Chemical Journal of Kazakhstan
Volume 2, Number 78(2022), 99-108 https://doi.org/10.51580/2022-2/2710-1185.69

УДК 541,183

INVESTIGATION OF ORGANIC AMINE DERIVATIVE ADSORPTION AT THE INTERFACE OF PIGMENT AND AIR

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Abstract: in systems based on bituminous film-forming agents, the adsorption of a surfactant AC-1 at the interface with air and aluminum powder has been studied. The effect of temperature, quantitative contents of the film-forming and initial surfactant concentration on the parameters and mechanism of AC-1 concentration on the interface has been established. The equilibrium indicators of the adsorption of surfactant AC-1 at the interface between the film-forming agent and air were determined (limiting adsorption Γ_∞, thickness of the adsorption layer δ of the surfactant AC-1 group, landing area S of the functional group of AC-1 surfactants). A decrease in Γ_∞in 1.5-2.0 times ($0.91 \cdot 10^{-5}$mol/m^2 with $\hat{C}_s = 1.78$) and δ ($2.80 \cdot 10^{-9} \div 3.00 \cdot 10^{-9}$ m) with an increase in temperature from 280 to 310K and solvent concentration up to $\hat{C}_s = 1.78$. The area S of the occupied amino group in the adsorption layer was $8.50 \cdot 10^{-20} \div 10.00 \cdot 10^{-20}$ m^2. The value of the resulting thermal effect (ΔH) for bituminous compositions with $\hat{C}_s = 1.17 \div 1.78$, regardless of the surfactant content, is plus 38-40 kJ/mol;for compositions with a solvent content of 1.78 ($\hat{C}_{SAA} = 0.06$) with an increase in temperature from 300 to 313 K, there was a decrease in the resulting adsorption index by $1.0 \cdot 10^{-6}$mol/m^2 and ΔH to a level of 20÷1.5 kJ/mol^2.A proportional increase in the adsorption of AC-1 on aluminum powder from the initial concentration of surfactants and an insignificant decrease in the adsorption index in systems more concentrated in bitumen were established.

Key words: adsorption, pigment, aluminum powder, bitumen, destructuring of bitumen compositions, modeling, thickness of the adsorption layer.

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Citation: Dyuryagina A.N., Kozik D.YU., Degert A.I., Lezhneva M.YU. Investigation of organic amine derivative adsorption at the interface of pigment and air. *Chem. J. Kaz.*, 2022, *2(78)*, 99-108. DOI: https://doi.org/10.51580/2022-2/2710-1185.69

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1.Introduction

The study of the surface-active properties of low-molecular surfactants in combination with oligomers (polymers) belongs to the modern, new direction of colloidal chemistry, which has been formed in recent years [1-3]. Hence, the information in the scientific literature is largely contradictory and applies mainly to aqueous solutions.

Meanwhile, it is known that the adsorption processes for surfactants in non-polar hydrocarbon media are in many respects the opposite, in comparison with aqueous media. This is evidenced by a decrease in the surface activity of amphiphilic compounds with an increase in the length of the hydrocarbon radical (inversion of the Duclos-Traube rule).

Undoubtedly, the determination of the processes of adsorption of surfactants introduced into paint and varnish compositions is not only of theoretical interest, but also practical, since it is the basis for optimizing the composition of paintwork materials and the subsequent improvement of the quality of coatings based on them. In this regard, it seemed appropriate to investigate the effect of the nature of film-forming on the surface activity of the low-molecular-weight amino derivative AC-1, synthesized in the laboratory of the PChRMNKSU [4], at the interface with air.

2.Experimental part

When studying the adsorption of AC-1 (TU 655-RK 05606434-001-2000), bitumen (ORB 90/130) was used as a film-forming agent, and white spirit was used as a solvent (GOST 3134-78).

The effect of the concentration of AC-1 (C, mol/dm^{3}1.2·10^{-3}-1.0 · 10^{-2}), temperature (T, K: 283÷313) and bitumen content (C_{Bit},% 20÷30) for bitumen compositions based on white spirit solvent. The development of the adsorption process was monitored by measuring the surface tension of the liquid phase using a Rebinder device.

