spectroscopy.

The sizes of the particles and polydispersity index were determined and the limiting values the sizes of decomposed particles were adjusted in the polymeric mass.

The condition were determined empirically which provide the decomposition of the particles in the polymer mass and the revelation of limiting indices of the sizes.

The qualitative decomposition of the particles in the polymeric composition allowed us the regulation of required area for pore formation in the membrane and the prediction of specific productivity of membrane film.

II. Experimental

2.1. Materials

Poly [N, N′–(1.3-phenylene) isophthalamide] was delivered by the Company DuPont (USA). Its chemical structure is presented in Fig 1. LiCl was delivered by Company DuPont (USA). N, N′-dimethilformamide – DMFA>99% was delivered by BASF (Germany). Distilled water was used as non-solvent in coagulation bath, electric conductivity of which comprised $5.2x10^{-4}$cm/m

2.2. Drying of polymer and lithium chloride

Drying of poly [N, N′–(1.3-phenylene) isophthalamide] was carried out in the thermostats at the temperatures of 60^0C, 75^0C, 90^0C, 105^0C, 120^0C and 135^0C over the interval of three hours. Drying of crystalline hydrate, was carries out at the temperatures of 60^0C, 75^0C, 90^0C, 105^0C, 120^0C and 135^0C over the interval of three hours till obtaining of totally waterless crystals of LiCl.

2.3. Preparation of membrane dispensing solution

For obtaining of membrane preparing composition Poly [N, N′–(1.3-phenylene) isophthalamide] - 10 (mas%), LiCl - 5 (mas%), DMFA – 85 (mas%) are placed in 100ml volume reaction vessel and is gradually mixed by magnetic mixed at the temperature of 55°C.

2.4. Monitoring of polymer solving in membrane preparing composition

Monitoring of the process of polymer solving was carried out by polarization – interference optical microscope – Biolar (Poland) with the range of magnification: 350-400 and by digital chamber, mounted on it, with clearness 10.7 MP, which magnifies the image by a factor of 1.5-3.0.

2.5. Determination of the parameters of the solving of polymeric composition

Monitoring of the process of polymer solving was carried out by photon-correlative spectroscopy. Determination of the

parameters of polymer solving in membrane preparing composition, particles size, concentration and polydispersity index (PDI) was performed by means of the device (Zetasizer Nano Zen 3690 – Malvern Instruments, England). For increase of particles stability the solution was treated at ultrasonic apparatus in water bath - Unitra-Unima, UM-4, Olsztyn(Poland), 50Hz, over on hour. Particles size, concentration and polydispersity index was determined at the temperatures of 60^0C, 75^0C, 90^0C, 105^0C, 120^0C and 135^0C in the solution, containing dried polymer and lithium chloride, diluted to 5g/l.

2.6. Manufacturing of membrane planar test samples obtained

Obtained dispensed composition of homogenous, transparent membrane was hold at 25^0C for removal of the air bubbles. Removal of suspended particles was performed in centrifugal apparatus – CENTRIFUGE MPW-210, MPW, Med Instruments (Poland). The solution is dispensed on the glassy polished plate (76mmx26mmx1mm), mounted on the filler as the layer of 0.1-0.2mm thickness, the leveling of which is carried out by the knife. (Fig.2)

