

PHYSICOCHEMICAL ASPECTS OF POLYELECTROLYTE INTERACTIONS IN AQUEOUS FILM-FORMER DISPERSIONS

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Abstract. *Introduction.* Waterborne coating materials offer environmental and technological advantages; however, the coatings they form may exhibit structural heterogeneity and increased porosity. One approach to controlling the properties of such systems is the introduction of functional modifiers. *Objective.* The aim of this study was to determine the effect of polyethylene polyamine on the surface and bulk properties of aqueous polyurethane and acrylic-polyurethane dispersions, as well as on the porosity of the coatings formed from them. *Methodology.* At 293 K, surface tension, specific and molar electrical conductivity, pH, and coating porosity were determined in systems containing 0-0.0024 mol/dm³ PEPA. *Results and Discussion.* In the binary «water-PEPA» system, changes in the character of the concentration dependences of surface tension, molar conductivity, and pH were observed in the region CPEPA = 0.0012 mol/dm³, corresponding to the critical association concentration. In ternary systems, the nature of changes in the investigated parameters depended on the film-former chemistry; the most pronounced decrease in surface tension was observed for the acrylic-polyurethane dispersion, from 39.60 to 35.15 mN/m. With increasing PEPA concentration, the porosity of the polyurethane coating decreased from 73.70 to 40.56%, while that of the acrylic-polyurethane coating decreased from 37.69 to 4.62%. *Conclusion.* The results demonstrate that PEPA affects the surface and bulk properties of the investigated aqueous dispersions and the structure of the coatings formed. The observed changes are consistent with the possibility of associative interactions between PEPA and the components of the studied systems, with the most pronounced modification effect established for the acrylic-polyurethane composition.

Keywords: acrylic-polyurethane; polyurethane; surface and bulk properties; polyethylene polyamine; aqueous film-former dispersions

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1. Introduction

Waterborne coating materials are widely used because of their environmental and technological advantages over solvent-borne systems. At the same time, the coatings formed from these materials may exhibit structural heterogeneity, increased porosity, and insufficient corrosion resistance [1-3].

One approach to controlling the properties of such materials is modification of heterochain film-forming polymers containing polar ester ($-\text{COO}-$), amide ($-\text{CONH}-$), and urethane ($-\text{NH}-\text{CO}-\text{O}-$) groups capable of participating in intermolecular interactions [3-5]. Another approach is the introduction of multifunctional surfactants that can alter the surface and bulk properties of aqueous dispersions [6, 7].

Amine-containing modifiers are of particular interest because they are capable of forming ion pairs and hydrogen bonds with polar groups of the polymer phase [6-8]. Polyethylene polyamine (PEPA) belongs to this class of compounds and contains primary and secondary amino groups. Therefore, the aim of this work was to establish the effect of PEPA on the surface and bulk properties of aqueous polyurethane and acrylic-polyurethane dispersions and on the porosity of the coatings formed from them.

2. Experimental part

The following materials were used: a waterborne polyurethane dispersion, TU 2313-001-254543-2015; a waterborne acrylic-polyurethane dispersion, TU 20.30.12-041-29525189-2019; and polyethylene polyamine, TU 2413-357-00203447-99. The composition and structural features of the samples were characterized by FTIR spectroscopy using a Shimadzu instrument (resolution 4 cm^{-1} , 45 scans, Happ-Genzel apodization). The spectra obtained are shown in Figure 1.

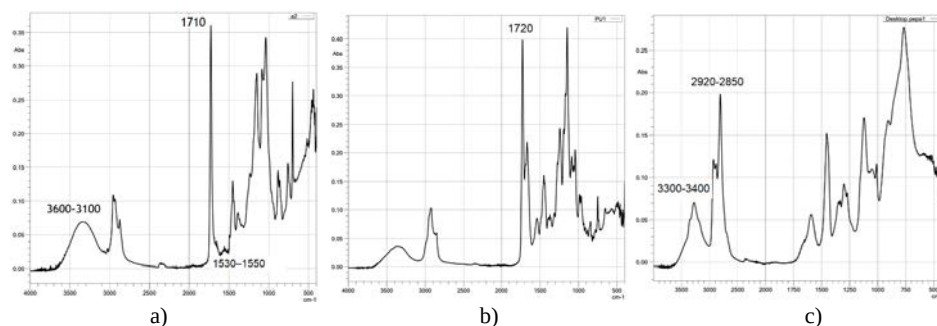


Figure 1 – FTIR spectra of the investigated samples: a – polyurethane; b – acrylic-polyurethane; c – polyethylene polyamine.