The development of adsorption processes was established in suspensions at a fixed pigment content (0.2 g). The duration of the experiments was limited to 40 minutes, which was sufficient to achieve adsorption equilibrium. Upon completion of the experiments, the suspensions were separated by centrifugation (in a thermostatted mode) and the surface tension in the liquid phase was measured by the method of the highest pressure of air bubbles [5]. The amount of adsorbed surfactant (Γ, mol/m^2) was determined from the previously obtained calibration dependences $\sigma = f(\hat{C}_{SAA})$.

3. Results

The results of the AC-1 adsorption study are presented systematically.

1. *System «bitumen-white-spirit-AC-1»*

In the initial sections of the isotherms (Figure 1) at 293 K, the adsorption of the AC-1 surfactant ($\Gamma_{l\text{-}g}$) is satisfactorily described by the dependence

$$\Gamma_{\mathbf{l\text{-}g}} = \mathbf{(}\hat{C}_{SAA}(40 + 15\cdot\mathbf{(}\hat{C}_{\mathbf{s}}\mathbf{\text{-}}0.94)^2) \quad (1)$$

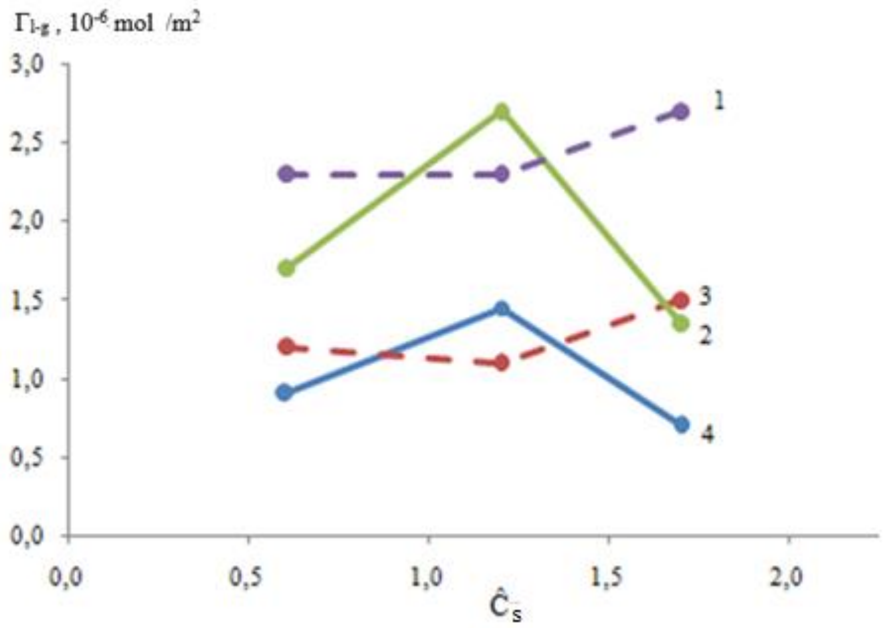
For systems with a solvent content of not more than 60%, the $\Delta\Gamma/\Delta\hat{C}_{SAA}$index remains practically unchanged and amounts to $3.7\cdot10^{-5}$mol/m^2; at the same time, with a further increase in the solvent content, this indicator significantly increases and amounts to $4.3\cdot10^{-5}$mol/m^2.

At low (not more than 293 K) temperatures (Figure 1), an increase in C_s to 1.17 practically does not affect the adsorption indicators (**$\Gamma_{l\text{-}g}$**), the values of which stabilize at $\hat{C}_{SAA} = 0.03$ and $\hat{C}_{SAA} = 0.06$ at the level of $1.25\cdot10^{-6}$ and $2.42\cdot10^{-6}$mol/m^2 and characterize exclusively the adsorption of AC-1. The development at T ≈ 293 K for dilute bitumen systems ($\hat{C}_p > 1.17$) destructuring processes is accompanied by an increase in the adsorption value by the amount of surfactants delocalized from bitumen. Thus, the increase in **$\Delta\Gamma_{l\text{-}g}$** at T = 293 K and $C_s = 1.78$ due to surfactants released from bitumen, at $\hat{C}_{SAA}$ = 0.03 and SAA = 0.06, was $0.28\cdot10^{-6}$ and $0.45\cdot10^{-6}$mol/m^2.

