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Д.В.Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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PHYSICAL-CHEMICAL AND COLLOID-MECHANICAL METHODS OF RESEARCH OF MODIFIED POLYMER REAGENTS OF THE M-PAA SERIES AND THEIR APPLICATION FOR OBTAINING OF OINTMENT

Abstract. This article discusses the surface, bulk and structural properties of polymers. It was determined that the limit value σ for M-PAA (MEA) and M-PAA (PV) is established faster than for individual solutions. It has been established that in polymers viscosity and electrical conductivity increase with increasing concentration, i.e. M-PAA (MEA) and M-PAA (PV) are polyfunctional polyelectrolytes, polymers M-PAA (MEA) and M-PAA (PV) are thermostable. For the study a complex of physicochemical methods were used: potentiometry, spectroturbidimetry, conductometry, viscometry, IR spectroscopy, elemental analysis, thermogravimetry, measurement of surface tension by the Wilhelm method.

Key words: modified polymers, M-PAA (MEA), M-PAA (PV), IR spectra, thermal stability.

Introduction

The preparation of new polymer reagents from their synthesis in the laboratory to their industrial production as an ointment is relatively long and expensive process. Therefore, the most promising and justified way is to expand the range of polymer reagents by modifying the already known base samples [1]. In the [2] work authors synthesized a serial of amino-modified PAN fibers by the reaction of PAN fiber with TETA in the presence of water. The weight and alkali content, namely, amino grafting degree, would be improved by prolonging reaction time and increasing reaction temperature.

2 Experimental part

2.1 Preparation of solutions

For deposition, a 1% polymer solution and a 0.15% hydrochloric acid solution were prepared. With constant stirring, a solution of hydrochloric acid was added to a 1% polymer solution; as a result, the pH dropped to 4 and a gel-like polymer precipitated out, which was washed 3 times with water. Dioxane was precipitated. In this case, the remnants of the starting products were dissolved in dioxane, and the polymer precipitated in the form of a gel. The precipitated, washed samples were dried in a vacuum desiccator. Dialysis of a 1% polymer solution was carried out in a celluloid packet, periodically changing the water (within 1-2 days). Next, the precipitated dialyzed samples were dried in a vacuum oven T = 40-450 ^{0}C for 8-10 hours. To determine the composition of the polymers, thermal analyzes were performed.

2.2 Research Methods

IR spectroscopy and electron microscopy analyzes.

The IR spectra of polymers were interpreted according to the guidelines [3]. The analysis was carried out on an IR-20 spectrometer (in vaseline oil in the range of 700-4000 m^{-1}).

Device: FTIR-spectrometer Shimadzu IR Prestige-21 with prefixed disturbed total internal reflection (ATR) Miracle from Pike Technologies.

Thermogravimetric studies of the polymers were carried out on a derivatogaf in an atmosphere of air at a rate of temperature rise up to 450 °C per minute, and a sample weight of 150 mg.

For a quantitative comparison of the thermal stability of the polymers under study, the activation energies of destruction were calculated [4].

The viscosity of solutions of polyelectrolytes was measured in a viscometer of the Ubbelohde type [5], with a hanging level. For viscometric studies, re-precipitated and thoroughly dried polymers were used.

The viscometer was placed in a thermostat, the temperature was maintained with an accuracy of ± 0.01 ° C.

The concentration of the solution after dilution was calculated by the formula:

$$C = \frac{gVi - 100}{V(Vi + Vj)} Pi / P_2 = \frac{ciVi}{Vi + Vj}, \quad (1)$$

where g is the polymer weight, g;

V is the volume of the volumetric flask, ml;

V_i is the volume of the solution filled in viscometers, ml;

V_J is the volume of the added solvent, ml;

P_i / P_J is the solvent density ratio.

The calculation of the relative η_ (rel.) Specific η (spec), The reduced η_ (red.) Viscosities was carried out according to the following formulas:

$$\eta_{rel.} = \frac{\tau_i}{\tau_0}; \quad (2)$$

$$\eta_{spec} = \eta_{rel.}^{-1;} \quad (3)$$

$$\eta_{red.} = \frac{\eta_{spec.}}{c} \quad (4)$$

The characteristic viscosity (η) was found from the graphical dependence η_{red} (C) by extrapolating the straight lines to zero polymer concentration [6].