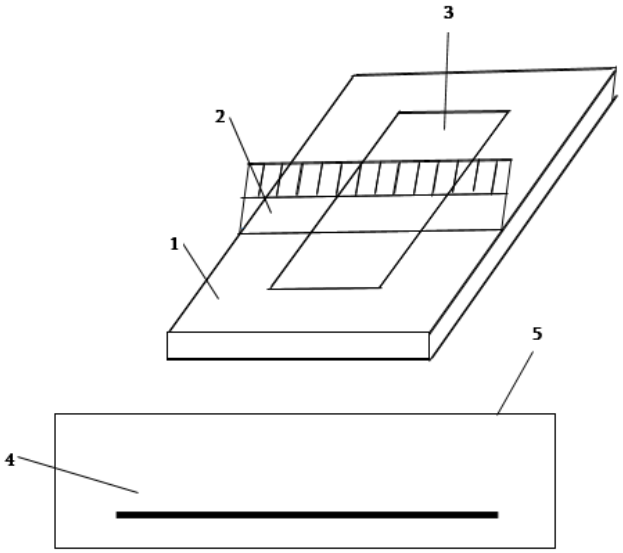


Fig.2. Principal scheme of membrane preparatory filler

1 –polymeric composition, 2 –the stainless steel knife, 3-4 –coagulation bath, 5 –coagulant (non-solvent)

The filler is placed in coagulation basin of the non-solvent at the temperature of 55^0C. Solid films (according to the temperatures of drying of polymer and lithium chloride, M60, M75, M90, M105, M120, M135 respectively) are prepared after coagulation. Obtained membranes are washed by the water at 60^0C, hold in washing bath for removal of water soluble compounds. Monitoring of membrane washing was carried out in the washing bath by measuring of the amount of Li^+ and Cl^- ions, passed from the membrane film into the water and pH-by means of ionometer (*И*-160, Belarus). Monitoring was continued as long as the neutral reaction of the water was fixed.

2.7 Characterization of membrane structure and morphology

Morphology and structure of the membrane were studied by scanning probe microscope – SPM, Certus Standard V, Nano Can technologies LTD, Russia). Relief of membrane surface, hillocks and cavities, pore amount per unit area and pore sizes were determined by cantilever of NSG20 type with large radius: 10nm, 5nm and 1nm. To study the membrane relief and topography the scanning and comprised 20-20mμ, for pore sizes – 1mμ -1mμ and for porosity – 5mμ -5mμ. Universal NSpec software provides the treatment of the obtained data and performance of visualization in the form of 2D and 3D images.

2.8. Membrane specific productivity

Experiments on determination of specific productivity were carried out at laboratory pant MTSI-JM-5, manufactured in experimental design office of the Institute.

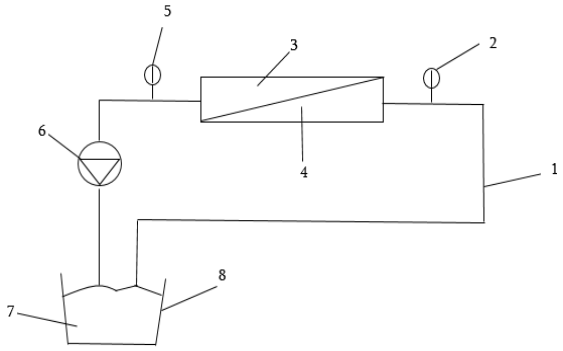


Fig.3. Principal scheme of the device MTSI-JM-5 for determination productivity of the membrane

1 –Circulatory contour, 2 –Manometer, 3 –Membrane pressure cell, 4 –Filtrate, 5 –Manometer, 6 –Pump, 7 –Processing fluid, 8 – Water tank

Plastic details of the plant (membrane cells) were manufactured by the program AutoCad-2019 at 3D printer (ULTIMAKER -2 Germany). Connecting details were prepared from the stainless steel of 316 class. Specific productivity of the membrane test samples were calculated by formula:

$$J = \frac{V}{St} \qquad (1)$$

where V – filtration volume, l;

S – membrane area. Membrane efficient area comprised 0.00270m^2;

t – filtration time, hours.

Filtrate purity was checked at the turbidity measurer - Turb555 IR (Germany).

III. Results And Discussion

3.1. Characterization of polymeric composition (Characterization of the parameters of polymeric composition).

At drying of Poly [N, N′–(1.3-phenylene) isophthalamide] at the temperatures of 60^{0}C, 75^{0}C, 90^{0}C and 105^{0}C the tendency of the weight decrease was revealed and at 120^{0}C and 135^{0}C the reduction of polymer weight wasn′t observed (Table 1). Increase of water molecules amount in polymer structure reduces the number of intermolecular hydrogen bonds of the polymer as well as common stability between the polymer and water molecules which causes the decomposition of polymer structure [16-19]. In Table 1 the temperature ranges of polymers drying, equal to 3hours, is presented.