FTIR spectra (Figure 1) confirmed the characteristic functional groups of the investigated components. Polyurethane exhibited N-H stretching bands at $3600\text{--}3100\text{ cm}^{-1}$, a urethane $\text{C}=\text{O}$ band near 1710 cm^{-1} , and C-N vibrations at 1530--

1550 cm^{-1} (Figure 1a) [9, 10]. The acrylic-polyurethane spectrum additionally showed an intense C=O band of the acrylate component near 1720 cm^{-1} (Figure 1b) [9, 10]. PEPA was characterized by N-H stretching at 3300–3400 cm^{-1} and CH_2 bands at 2920–2850 cm^{-1} (Figure 1c) [11].

The studies were carried out in binary «water-PEPA» systems and ternary «polyurethane/acrylic-polyurethane-water-PEPA» compositions. The concentration of the amine-containing modifier was varied from 0 to 0.0024 mol/dm^3 in increments of 0.0004 mol/dm^3 . Freshly prepared solutions and compositions were used for all measurements, which were performed at 293 K.

PEPA solutions of the required concentrations for the binary systems were prepared in a specified volume of distilled water under stirring until complete homogenization. Ternary compositions were prepared by adding PEPA to a calculated volume of the polyurethane or acrylic-polyurethane dispersion, followed by stirring until a visually homogeneous system without visible aggregates was obtained. Surface tension, specific electrical conductivity, and pH were monitored over time until stable readings were established, which for the investigated systems occurred predominantly after 30–40 min. Statistical treatment was performed using the results of three independent replicate determinations ($n=3$) recorded after stabilization of the corresponding parameter. For each experimental point, the arithmetic mean ($\bar{x}$), standard deviation (SD), and relative standard deviation (RSD) were calculated:

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n}; SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}; RSD = \frac{SD}{\bar{x}} \times 100\%, \quad (1)$$

where x_i – the result of an individual determination;

n – the number of replicate measurements.

Experimental data in the figures are presented as $\bar{x} \pm SD$.

Surface tension σ was determined using an automated ACAM instrument by the pendant-drop method. The drop profile was recorded by a video-imaging system, and σ was calculated by the instrument software using the Young-Laplace equation [12].

Specific electrical conductivity χ was measured using a Multitest conductivity meter calibrated with a standard solution. Molar conductivity λ_m was calculated from the experimentally measured specific conductivity according to [13]:

$$\lambda_m = \frac{\chi}{C}, \quad (2)$$

where λ_m – molar conductivity, $\text{S} \cdot \text{cm}^2/\text{mol}$;

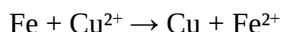
χ – specific electrical conductivity, mS/cm ;

C – the molar concentration of PEPA, mol/dm^3 .

The pH values were measured using a laboratory pH-150MI pH meter equipped with an ESK-10603/7 combination electrode and automatic temperature compensation. Before measurements, the instrument was calibrated using standard buffer solutions.

Coating porosity was determined by indicator deposition of metallic copper with quantitative evaluation of the relative area of the formed copper-deposition sites [14]. Steel plates measuring 3×9 cm were sequentially ground with P800 and P1000 abrasive paper, degreased with ethanol, and dried at room temperature. The test compositions were applied by wiping with a lint-free cotton cloth and dried for 5 days. The actual mass of the formed coatings was 0.0038-0.0065 g for the polyurethane system and 0.0069-0.0123 g for the acrylic-polyurethane system.