With an increase in temperature to 300÷313 K, a similar effect manifested itself only at $\hat{C}_s$ less than 1.17. The degree of concentration of surfactants that make up the bitumen at the interface in this case is much higher (approximately 2-2.5 times), which is explained by more quantitative destructuring and destruction of intra- and intermolecular associates, and as a consequence - an increase in the equilibrium concentrations of unbound surfactants in near-surface films.

Thus, it can be concluded that the presented indicators of adsorption of $\Gamma_{l\text{-}g}$ are an integral value that takes into account the contribution of two active components – the introduced AC-1 and surfactants, released in the process of destructuring bituminous systems. The above-mentioned regularities are also typical for compositions concentrated in bitumen ($\hat{C}_s = 0.54$), which is confirmed by the curves shown in Figure 2.

A decrease in Γ with increasing temperature indicates that the fixation of AC-1 at the interface with air is carried out by physical forces. As bitumen is diluted with white spirit, up to a concentration of the latter of 70 %, with an increase in temperature to 300 K, the surfactant content in the adsorption layer increases. An increase in adsorption in this area is achieved due to surfactants that are part of the bitumen, as indicated above. So, with $\hat{C}_s = 1.17$ and the content$\hat{C}_{SAA}$ = 0.06, with an increase in temperature from 283 to 300 K, the increase in the adsorption index was 1.25-10^{-6} mol/m^2, which is 2 times higher than at T = 283 K. A further increase in temperature at a given solvent content has practically no effect on the $G_{l\text{-}g}$ value. Based on this, it can be concluded that for this bitumen composition, the limiting temperature that ensures complete destructuring is 299-301 K.

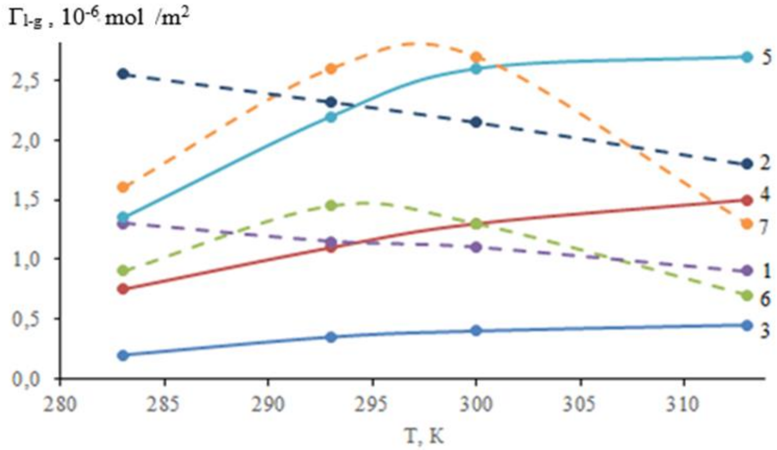


1 – $\hat{C}_{SAA}$= 0.06, T = 293К; 2 – $\hat{C}_{SAA}$= 0.06, T = 313 К;
3 – $\hat{C}_{SAA}$= 0.03, T = 293 К;4 – $\hat{C}_{SAA}$= 0.03, T = 313 К.

Figure 1 – Influence of solvent concentration on the surfactant adsorption

The limiting role of the destructuring processes of bituminous compositions, characterized by additional energy consumption (endothermic effect), on the adsorption index is also evidenced by the values of the calculated heats of adsorption ΔH, which for bitumen compositions with $\hat{C}_s$ = 1.17÷1.78, regardless of the surfactant content, are plus 38-40 kJ/mol.

At the same time, for compositions with a solvent content of 1.78 ($\hat{C}_{SAA}$ = 0.06) with an increase in temperature from 300 to 313 K, a decrease in the resulting adsorption index by $1.0 \cdot 10^{-6}$mol/m^2 was noted. Obviously, this value of the decrease in the adsorption index corresponds to the amount of desorbed AS-1 from the interface, bound by the forces of physical adsorption (ΔH ≈ (20÷1.5) kJ/mol^2).



1 – $\hat{C}_p$ = **0**.54, $\hat{C}_{SAA}$ = 0.03; 2 – $\hat{C}_p$ = **0**.54, $\hat{C}_{SAA}$ = 0.06; 3 – $\hat{C}_p$ = 1.17, $\hat{C}_{SAA}$ = **0.01;**
4 – $\hat{C}_p$ = 1.17, $\hat{C}_{SAA}$ = 0.03; 5 – $\hat{C}_p$ = 1.17, $\hat{C}_{SAA}$= 0.06; 6 – $\hat{C}_p$ = 1.78, $\hat{C}_{SAA}$= 0.03;
7 – $\hat{C}_p$ = 1.78, $\hat{C}_{SAA}$ = 0.06.