Table 1. Weight of Poly [N, N′– (1.3-phenylene) isophthalamide] by temperature

Name of the substance	Drying temperature, t^{0}C	Sample weight, g		
		Before drying	After drying	After Weight loss
Poly [N, N′-(1.3-phenylene) isophtalamide]	60	0,550	0,517	0,035
	75	0,542	0,511	0,031
	90	0,511	0,508	0,003
	105	0,508	0,505	0,003
	120	0,505	0,505	0
	135	0,505	0,505	0

In the hydroscopic crystalline hydrate - LiClx3H_2O the amount of water molecules depends on crystallization temperature (Table 2) [20-22].

Number of water molecules - Name of the substance

Table 2. Dependence of the amount of water molecules in crystalline hydrate LiClx3H2O of temperature

Temperature, t^0C	-15	-15-21,5	21,5-90	90
Number of water molecules	3	2	1	0

At heating of the crystalline hydrate $LiClx3H_2O$ at the temperatures of 60^0C, 75^0C, 90^0C, 105^0C prepared by well-known preparative method, the weight reduction was fixed, according to which the most part of the water was separated from the salt in the temperature interval: 60^0-90^0C and negligible amount – in temperature range from 90^0C to 105^0C. Preparation of totally waterless crystals of LiCl was possible at 105^0-135^0C at the expense of the removal of hydration free water (Table 3).

Table 3. Weight of lithium chloride in accordance with drying temperature

Name of the substance	Name of the substance	Sample weight, g		
		Before drying	After drying	After Weight loss
Lithium chloride	60	0,157	0,145	0,012
	75	0,141	0,128	0,013
	90	0,128	0,124	0,004
	105	0,124	0,121	0,003
	120	0,121	0,121	0
	135	0,121	0,121	0

In the process of preparation of polymeric composition for membrane dispersion at the temperature of 55^0C the monitoring of solving was carried out by polarization-interference optical microscopy. In the Picture 3a and 3b the micrographic images are presented, obtained in the process of composition solving. They contain the polymer dried at 60^0C and 135^0C lithium chloride. Both samples represent the heterogenic system. Light color on the picture is indicative of solution uniformity.

On the sample microgram (Fig. 3b) there are no polymer non-solved inclusions and needle shaped formation which is indicative of polymer total solving.

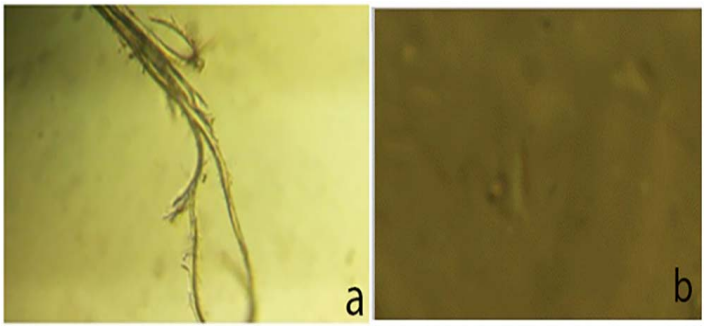


Fig.4. Micrographic images obtained by polarization-interference optical microscopy from the components of prepared composition dried at a) 60^0C and b) 135^0C

Hereafter the solutions were studied by dynamical method of light scattering. Polymeric composition presents the polydispersity system in which the particle size depends on the temperature of solution preparation, solvent nature and concentration. At the mentioned temperatures, the sizes of nanoparticles, obtained after solving of dry components containing polymeric composition and specific productivities of the membranes, prepared from the mentioned solution are presented in Table 4 and Table 5

Table 4. Sizes of nanoparticles and polydispersity index for polymeric composition containing dried at 600C, 750C, 900C,1050C, 1200C and 1350C components