Measurement of the surface tension was carried out according to the method of Wilhelmy.

When determining the surface tension of the solutions by the method of Wilhelmi, the immersion force of the plates in the liquid was continuously measured. The magnitude of this force depends on the wetting of the measuring plate with liquid.

In the present work, a polished glass plate was used as a measuring plate, as well as made from a plate.

The calculation was carried out as follows:

$$\sigma = \frac{p + shd}{2(1+b)}, \quad (5)$$

p-weight plate, g; s- cross-sectional area, cm; 1 is the width of the plate, cm; -b is plate thickness (-), cm; h-immersion depth, cm; d-density of the measured liquid, g / cm; g-acceleration of gravity.

The surface tension was measured with a thermostatically controlled glass cell with a lid at a temperature of 25 °C; the temperature was kept constant using an I-2 ultra thermostat with an accuracy of +/- 0.02. The reading of the values of P_x was carried out using toroidal weights of the type VT-500 with an accuracy of +/-1 mg.

The pH of polymer solutions was measured on a pH-340 potentiometer with an accuracy of +/- 0.05. The pH in a thermostatically controlled cell, in which the temperature was maintained with an accuracy of 25 +/- 0.01. Further, the formula determined the conductivity (χ):

$$\chi = \frac{\alpha}{Rx}; \quad (6)$$

where - α is the cell constant: α = 0.000147m-1cm-1, at T = 250

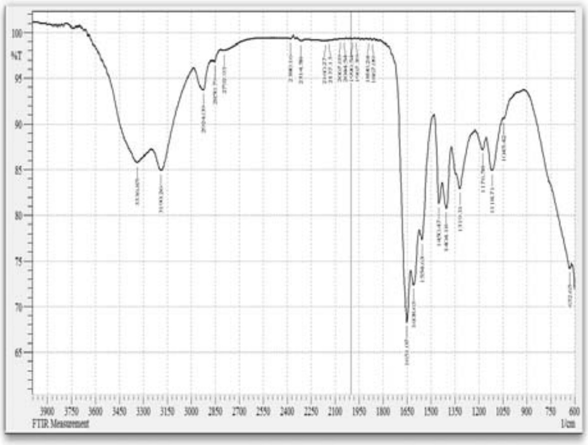
Turbidity was determined on a calorimeter of the type FEK-56 with α = 434Nm. Solutions of the corresponding fractions of polymers were used as reference solutions.

3 Results and discussions

The study of the structural properties of the polymers M-PAA (MEA), M-PAA (PV).

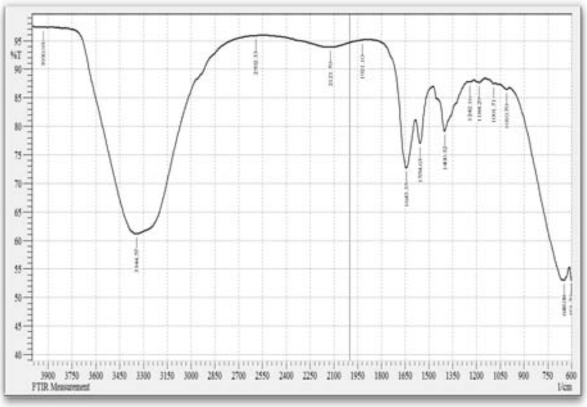
The IR spectra of polymers, whose interpretation was carried out according to the guidelines [7], contain absorption bands in the range of 3200-3500 cm^{-1}, which can be attributed to hydroxyl groups of an alcoholic nature, both free and hydrogen bonded. Bands with frequencies of 1405-1410 and also 1610-1630 cm^{-1} characterize the presence of -NO_2, -COO^- groups. According to some authors [8], the pictogroup included in the core contributes to the stability of the first to destruction.

Absorption bands in the range of 1600–1630 cm^{-1} can be attributed to skeletal vibrations of –C = C – bonds, rather distinct bands of stretching vibrations — CH^{2-} groups are observed in the short-wave part of the spectra (2940-2970) cm^{-1}. Characteristic for –CH groups are peaks at frequencies of 780–790 cm^{-1}, and a maximum at a frequency of 1370 cm^{-1} can be attributed to deformational vibrations of –CH groups. In the spectrum of the studied samples, new bands are noted at frequencies of 1480 and 1690 cm^{-1}, which can be identified as -$COONH_2$ and – $(CO)_2NH$ (Figure 1-5) [9].