Figure 2 – Influence of solvent concentration on the surfactant adsorption

It has been established (Table 1) that an increase in temperature and solvent concentration in bitumen compositions leads to an intensification of the vibrational component of surfactant molecules, concentrated in the adsorption layer, and, as a consequence, to a decrease in their distribution density and a decrease in the values of Γ_∞.

Table 1 – Equilibrium characteristics of the adsorption of surfactant AC-1 depending on the temperature and composition of bitumen mixtures

T, K	**283**			**293**			**300**			313		
$\hat{C}_p$	1.78	1.17	0.54	1.78	1.17	0.54	1.78	1.17	0.54	1.78	1.17	0.54
$\Gamma_\infty \cdot 10^5$,**mol/m²**	2.13	2.25	3.08	1.75	1.95	2.77	1.34	1.**66**	2.79	0.91	1.29	1.95
$S\ 10^{-20}$,**m²**	7.79	7.40	5.39	9.43	8.51	5.99	12.4	10.0	5.95	18.2	12.9	8.51
$\delta \cdot 10^9$, m	3.87	4.09	5.60	3.18	3.55	5.04	2.44	3.02	5.07	1.65	2.35	3.55

With an increase in temperature from 280 to 310 K, the limiting adsorption index decreased 1.5-2.0 times and at $\hat{C}_s$ 1.78 and 0.74, respectively, $0.91\text{-}10^{-5}$ and $1.95\text{-}10^{-5}$ mol/m². A similar effect was noted for compositions with a high bitumen content. As the specific content of white spirit increases from 0.54 to 1.78 (300 K), accompanied, as a result, by a decrease in the viscosity of the system, the limiting adsorption index Γ_∞ decreases approximately 2 times and does not exceed $1.35\text{-}10^{-5}$ mol/m²

The values of the areas (landing area according to [6]) attributable to active functional groups of surfactants (amino groups), equal to $8.5\cdot10^{-20}$-$13.0\cdot10^{-20}$ m^2, coincide with the values calculated by the geometric model of Stuart-Brigleb , and taking into account the molecular-kinetic features with the experimental data presented in the literature [7-17].

As the temperature of the compositions decreased and the content of bitumen in them increased (Table 1), in the region of maximum adsorption, a decrease in the area attributable to the polar groups of surfactants was noted, which is due to the predominance of structured complexes in the surface films and the lack of kinetic activity of surfactant molecules.

So, for example, with an increase in temperature from 293 K to 313 K, at $\hat{C}_s = 1.17$, the S values increase from $8.51\text{-}10^{-20}$ to $12.90\text{-}10^{-20}$ m^2, the same indicator at $\hat{C}_s = 0.54$ and other things being equal, varies from $5.99\cdot10^{-20}$ to $8.51\cdot10^{-20}$ m^2.

According to the data presented (Table 1), the thickness of the adsorption layer naturally decreases with increasing temperature and solvent content in the bitumen composition. The nature of the change in the thickness of the adsorption layer δ ($1.5\cdot10^{-9}$-$5.6\cdot10^{-9}$ m) from the temperature and concentration of bitumen is also consistent with the change in the physical properties of the system; as the viscosity and density of the compositions used decrease, δ regularly decreases.

Taking into account the presented physicochemical regularities and characteristics, it can be concluded that the main provisions of the theory of monomolecular adsorption are applicable to the processes developing at the "bitumen composition-air" interface with the participation of the AC-1 surfactant.

2. System «bitumen-white-spirit-pigment-surfactant-AC-1»

An experimental study on the adsorption of AC-1 on aluminum powder was carried out in bitumen compositions with different bitumen content (Figure 3): 20 and 30 %.

For bitumino us compositions, a proportional increase in the adsorption index of AC-1 was not ed with an increasein the concentration of surfactants.