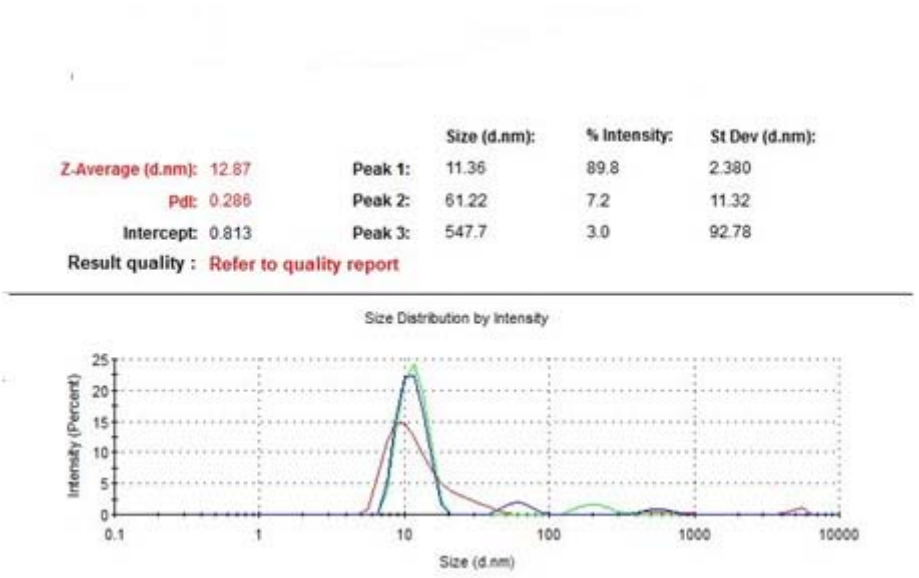
№	Name of the substance	peak 1, nm	%	peak 2, nm	%	peak 3, nm	%	Ksps	Poly-dispersity index, PDI	Z-Average, nm
1	60^0C dry Polymer and LiCl polymer Solution (5g/l)	9.549	47.2%	74.07	6.6%	1045	46.2%	53.3	0.987	619.1
2		8.710	49.4%	20.79	7.5%	955.4	40.4%	53.9	1.00	821.9
3		9.065	45.9%	54.76	6.2%	1102	47.9%	52.8	1.00	667.5
1	75^0C dry Polymer and LiCl polymer Solution (5g/l)	7.453	42.3%	19.25	15.9%	1048	41.8%	41.2	0.889	415.3
2		8.458	48.9%	28.44	9.1%	1104	42.0%	39.5	0.821	493.8
3		8.785	48.5%	30.65	6.9%	1027	44.6%	41.5	0.839	636.3
1	90^0C dry Polymer and LiCl polymer Solution (5g/l)	8.879	47.0%	20.67	9.1%	888.1	43.9%	49.4	1.00	648.7
2		6.297	16.6%	10.74	37.8%	995.4	41.0%	46.2	0.826	626.0
3		6.449	13.3%	11.17	37.2%	1279	46.0%	45.1	0.882	544.5
1	105^0C dry Polymer and LiCl polymer Solution (5g/l)	9.076	4.1	536.8	39.7	18.10	19.3	54.5	0.978	759.0
2		615.1	42.0	8.862	39.7	18.06	18.3	50.8	0.856	656.1
3		1079	51.0	11.76	49.0	0	0	46.6	0.727	436.0
1	120^0C dry Polymer and LiCl polymer Solution (5g/l)	9.908	82.9%	49.62	8.7%	694.3	8.5%	18.3	0.210	16.95
2		9.773	83.4%	76.36	8.0%	959.2	8.6%	18.0	0.228	15.33
3		10.05	84.3%	41.42	9.1%	515.8	6.8%	17.9	0.153	28.59
1	135^0C dry Polymer and LiCl polymer Solution (5g/l)	13.21	95.3	582.9	2.9	5183	1.8	20.4	0.257	12.07
2		11.68	93.4	208.3	6.6	0	0	20.4	0.232	13.56
3		11.36	89.8	61.22	7.2	547.7	3.0	19.9	0.286	12.87