A 1.0 mL aliquot of solution containing 10% CuSO₄ and 0.5% H₂SO₄ was uniformly distributed over a 2×2 cm² area of the coating bounded by an adhesive contour and maintained at 295 K for 30 min. Penetration of the electrolyte through coating defects was accompanied by reduction of copper ions on the steel surface:



After removal of the solution, the surface was photographed under constant imaging conditions. The images were processed using Particle Analyzer v2.0 with color-segmentation and contour-analysis algorithms implemented in Python using the OpenCV library. Coating porosity CP (%) was calculated as the ratio of the total area of copper-deposition sites S_p to the total analyzed area S_0 :

$$\overline{CP} = \frac{S_p}{S_0} \times 100\%, \quad (3)$$

For each experimental point, three independent porosity determinations were performed; the results were expressed as $\overline{CP} \pm SD$.

3. Results and Discussion

The experimental data showed high reproducibility: RSD values did not exceed 0.26% for pH, 3.44% for specific electrical conductivity, and 2.41% for surface tension.

3.1 «Water-PEPA» system

The effect of additive concentration on the surface tension of water is shown in Figure 2.

The surface-tension isotherm (σ_{T-g}) of aqueous polyethylene polyamine solutions at 293 K is characterized by two concentration regions. As C_{PEPA} increased from 0 to 0.0012 mol/dm³, the surface tension decreased by 1.49 mN/m to 71.28 mN/m, after which it remained practically unchanged, forming a plateau region.

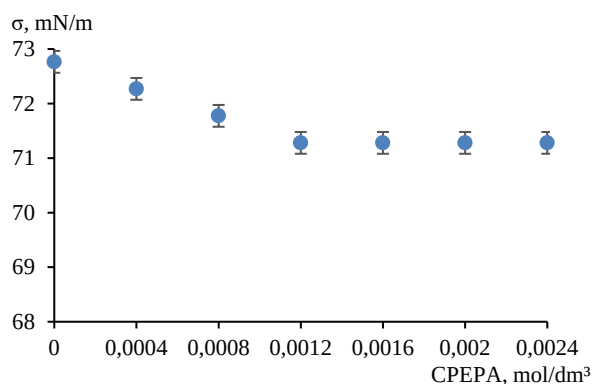


Figure 2 – Surface-tension isotherm of the «water-PEPA» system ($T = 293$ K).

The change in the character of the concentration dependence at $C_{PEPA} = 0.0012$ mol/dm³ allows this concentration to be considered the critical association concentration (CAC), which is consistent with literature data [6] and is confirmed by changes in the bulk characteristics of the corresponding systems (Figures 3 and 4).

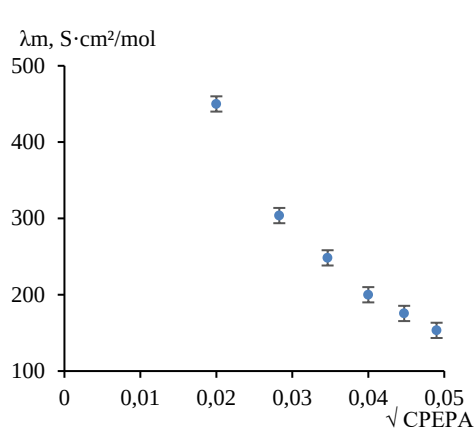


Figure 3 – Dependence of molar conductivity in the binary «water-PEPA» system on the square root of PEPA concentration.

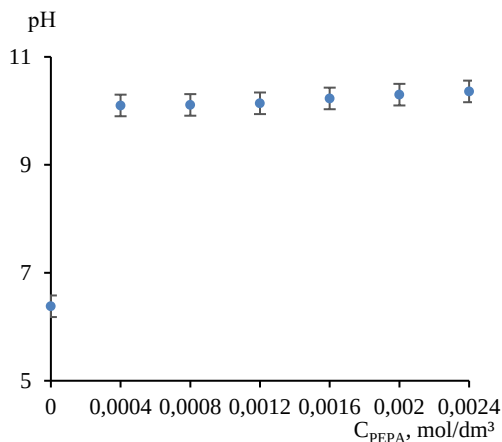


Figure 4 – Effect of polyethylene polyamine concentration on pH in the binary «water-PEPA» system.