The generalized equation reflecting the combined effect of the initial concentrations of surfactant and bitumen in the studied compositions on the adsorption rate of AC-1 has the form:

$$\Gamma_1=[4.76 - 0.05\cdot(C_{bit} - 20)]\cdot C_{SAA} \quad (2)$$

where, C_{Bit} - bitumen concentration,%;

C_{SAA}–AC-1 surfactant concentration, mol/dm^3.

As the temperature rises to 295 K, the adsorption index on the aluminum powder decreases, however, with a further increase in temperature, this index stabilizes at $6.49\cdot10^{-3}$mol/g.

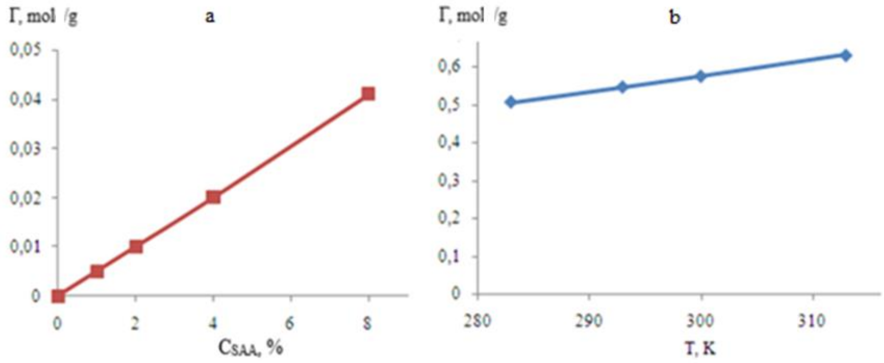


Figure 3 – Partial dependences of the adsorption of surfactant AC-1 from the solvent on the surface of aluminum powder on the initial concentration (a) and temperature (b)

$$\Gamma_2=7.69\cdot 10^{-6}\cdot T^2 - 4.28\cdot 10^{-4}\cdot T + 0.012 \qquad (3)$$

The resulting thermal effect was ΔH = -18 kJ/mol, which confirms the preference of fixing the amino-containing surfactant on the aluminum powder in this film-forming agent.

4. Conclusions

Generalized correlation equations G = ƒ(σ) have been obtained, which make it possible to establish a mutual relationship between the surface tension indicators and the nature of the distribution of AC-1 surfactants in the bulk and surface phases, as well as with operating parameters (temperature, surfactant concentration, composition of the bitumen composition). For the first time, the equilibrium indicators of the adsorption of surfactant AC-1 at the interfaces between the phases of the film-forming agent and air were determined (maximum adsorption Γ_∞, thickness of the adsorption layer δ, landing area S of the functional group of surfactant AC-1, The area S occupied by the amino group in the adsorption layer was $8.50\cdot 10^{-20}\div 10.00\cdot 10^{-20}$ m^2.

The limiting role of the processes of destructuring of bituminous compositions, characterized by additional energy consumption (endothermic effect), on the adsorption rate has been established. The calculated heat effects of adsorption ΔH for bitumen compositions with $\hat{C}_s$ = 1.17÷1.78, regardless of the surfactant content, are plus 38-40 kJ/mol. For compositions with a solvent content of 1.78 ($\hat{C}_{SAA}$ = 0,06) with an increase in temperature from 300 to 313 K, there was a decrease in the resulting adsorption index by $1.0\cdot 10^{-6}$mol/m^2 and ΔH to a level of 20÷1.5 kJ/mol^2.

A proportional increase in the adsorption of AC-1 on aluminum powder from the initial concentration of surfactants and an insignificant decrease in the adsorption index in systems more concentrated in bitumen were established.

The work was carried out with the financial support of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AP08856284).