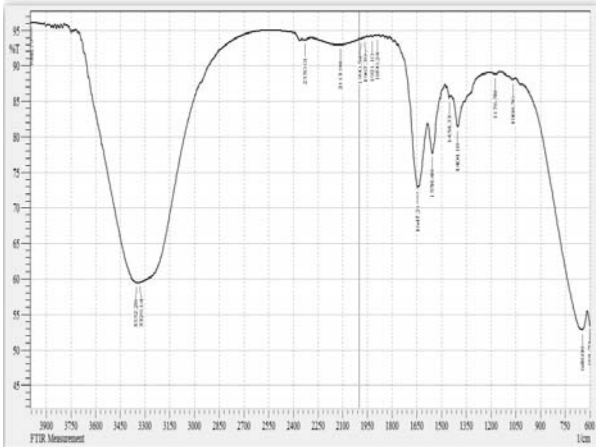
No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	632.65	74.183	1.297	918.12	621.08	22.196	0.246
2	1045.42	90.603	0.123	1049.28	921.97	4.167	0.009
3	1118.71	84.906	4.264	1153.43	1049.28	6.068	1.085
4	1176.58	87.194	1.502	1222.87	1157.29	3.605	0.232
5	1319.31	82.899	5.154	1369.46	1226.73	9.205	1.527
6	1404.18	80.802	4.411	1427.32	1373.32	4.292	0.651
7	1450.47	81.298	5.208	1481.33	1431.18	3.602	0.579
8	1554.63	77.284	3.312	1570.06	1485.19	6.197	0.371
9	1608.63	72.370	2.803	1624.06	1573.91	6.372	0.457
10	1651.07	68.236	9.961	1762.94	1627.92	10.149	1.958
11	1867.09	99.142	0.180	1874.81	1855.52	0.063	0.006
12	1890.24	99.270	0.062	1897.95	1878.67	0.058	0.002
13	1967.39	99.299	0.075	1978.97	1955.82	0.066	0.003
14	1990.54	99.279	0.083	2002.11	1982.82	0.056	0.003
15	2044.54	99.323	0.027	2048.40	2032.97	0.044	0.001
16	2067.69	99.286	0.025	2071.55	2056.12	0.047	0.001
17	2137.13	99.123	0.036	2148.70	2071.55	0.269	0.008
18	2160.27	99.108	0.020	2218.14	2156.42	0.235	0.005
19	2314.58	99.102	0.261	2337.72	2279.86	0.201	0.034
20	2380.16	99.325	0.164	2391.73	2364.73	0.068	0.012
21	2792.93	98.027	0.160	2812.21	2580.76	1.207	0.039
22	2850.79	96.833	0.361	2862.36	2816.07	0.500	0.017
23	2924.09	93.759	2.771	2981.95	2881.65	2.213	0.653
24	3190.26	84.900	4.279	3248.13	2985.81	11.920	2.002
25	3336.85	85.753	4.188	3556.74	3251.98	14.786	3.613

Figure 1 - The main polymer PAA (Polyacrylamide) powder



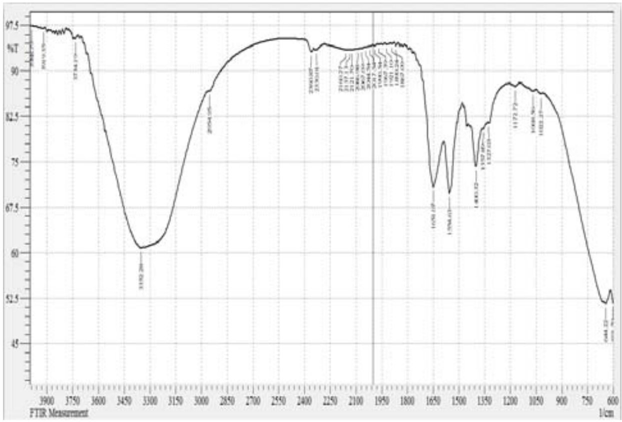
No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	601.79	52.995	0.583	613.36	597.93	4.186	0.039
2	648.08	52.950	0.567	655.80	617.22	10.380	0.147
3	1010.70	86.409	0.473	1053.13	995.27	3.554	0.056
4	1091.71	87.389	0.285	1122.57	1083.99	2.190	0.020
5	1184.29	87.646	0.228	1192.01	1145.72	2.562	0.020
6	1242.16	87.742	0.147	1257.59	1215.15	2.382	0.013
7	1400.32	79.098	6.419	1446.61	1261.45	14.420	2.561
8	1554.63	76.917	5.692	1581.63	1477.47	8.689	0.960
9	1643.35	72.667	11.637	1840.09	1585.49	17.100	4.014
10	1921.10	95.075	0.025	1924.96	1905.67	0.419	0.000
11	2121.70	93.826	1.364	2360.87	1928.82	10.707	1.483
12	2592.33	95.873	0.011	2596.19	2565.33	0.562	0.001
13	3344.57	61.128	35.473	3741.90	2600.04	110.148	92.111
14	3930.93	97.308	0.053	3938.64	3915.50	0.272	0.003

