

UDC 661.185

SYNTHESIS AND STUDY INTO COLLOID-CHEMICAL PROPERTIES OF SURFACTANTS ON THE BASIS OF PALM OIL AND ETHANOLAMINES**I.T.Ismayilov**

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Anionic surfactants Na^+ , K^+ , NH_4^+ , $-\text{NH}_2^+-\text{CH}_2-\text{CH}_2-\text{OH}$ and $-\text{NH}^+(\text{CH}_2-\text{CH}_2-\text{OH})_2$ and salts of sulphate-derivatives of higher carboxylic acids have been synthesized. Sulphate-derivatives have been obtained by the interaction of palm oil with ethanolamines (monoethanolamine and diethanolamine) at molar ratio 1:2 followed by modification of the reaction products by sulphuric acid. Basic colloid-chemical properties of synthesized salts and their oil-collecting and oil-dispersing properties on water surface with different degree of mineralization analyzed.

Keywords: *palm oil, ethanolamines, interfacial tension, critical micelle concentration, free adsorption energy*

Introduction

Surfactants are most widely used in practically all fields of industry and welfare, too [1, 2]. Application of surfactants can influence on energy state and structure of boundary surface and control the properties of heterogeneous systems through it. Due to its cleansing, moisturizing, dispersing, foaming, and many other properties, they have become the products of choice in many fields of application in oil industry. They are used for drilling, corrosion inhibition of oilfield equipment, dehydration of oil, and added to lubricating oils as additives, etc. Possibility of application of surfactants as corrosion inhibitors have been thoroughly examined on a large scale [4]. Various surfactant mixtures, as well as surfactants of multifunctional purposes are known to be applied in resolving a number of problems through the use of their properties. [3-6]. Hence, selection of a suitable product should be made with due regard for appropriate conditions and product assignment.

Recently, environmental standards conformably to industrial consumption and wider use of new corrosion inhibitors have been applied in industry.

Contemporary hygienic and environ-

mental standards provide for replacement of corrosion inhibitors by toxic reagents of the same efficiency with maximum safety for industry and everyday practice. Besides, of great importance is the use of available and inexpensive raw materials for preparation of inhibitors to promote search for new perspective technologies and initial products.

Note that main raw materials for production of surfactants are oil and gas refining products, as well as products of vegetable origin [7-9]. Benefits of vegetable raw materials are, first of all, their renewability and greater ecological safety. Currently, high emphasis is placed on these factors due to the development of "green chemistry". Data given in [10-13], deal with obtaining and analysis of the properties of anionic surfactants synthesized on the basis of triglycerides of some vegetable oils. According to their renewability and ecological acceptability, they may be applied as perspective raw materials for the production of reagents in oil industry.

The paper focuses on the results of obtaining anionic surfactants on the basis of palm oil to comply with their colloid-chemical properties.

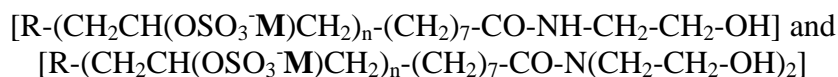
Experimental part

Sulphate-derivatives of higher carboxylic acids have been synthesized on the basis of palm oil by aminolysis and sulphation.

Palm oil aminolysis has been carried out in the presence of monoethanolamine (MEA) and diethanolamine (DEA) at molar ratio of

initial components of palm oil as follows: ethanolamine = 1:2.

According to the reaction scheme published in [12], anionic surfactant salts of below-given structures have been obtained by the interaction of bases with sulphate-derivatives of higher carboxylic acids:



where $n = 1-3$; $\text{M} - \text{Na}^+, \text{K}^+, \text{NH}_4^+, -\text{NH}_2^+-\text{CH}_2-\text{CH}_2-\text{OH} (\text{M}\ddot{\text{A}}^+) \text{ и } -\text{NH}^+(\text{CH}_2-\text{CH}_2-\text{OH})_2 (\text{DEA}^+)$.

The salts are identified as: PM_I , PM_II , PM_III , PM_IV , PM_V , PD_I , PD_II , PD_III , PD_IV and PD_V , where P is the index of sunflower oil, M – aminolysis of palm oil carried out in the presence of MEA, D – aminolysis of palm oil carried out in the presence of DEA, I- Na^+ , II- K^+ , III- NH_4^+ , IV- MEA^+ , V- DEA^+ . Basic physicochemical properties of intermediate and end-products of the reaction, as well as the compound and structure as set forth by IR-spectroscopy have been presented in [12].