Table 5. Constitution of polymeric composition containing dried (at 600C, 750C, 900C,1050C, 1200C and 1350C) components and specific productivity of prepared membranes

Drying temperature of components of polymer composition, t^0C	Particles size, Z-average, nm	Polydispersity index, PDI	Flux of pure water, $L/m^2\,h$
60	667.5	1.00	1200
75	636.3	0.839	1877
90	544.5	0.882	2188
105	436.0	0.727	2510
120	28.59	0.153	3600
135	12.87	0.286	4200

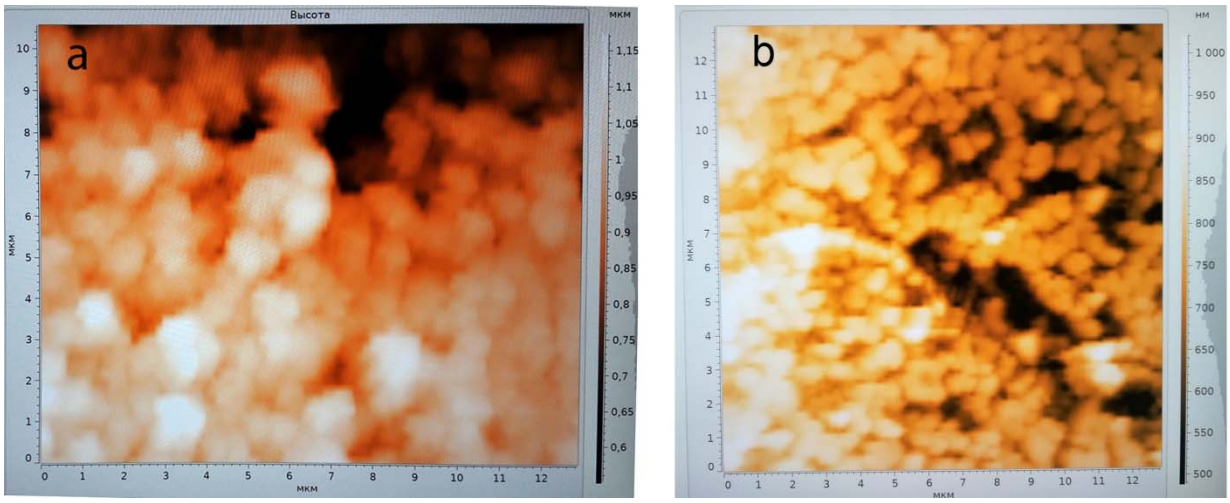
Effect of the drying temperature of the polymer and lithium chloride was studied the composition diluted to 5 g/l.

From the data of Table 4 it is evident that the particles size and polydispersity index in the solution and the values of specific productivity of the membranes, prepared from mentioned solutions, depend on drying temperature of polymer and lithium chloride. By increase of drying temperature for the components of polymeric composition the polydispersity index of the solution decreases from 1.00 to 0.286, size of macromolecules – from 667.5nm to 12.87nm. The specific productivity of the membrane prepared by dried components at 135^0C, exceeds the same value for the membrane obtained from the solution, prepared by dried components at 60^0C, 75^0C, 90^0C. The productivity comprises 4200 L/m^2h. The size of nanoparticles of polymeric composition prepared by dried components (135^0C) is given in Pig 5.