Figure 2 - MEA-PAA 1, colorless, viscous, non-flowing solution



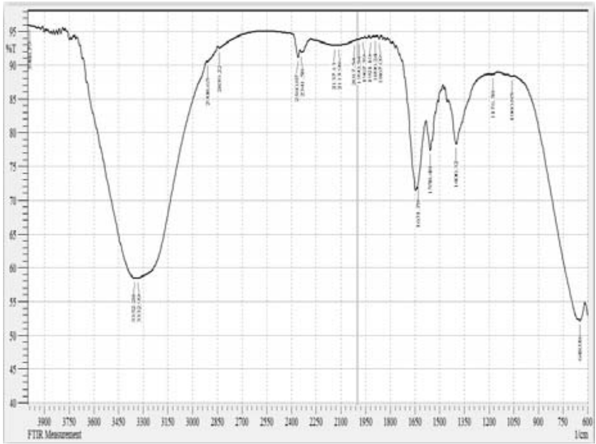
No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	601.7	53.359	0.428	617.22	597.93	5.136	0.014
2	648.0	52.773	4.816	1010.70	621.08	63.232	4.628
3	1068.	88.052	0.328	1145.72	1053.13	4.884	0.054
4	1176.	88.831	0.285	1203.58	1149.57	2.743	0.041
5	1404.	81.358	4.981	1442.75	1207.44	14.997	1.418
6	1454.	85.743	0.508	1465.90	1446.61	1.279	0.040
7	1558.	77.707	5.328	1581.63	1481.33	8.291	1.029
8	1647.	72.870	12.862	1762.94	1585.49	15.317	4.731
9	1890.	94.216	0.097	1897.95	1878.67	0.493	0.004
10	1921.	94.090	0.169	1928.82	1901.81	0.700	0.010
11	1967.	93.962	0.068	1971.25	1951.96	0.513	0.002
12	1990.	93.763	0.098	1998.25	1975.11	0.634	0.003
13	2113.	92.936	0.092	2125.56	2002.11	3.743	0.069
14	2330.	93.666	0.054	2333.87	2295.29	1.069	0.005
15	3329.	59.424	0.177	3332.99	2557.61	68.814	1.253
16	3352.	59.358	1.186	3703.33	3340.71	47.856	2.637
17	3988.	96.108	0.022	4000.36	3984.93	0.265	0.001