In the concentration range $C_{PEPA}=0.0004$ – 0.0012 mol/dm³, the observed decrease in molar conductivity from 442.5 to 248.1 S·cm²/mol is consistent with gradual protonation of PEPA amino groups in aqueous medium [15] (Figure 3). Binding of protons from water by the amino groups is accompanied by formation of hydroxide ions, which is manifested by a shift of the medium from near-neutral ($C_{PEPA}=0$ mol/dm³) to alkaline ($C_{PEPA} = 0.0012$ mol/dm³) and is confirmed by the increase in pH from 6.38 to 10.14 (Figure 4).

The change in the character of the molar-conductivity (Figure 3) and pH (Figure 4) dependences near $C_{\text{PEPA}}=0.0012 \text{ mol/dm}^3$, together with formation of a plateau in the surface-tension isotherm (Figure 2), suggests a transition of the system toward an associated state [16]. Formation of PEPA associates may be governed by a combination of hydrophobic interactions between methylene segments, hydrogen bonding, and electrostatic interactions involving protonated amino groups [16, 17]. Upon association, the mobility of the resulting structures decreases relative to individual polyions, which may contribute to the decrease in molar conductivity.

With a further increase in PEPA concentration from 0.0012 to 0.0024 mol/dm^3 , molar conductivity decreased from 248.1 to 153.2 $\text{S}\cdot\text{cm}^2/\text{mol}$ (Figure 3), whereas the change in pH became less pronounced, from 10.14 to 10.36 (Figure 4). Over the same concentration interval, surface tension remained practically unchanged at approximately 71.28 mN/m (Figure 2), which is consistent with formation of a plateau after the CAC had been reached.

3.2 «Water-film former-PEPA» systems

The effects of film-former chemistry and additive concentration on the surface tension of the investigated systems are shown in Figure 5.

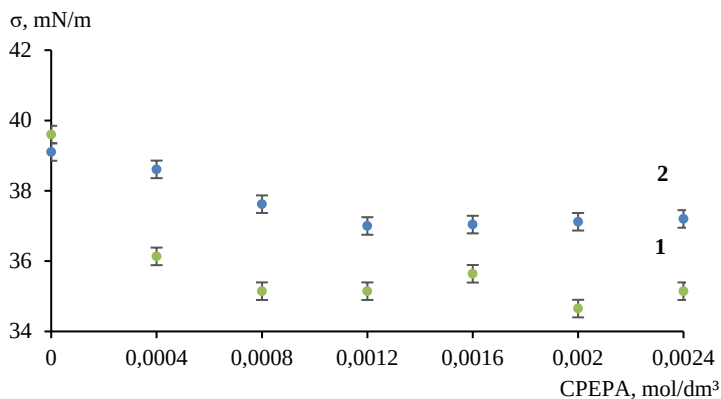


Figure 5 – Surface-tension isotherm ($T = 293 \text{ K}$): 1 – acrylic-polyurethane; 2 – polyurethane.

In the absence of PEPA, the aqueous dispersions of both film formers exhibited similar surface-tension values, $\sigma_{\text{l-g}}=39.11\text{--}39.60 \text{ mN/m}$. The similarity of these values may be related to the presence of polar urethane ($-\text{NH}-\text{CO}-\text{O}-$) and ether ($-\text{O}-$) groups in both systems (Figure 1). The small difference between the polyurethane and acrylic-polyurethane dispersions ($\Delta\sigma_{\text{l-g}}=0.49 \text{ mN/m}$) may be associated with the functional composition of the acrylic-polyurethane film former, which also contains acrylate fragments.

The surface tension of the initial film-former dispersions ($\sigma_{\text{l-g}}=39.11\text{--}39.60 \text{ mN/m}$) was substantially lower than that of water (72.77 mN/m), indicating pronounced surface activity of the dispersion components and formation of an

adsorption layer at the liquid-air interface [12]. After PEPA addition, differences between the investigated film formers became apparent in the way surface tension changed with increasing additive concentration (Figure 5).