Surface activity of obtained salts has been analyzed on water-kerosene interface by stalagmometric method in the work. As is known, the interfacial tension at this phase boundary in the absence of surfactants at 22-24°C amounts to 46.0-46.5 mN/m. Note that obtained data on surface activity of salts at 22°C are presented in Table 1. Interfacial tension in the presence of PD_II on the basis of K^+ -salts of sulphate-derivatives of higher carboxylic acids synthesized on the basis of palm oil and DEA at 0.175 wt. % concentration leads to the reduction of interfacial tension on water-kerosene interface down to 1.60 mN/m which evinces maximum surface activity among salts previously synthesized in the concentration of 0.005-0.2 wt. %.

As a result of stalagmometric measurements, isotherms of interfacial tension

have been formed (Fig. 1 and 2.) where the dependence of interfacial tension on water-kerosene interface in the surfactant concentration is given by coordinates $\sigma - \ln C$. As is seen from the graph, interfacial tension decreases in the initial section with rise in salt concentration. That follows from adsorption process of surfactants at phase boundary and subsequent drop in the difference between polar liquid phases due to their confusion. The transition point to the horizontal portion of the isotherm corresponds to the critical micelle concentration. Research results reaffirm a fairly high surface activity of the salts.

According to the well-known theoretical formulas, some basic parameters of surfactants have been calculated on the basis of the data obtained and some thermo-dynamical parameters of micelle formation and adsorption processes. Calculated values of colloid-chemical parameters of the anionic surface active salts are presented in Table 2 where C_{CMC} - critical micelle concentration, σ_{CMC} - interfacial tension of the solution at CMC, π_{CMC} - surface pressure, Γ_m - maximum adsorption, A_o - minimum cross-sectional area of polar group, d - average length of the molecule, ΔG_{mic} - standard free energy of micelle - ΔG_{ads} free energy of adsorption.

Table 1. Results of stalagmometric studies into surfactants synthesized on the basis of palm oil and ethanolamines

Salt symbols	Salt concentrations in aqueous phase, wt. %										
	0.005	0.0075	0.01	0.025	0.05	0.075	0.1	0.125	0.15	0.175	0.2
	Interfacial tension on water-kerosene interface, mN/m (22°C)										
PM _I	39.36	35.75	33.15	25.52	18.69	14.82	11.64	9.13	6.42	4.34	4.34
PM _{II}	39.15	35.58	33.02	24.19	16.87	12.59	8.61	6.04	5.18	4.86	4.86
PM _{III}	32.28	27.96	25.06	17.48	12.55	9.70	7.53	6.91	6.87	6.87	6.87
PM _{IV}	27.19	23.53	20.97	13.98	8.50	6.21	4.43	3.10	3.10	3.10	3.10
PM _V	31.99	26.99	23.87	15.84	8.99	6.39	4.79	3.39	3.39	3.39	3.39
PD _I	40.40	35.86	32.89	23.76	17.52	13.85	11.06	8.61	6.04	4.12	4.12
PD _{II}	40.70	36.75	32.85	21.69	12.85	8.83	5.62	4.01	3.21	1.60	1.60
PD _{III}	30.29	26.88	24.94	19.10	14.08	11.51	9.59	8.54	8.26	8.26	8.26
PD _{IV}	31.56	27.12	24.61	17.13	11.32	8.94	6.14	5.12	4.86	4.86	4.86
PD _V	29.08	25.56	23.77	17.72	13.29	10.91	9.72	8.64	7.28	7.28	7.28