Pig.5. Size of nanoparticles of polymeric composition prepared by dry components at 135^0C by intensity, determined at the analyses Zetasizer Nano Zen3690

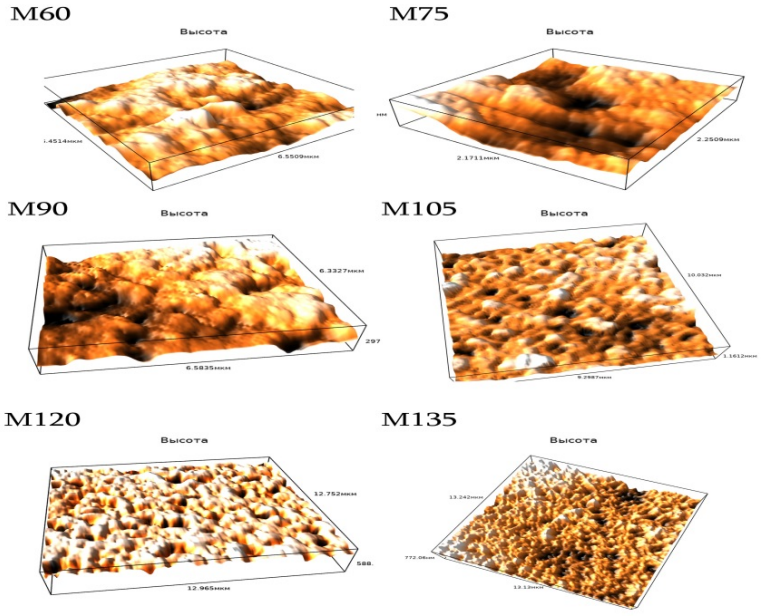
Membrane films obtained from polymeric composition, containing components dried at various temperatures, were studied by scanning probe microscope. Micrographic images of the membranes obtained from the solution, prepared by dried components at 60^0C and 135^0C, are presented in Pig. 6. The surface of both samples presents the form similar matrix, the light color formations on the picture are interpreted as the surface and dark color – as the pores.



Pig6. Microphotographic 2D images of membrane surfaces obtained by scanning probe microscope: (a) at 60^0C and (b) at 135^0C from the composition prepared by dried components

On the surface of a – sample dark and light sections are distributed non-uniformly, light color is dominant which is indicative of the small amount of the pores on the surface. On the surface of b - sample light and dark colors are distributed uniformly which is indicative of uniformity and porosity of membrane surface.

In polymeric composition, prepared from the components dried at high temperature, reduction of particle sizes and polydispersity index is caused by structural changes in the solution, containing the components dried at 135^0C. This fact reduces the size of micro-gel particles (disperse phase of the solution in the form of aggregate molecules) and increases their amount in unit volume. Yow sizes of micro-gel particles in the composition and their distribution in the solution determine the large areas of the pores formation (Pig.7), high porosity, causing the relatively high specific productivity of obtained membrane [22-23].



Pig. 7. Micro photographic 3D images of membrane surfaces obtained by scanning probe microscope: (M60) at 600C, (M75) at 750C, (M90) at 900C, (M105) at 1050C, (M120) and (M135) at 1350C from the composition prepared by dried components

3.2. Characterization of membrane porosity and specific productivity

The results of the tests of the membranes, prepared from the components dried at various temperatures, for sterile-finishing filtration of water and wine, on specific productivity are given in Table 5.

By increase of drying temperature of poly [N, N′– (1,3-phenylene) isophthalamide] and lithium chloride the membranes porosity and specific productivity enhance. The mentioned value for the membrane, prepared by the components dried at 135^0C, is higher than for the membrane obtained by the components dried at 60^0C, 75^0C, 90^0C, 105^0C, 120^0C, which is caused by increase of the pores amount per unit area.

IV. Conclusions

- As a result of empiric research the conditions were determined which provide the decomposition of the particles to minimal size in polymeric mass and the revealing of their maximum indices, allowing the prediction of specific productivity of membrane film by regulation of necessary area for formation of membrane pores;
- It has been established that by the temperature variation of drying of the components of polymeric composition such distribution of polydispersity index, nanoparticles size and concentration is attained which determines the increase of specific production of obtained membranes;
- The increase of the porosity of obtained membranes and, respectively, of specific productivity was attained by regulation of the drying temperature of the components of polymeric composition as well as by control of the sizes of nanoparticles.

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