For the acrylic-polyurethane system, surface tension decreased from 39.60 to 35.15 mN/m. This effect may be associated with additional intermolecular interactions between PEPA amino groups and polar groups of the acrylic-polyurethane film former, including hydrogen bonding and acid-base interactions [15, 16]. The results suggest that such interactions contribute to changes in the composition and organization of the surface layer.

For the polyurethane dispersion, the change in surface tension upon addition of PEPA was less pronounced: after the initial decrease, σ_{l-g} changed only slightly over the subsequent concentration range (Figure 5). The different concentration dependences of the two film formers indicate that the chemical nature of the polymer phase affects its interaction with PEPA.

Comparison of the acid-base characteristics of the aqueous dispersions in the absence of PEPA showed pH values of 8.19 for the acrylic-polyurethane dispersion and 6.87 for the polyurethane dispersion, whereas the pH of water was 6.38 (Table 1). These differences in initial pH characterize the acid-base state of the dispersions and may affect subsequent interactions between functional groups of the film formers and PEPA.

Table 1 – Effect of additive concentration on the pH of water, acrylic-polyurethane, and polyurethane compositions

$C_{PEPA}, \text{mol/dm}^3$	0	0.0004	0.0008	0.0012	0.0016	0.0020	0.0024
pH (polyurethane)	6.87	8.69	9.46	9.85	9.94	10.23	10.41
pH (acrylic-polyurethane)	8.19	8.85	9.37	9.81	10.10	10.98	11.08
pH (water)	6.38	10.10	10.11	10.14	10.23	10.30	10.36

The shift in pH toward the alkaline region after PEPA addition to the film former-water systems is consistent with the protonation of amino groups by water molecules discussed for the binary system, accompanied by release of hydroxide ions (OH^-). The pH increased continuously with increasing PEPA concentration and reached maximum values at $C_{PEPA}=0.0024 \text{ mol/dm}^3$: 10.41 for polyurethane and 11.08 for acrylic-polyurethane. These values are close to the pH of 10.36 measured for the binary «water-PEPA» system at the same additive concentration (Table 1).

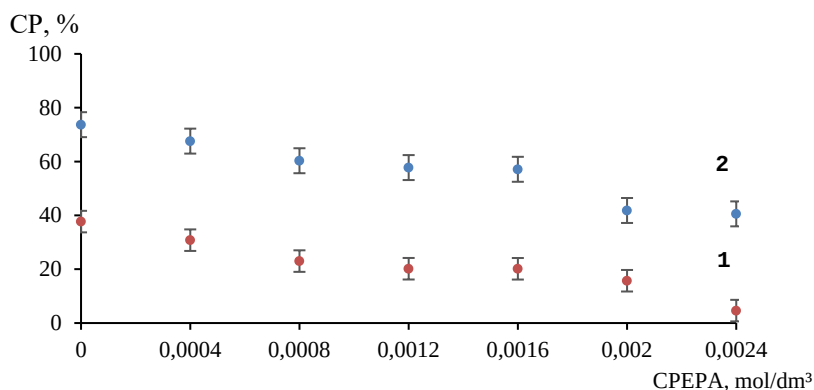
Concentration-dependent changes in the specific electrical conductivity of the investigated systems are presented in Table 2.

Table 2 – Effect of additive concentration on the specific electrical conductivity of water, acrylic-polyurethane, and polyurethane compositions

C_{PEPA} , mol/dm ³	0	0.0004	0.0008	0.0012	0.0016	0.0020	0.0024
χ (polyurethane), mS/cm	1.39	1.39	1.34	1.29	1.24	1.29	1.26
χ (acrylic-polyurethane), mS/cm	3.80	3.71	3.42	3.50	3.19	3.06	2.57
χ (water), mS/cm	0.0052	0.1770	0.2427	0.2977	0.3197	0.3507	0.3677

The overall tendency toward lower specific electrical conductivity after PEPA addition may be related to changes in the state of polar film-former groups and in the mobility of charged species as a result of intermolecular interactions with protonated PEPA amino groups. The results are consistent with the possibility of heteroassociative interactions between PEPA and the hydrophilic fragments of both film formers [18].