ПИГМЕНТПЕН ЖӘНЕ АУА МЕН ИНТЕРФАЗАЛЫҚ ШЕКАРАЛАРДА ОРГАНИКАЛЫҚ АМИН ТУЫНДЫСЫНЫҢ АДСОРБЦИЯСЫН ЗЕРТТЕУ

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Түйіндеме. Битумды пленка түзуші жүйелерде ауамен алюминий ұнтағымен фаза аралық шекараларда АС-1 беттік-белсенді заттың (ББЗ) адсорбциясы зерттелді. Беттік-белсенді заттардың температурасын, пленканы құрайтын сандық құрамын және бастапқы концентрациясын фаза аралық бетке АС-1 концентрациясының көрсеткіштерімен механизміне әсері анықталды. Үлдір түзгіштің ауамен бөлінушекарасында АС-1 ПБЗ адсорбциясының тепе-теңдік көрсеткіштері анықталды (шекті адсорбция Γ_∞, адсорбциялық қабаттың қалыңдығы δас-1 ББЗ тобы, АС-1 ББЗ функционалдық тобының S қонуалаңы). Γ_∞ 1,5-2,0 есе ($0{,}91\cdot10^{-5}$моль/м^2 C_p=1,78 кезінде) және δ ($2{,}80\cdot10^{-9}\div3{,}00\cdot10^{-9}$ м) температураның 280-ден 310 К-ге дейін жоғарылауымен және еріткіштің концентрациясы C_p=1,78 дейін. Адсорбциялық қабаттағы амин тобыалатын S аудандарының мәні $8{,}50\cdot10^{-20}\div10{,}00\cdot10^{-20}$ м^2 құрады. ББЗ құрамына қарамастан, с C_p = 1,17÷1,78 битумдық композициялар үшін алынған жылу әсерінің мәні (ΔН) плюс 38-40 кДж/моль құрайды; құрамында 1,78 ($C_{пав}$ = 0,06) еріткіші бар құрамдарүшін температураның 300-ден 313 К-ге дейін жоғарылауы кезінденәтижесінде адсорбцияның көрсеткішінің $1{,}0\cdot10^{-6}$моль/м^2 және ΔН 20÷1,5 кДж/моль^2 деңгейіне дейін төмендеуі байқалды. Алюминий ұнтағындағы АС-1 адсорбциясының ББЗ бастапқы концентрациясынан пропорционалды ұлғаюы және битум бойынша неғұрлым шоғырланған жүйелердегі адсорбция көрсеткішінің шамалы төмендеуі анықталды.

Түйінді сөздер: адсорбция, пигмент, алюминий ұнтағы, битум, битум композицияларын бұзу, модельдеу, адсорбция қабатының қалыңдығы.

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ИЗУЧЕНИЕ АДСОРБЦИИ ОРГАНИЧЕСКОГО АМИНОПРОИЗВОДНОГО НА МЕЖФАЗНЫХ ГРАНИЦАХ С ПИГМЕНТОМ И ВОЗДУХОМ

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Резюме. В системах на основе битумных пленкообразующих изучена адсорбция поверхностно-активного вещества (ПАВ) АС-1 на межфазных границах с воздухом и алюминиевой пудрой. Установлено влияние температуры, количественных содержаний пленкообразующего и исходной концентрации ПАВ на показатели и механизм концентрирования АС-1 на межфазной поверхности. Определены равновесные показатели адсорбции ПАВ АС-1 на границе раздела пленкообразователя с воздухом (предельная адсорбция Γ_∞, толщина адсорбционного слоя δ группы ПАВ АС-1, посадочная площадь S функциональной группы ПАВ АС-1). Выявлено уменьшение Γ_∞ в 1,5-2,0 раза ($0{,}91\cdot10^{-5}$ моль/м^2 при Ср = 1,78) и δ ($2{,}80\cdot10^{-9}\div3{,}00\cdot10^{-9}$ м) с увеличением температуры от 280 до 310 К и концентрация растворителя до Ср=1,78. Значение площадей S занимаемой аминогруппой в адсорбционном слое составили $8{,}50\cdot10^{-20}\div10{,}00\cdot10^{-20}$ м^2.
Значение результирующего теплового эффекта (ΔH) для битумных композиций с Ср = 1,17÷1,78, независимо от содержания ПАВ, составляют плюс 38-40 кДж/моль; для составов с содержанием растворителя 1,78 ($C_{пав}$ = 0,06) при увеличении температуры от 300 до 313 К, отмечалось снижение результирующего показателя **адсорбции на** $1{,}0\cdot10^{-6}$ моль/м^2 и ΔH до уровня 20÷1,5 кДж/моль^2.
Установлено пропорциональное увеличение адсорбции АС-1 на алюминиевой пудре от исходной концентрации ПАВ и незначительное снижение показателя адсорбции в более концентрированных по битуму системах.

Ключевые слова: адсорбция, пигмент, алюминиевая пудра, битум, деструктурирование битумных композиций, моделирование, толщина адсорбционного слоя.

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