Figure 3 - MEA-PAA 2, yellowish, viscous, non-flowing solution



No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	601.7	51.618	0.536	613.36	597.93	4.365	0.035
2	644.2	51.623	0.947	659.66	617.22	11.923	0.192
3	1022.	86.192	0.221	1049.28	1014.56	2.205	0.026
4	1068.	86.598	0.383	1095.57	1053.13	2.608	0.043
5	1172.	87.397	0.524	1203.58	1149.57	3.091	0.071
6	1327.	81.407	0.288	1330.88	1219.01	7.480	0.027
7	1357.	80.352	0.214	1361.74	1346.31	1.428	0.007
8	1400.	74.240	6.423	1442.75	1369.46	7.899	1.076
9	1554.	69.800	10.598	1589.34	1481.33	12.115	2.573
10	1651.	70.748	13.039	1724.36	1593.20	14.411	4.554
11	1867.	94.105	0.485	1874.81	1855.52	0.484	0.018
12	1890.	94.432	0.158	1897.95	1878.67	0.471	0.007
13	1921.	94.303	0.264	1928.82	1901.81	0.669	0.016
14	1967.	94.291	0.121	1975.11	1951.96	0.580	0.004
15	1990.	94.113	0.169	1998.25	1978.97	0.496	0.006
16	2017.	93.991	0.098	2025.26	2002.11	0.612	0.004
17	2044.	93.841	0.040	2048.40	2025.26	0.629	0.002
18	2067.	93.664	0.038	2071.55	2048.40	0.648	0.002
19	2086.	93.548	0.025	2090.84	2071.55	0.551	0.000
20	2121.	93.469	0.017	2125.56	2098.55	0.788	0.002
21	2137.	93.453	0.022	2148.70	2125.56	0.679	0.001
22	2160.	93.457	0.040	2283.72	2156.42	3.498	0.024
23	2330.	93.412	0.391	2345.44	2287.58	1.597	0.046
24	2360.	93.064	0.710	2488.17	2349.30	3.205	0.044
25	2954.	86.586	0.077	2958.80	2519.03	14.179	0.003
26	3352.	60.780	29.214	3660.89	2962.66	100.831	67.430
27	3734.	95.268	0.426	3741.90	3718.76	0.448	0.016
28	3919.	96.907	0.278	3934.78	3911.64	0.307	0.019
29	3988.	97.366	0.024	4000.36	3984.93	0.178	0.001

Figure 4 - PAA-H_2O_2, colorless, viscous, weakly flowing solution



No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	648.08	52.165	0.967	659.66	621.08	10.644	0.243
2	1060.85	88.338	0.107	1130.29	1053.13	4.081	0.035
3	1176.58	88.582	0.224	1203.58	1149.57	2.812	0.031
4	1400.32	78.327	7.148	1446.61	1207.44	17.382	2.624
5	1558.48	77.466	5.460	1581.63	1481.33	8.491	1.124
6	1631.78	71.775	0.765	1635.64	1585.49	5.618	0.016
7	1867.09	93.871	0.462	1874.81	1855.52	0.506	0.017
8	1890.24	94.165	0.155	1897.95	1878.67	0.495	0.006
9	1921.10	94.022	0.256	1928.82	1901.81	0.704	0.016
10	1967.39	93.947	0.105	1971.25	1951.96	0.512	0.003
11	1990.54	93.723	0.161	1998.25	1975.11	0.634	0.005
12	2017.54	93.548	0.090	2025.26	2002.11	0.658	0.003
13	2113.98	92.906	0.058	2121.70	2025.26	2.970	0.042
14	2137.13	92.908	0.081	2279.86	2125.56	4.662	0.068
15	2341.58	91.890	0.381	2345.44	2283.72	1.983	0.065
16	2360.87	91.136	1.344	2526.75	2349.30	4.516	0.085
17	2839.22	92.473	0.267	2846.93	2557.61	7.479	0.024
18	2908.65	90.507	0.309	2916.37	2850.79	2.568	0.094
19	3332.99	58.421	0.598	3340.71	2916.37	62.493	5.256
20	3352.28	58.406	1.192	3664.75	3340.71	48.513	3.723
21	3988.79	95.811	0.032	4000.36	3984.93	0.286	0.002

Figure 5 - PAA-H_2O_2, yellowish, viscous, non-flowing solution

Thus, the IR spectra of the studied samples contain: -NO_2, -$COOH$, $-(CO)_2NH$, -$CONH_2$, -OH groups.

The stability of the obtained polymer to thermo-oxidative degradation was studied on a derivatograph of the Paulik-Paulik-Erdey system using the method of "dynamical thermogravimetry". The heating rate is 6 deg/min., The interval is 293-870 K. (Table 1).

Table 1 - Characteristics of thermal stability of polymers M-PAA (MEA), M-PAA (PV), K-9

Polymer	T	E, кj/ mol	Weight loss	Initial weight, kg-10^{-1}
M-PAA (MEA)	698,4	96,2	31,5	552,6
K-9	670,7	78,7	49,8	552,3
M-PAA (PV)	703,4	94,3	39,4	550,8

From the data in the table it can be seen that the weight loss in thermogravimetry K-9 is 48.3%, and the polymer M-PAA (MEA) obtained on its basis is 30%, which is 1.5 times less. The weight loss of the polymer M-PAA compared with K-9 is also lower. In addition, the temperature of the onset of active decomposition in new samples increases markedly compared with the baseline ones. Thus, a qualitative assessment of the thermal stability of the process is obtained.