The change in porosity of the formed coatings reflects the effect of PEPA on the structural organization of the film-forming matrix. In the absence of PEPA, the porosity of the polyurethane coating was 73.70%, whereas that of the acrylic-polyurethane coating was 37.69% (Figure 6). The lower initial porosity of the acrylic-polyurethane coating may be related to the chemical composition of the hybrid polymer system and to conditions favoring formation of a more homogeneous film structure [19].

**Figure 6** – Porosity of coatings based on acrylic-polyurethane (1) and polyurethane (2) film formers as a function of PEPA concentration.

Replicate determinations confirmed the reproducibility of the observed concentration dependence of porosity. For polyurethane coatings, RSD values ranged from 3.27 to 5.89%. For acrylic-polyurethane coatings, the relative scatter increased as the absolute porosity decreased, reaching its maximum value at the minimum porosity of 4.62%.

As the additive concentration increased from 0 to 0.0024 mol/dm³, the porosity of the polyurethane coating decreased from 73.70 to 40.56%. This

decrease corresponded to the maximum pH value of 10.41 and the minimum specific electrical conductivity of 1.26 mS/cm. For the acrylic-polyurethane coating, the decrease was more pronounced: porosity decreased from 37.69 to 4.62%, while the corresponding pH and specific electrical conductivity values were 11.08 and 2.57 mS/cm, respectively.

Thus, addition of PEPA was accompanied by a decrease in the porosity of both coatings, with the strongest effect observed for the acrylic-polyurethane system. The change in porosity, together with changes in pH, electrical conductivity, and surface tension, is consistent with possible involvement of intermolecular interactions between PEPA and polar groups of the film former in the formation of a less porous coating with a porosity of 4.62%.

4. Conclusion

The «water-PEPA» system exhibited concentration-dependent changes, with the region near $C_{PEPA}=0.0012$ mol/dm³ interpreted as the critical association concentration based on concurrent changes in surface tension, molar conductivity, and pH. Increasing PEPA from 0.0004 to 0.0024 mol/dm³ reduced molar conductivity from 442.5 to 153.2 S·cm²/mol. The modifier affected the two film formers differently: the acrylic-polyurethane system showed the stronger response, including a decrease in surface tension from 39.60 to 35.15 mN/m. PEPA also reduced coating porosity from 73.70 to 40.56% for polyurethane and from 37.69 to 4.62% for acrylic-polyurethane. These results are consistent with possible intermolecular interactions between PEPA and polar groups of the film formers and support its use for controlling the surface and structural properties of waterborne coating systems.

Conflict of Interest: The authors declare no conflict of interest.

ҚАБЫҚ ТҮЗГІШТЕРДІҢ СУЛЫ ДИСПЕРСИЯЛАРЫНДАҒЫ ПОЛИЭЛЕКТРОЛИТ ӨЗАРА ӘРЕКЕТТЕСУІНІҢ ФИЗИКА-ХИМИЯЛЫҚ АСПЕКТІЛЕРІ

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Түйіндемe. *Kіріспе.* Сулы-дисперсиялық лак-бояу материалдары экологиялық және технологиялық артықшылықтарға ие, алайда олар түзетін жабындар құрылымдық біртексіздікпен және жоғары кеуектілікпен сипатталуы мүмкін. Мұндай жүйелердің қасиеттерін реттеу тәсілдерінің бірі функционалдық модификаторларды енгізу болып табылады. *Жұмыстың мақсаты.* Полиэтиленполиаминнің сулы полиуретандық және акрил-полиуретандық дисперсиялардың беттік және көлемдік қасиеттеріне, сондай-ақ олар түзетін жабындардың кеуектілігіне әсерін анықтау. *Әдістеме.* Құрамында 0-0,0024 моль/дм³ ПЭПА бар жүйелерде 293 К температурада беттік керілу, меншікті және молярлық электрөткізгіштік, рН және жабындардың кеуектілігі анықталды. *Нәтижелер және талқылау.* «Су-ПЭПА» бинарлық жүйесінде беттік керілу, молярлық электрөткізгіштік және рН концентрациялық тәуелділіктерінің сипатының өзгеруі $C_{ПЭПА}=0,0012$ моль/дм³ аймағында байқалды, бұл ассоциацияның критикалық концентрациясына сәйкес келеді. Үш компонентті жүйелерде зерттелген көрсеткіштердің өзгеру сипаты қабық түзгіштердің табиғатына тәуелді болды; беттік керілудің ең айқын төмендеуі акрил-полиуретандық дисперсия үшін байқалды – 39,60-тан 35,15 мН/м-ге дейін. ПЭПА концентрациясының

жоғарылауымен полиуретандық жабынның кеуектілігі 73,70-тен 40,56%-ға дейін, ал акрил-полиуретандық жабынның кеуектілігі 37,69-дан 4,62%-ға дейін төмендеді. Қорытынды. Алынған нәтижелер ПЭПА-ның зерттелген сулы дисперсиялардың беттік және көлемдік қасиеттеріне және түзілетін жабындардың құрылымына әсер ететінін көрсетеді. Байқалған өзгерістер ПЭПА-ның зерттелген жүйелер компоненттерімен ассоциативтік өзара әрекеттесу мүмкіндігімен үйлеседі; модификациялаудың ең айқын әсері акрил-полиуретандық композиция үшін анықталды.

Түйінді сөздер: акрил-полиуретан, полиуретан, беттік және көлемдік қасиеттер, полиэтиленполиамин, қабық түзгіштердің сулы дисперсиялары

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ФИЗИКО-ХИМИЧЕСКИЕ АСПЕКТЫ ВЗАИМОДЕЙСТВИЯ ПОЛИЭЛЕКТРОЛИТА В ВОДНЫХ ДИСПЕРСИЯХ ПЛЕНКООБРАЗУЮЩИХ

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Резюме. Введение. Водно-дисперсионные лакокрасочные материалы обладают экологическими и технологическими преимуществами, однако формируемые ими покрытия могут характеризоваться структурной неоднородностью и повышенной пористостью. Одним из подходов к регулированию свойств таких систем является введение функциональных модификаторов. *Цель* настоящей работы – определить влияние полиэтиленполиамин на поверхностные и объемные свойства водных полиуретановых и акрил-полиуретановых дисперсий, а также пористость формируемых ими покрытий. *Методология.* При 293 К в системах, содержащих 0-0,0024 моль/дм³ ПЭПА, определяли поверхностное натяжение, удельную и молярную электропроводность, pH, пористость покрытий. *Результаты и обсуждение.* В бинарной системе «вода-ПЭПА» изменение характера концентрационных зависимостей поверхностного натяжения, молярной электропроводности и pH наблюдалось в области $C_{\text{ПЭПА}}=0,0012$ моль/дм³, соответствующей критической концентрации ассоциации. В тройных системах характер изменения исследуемых показателей зависел от природы пленкообразующих; наиболее выраженное снижение поверхностного натяжения наблюдалось для акрил-полиуретановой дисперсии – с 39,60 до 35,15 мН/м. При увеличении концентрации ПЭПА пористость полиуретанового покрытия снизилась с 73,70 до 40,56%, а акрил-полиуретанового – с 37,69 до 4,62%. *Заключение.* Полученные результаты свидетельствуют о влиянии ПЭПА на поверхностные и объемные свойства исследуемых водных дисперсий и структуру формируемых покрытий. Наблюдаемые изменения согласуются с возможностью ассоциативных взаимодействий ПЭПА с компонентами исследуемых систем, наиболее выраженный эффект модифицирования установлен для акрил-полиуретановой композиции.

Ключевые слова: акрил-полиуретан; полиуретан; поверхностные и объемные свойства; полиэтиленполиамин; водные дисперсии пленкообразующих

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