A quantitative comparison of the thermal stability of the samples under study will allow the calculation of the activation energy of destruction [3].

So, for thermo-oxidative destruction of K-9, 78.5 kJ / mol is required, while for the destruction of the polymer M-PAA (PV) - 85.3 kJ / mol, polymer M-PAA (MEA) - 84.1 kJ / mole. This gives grounds to attribute them to thermostable polymers [7].

Conclusion. Summarizing the above, we can draw the following conclusions:

a) Surface properties:

- The limiting value of σ for M-PAA (MEA) and M-PAA (PV) is established faster than for individual solutions;

- adsorption in these polymers is mainly determined by the diffusion of the macromolecule;

c) Bulk properties:

- in the polymers, with increasing concentration, viscosity and electrical conductivity increases, i.e. M-PAA (MEA) and M-PAA (PV) are polyfunctional polyelectrolytes.

c) Structural properties:

- polymers M-PAA (MEA) and M-PAA (PV) are thermostable;

- IR spectra of samples contain -NO_2, -$COOH$, -CN, $-SO_3Na$, $-OH$, -$CONH_2$ groups.

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М-РАА СЕРИЯЛЫ МОДИФИЦИРЛЕНГЕН ПОЛИМЕРЛІ РЕАГЕНТТЕРДІҢ ФИЗИКА-ХИМИЯЛЫҚ ЖӘНЕ КОЛЛОИДТЫ-МЕХАНИКАЛЫҚ ӘДІСТЕРМЕН ЗЕРТТЕУ ЖӘНЕ ОЛАРДЫ МАЗЬДЕРДІ АЛУДА ҚОЛДАНУ

Аннотация. Бұл мақалада полимерлердің беттік, көлемдік және құрылымдық қасиеттері қарастырылады. M-PAA (MEA) және M-PAA (PV) шекті мәні жеке ерітінділерге қарағанда тезірек орнығатыны анықталды. Концентрацияларының артуына байланысты полимерлердің тұтқырлығы мен электрөткізгіштігі артатыны анықталды, яғни M-PAA (MEA) и M-PAA (PV) полифункциналды полиэлектролиттер болып табылады, M-PAA (MEA) и M-PAA (PV) полимерлері термостабильді болып табылады. Зерттеу үшін физика-химиялық әдістер жиынтығы пайдаланылды: потенциометрия, спектротурбидиметрия, кондуктометрия, вискозиметрия, ИК-спектроскопия, элементтік анализ, термогравиметрия, Вильгельми әдісімен беттік керілуді өлшеу.

Түйін сөздер: модифицирленген полимерлер, M-PAA (MEA), M-PAA (PV), ИҚ спектрлері, жылу тұрақтылығы

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ФИЗИКО-ХИМИЧЕСКИЕ И КОЛЛОИДНО-МЕХАНИЧЕСКИЕ МЕТОДЫ ИССЛЕДОВАНИЯ МОДИФИЦИРОВАННЫХ ПОЛИМЕРНЫХ РЕАГЕНТОВ СЕРИИ М-РАА И ИХ ПРИМЕНЕНИЕ ДЛЯ ПОЛУЧЕНИЯ МАЗЕЙ

Аннотация. В данной статье рассматриваются поверхностные, объемные и структурные свойства полимеров. Было определено, что предельное значение σ для М-РАА (МЕА) и М-РАА (PV) устанавливается быстрее, чем для отдельных растворов. Установлено, что в полимерах вязкость и электропроводность увеличиваются с увеличением концентрации, то есть М-РАА (МЕА) и М-РАА (PV) являются полифункциональными полиэлектролитами, полимеры М-РАА (МЕА) и М-РАА (PV) являются термостабильными. Для исследования использовали комплекс физико-химических методов: потенциометрия, спектротурбидиметрия, кондуктометрия, вискозиметрия, ИК-спектроскопия, элементный анализ, термогравиметрия, измерение поверхностного натяжения методом Вильгельми.

Ключевые слова: модифицированные полимеры, М-ПАА (МЭА), М-ПАА (ПВ), ИК-спектры, термостабильность.

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