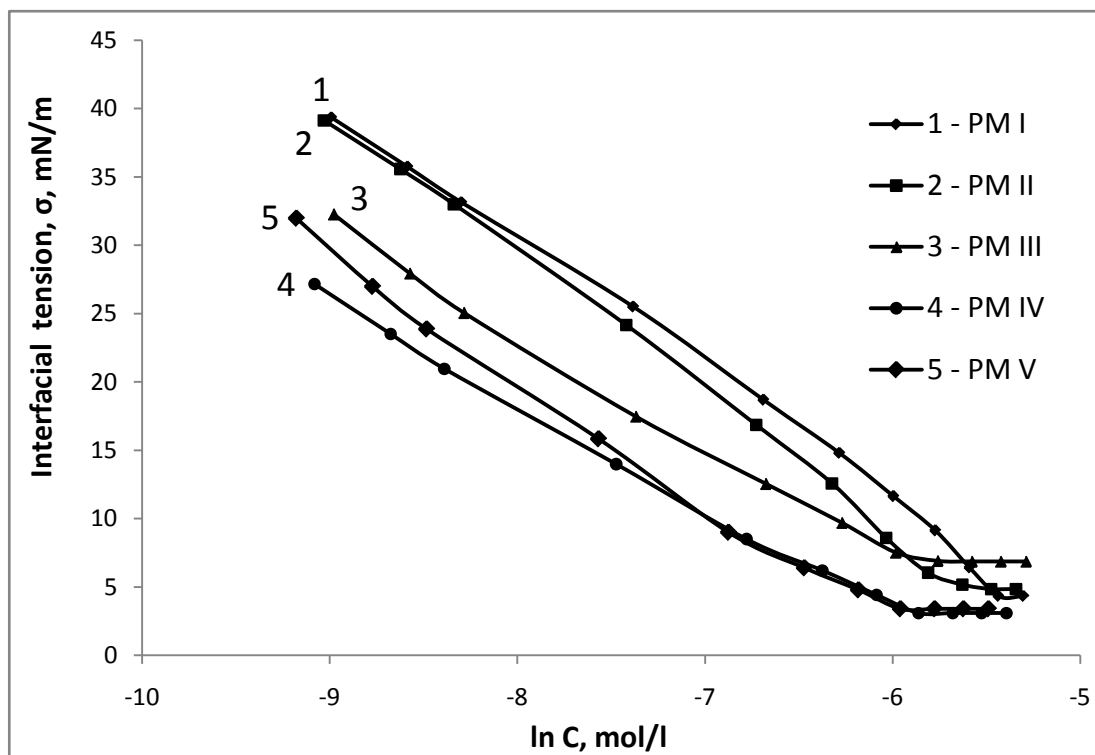


Fig. 1. Isotherms of interfacial tensions of surfactants synthesized on the basis of palm oil and monoethanolamine

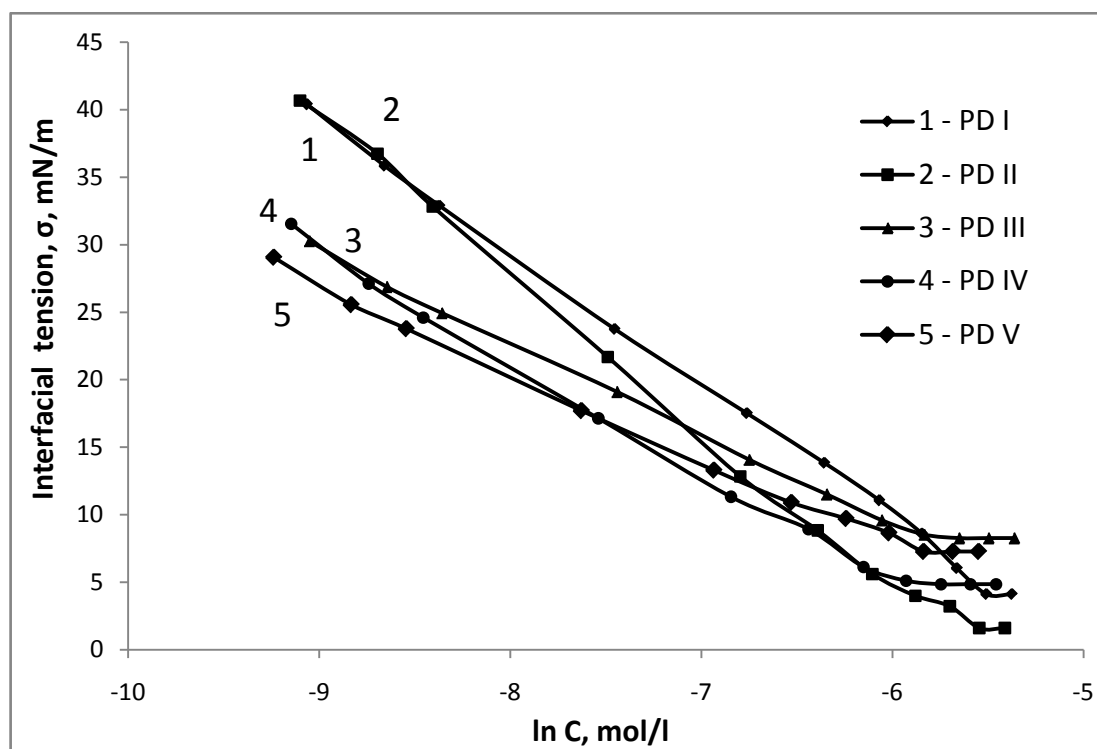


Fig. 2. Isotherms of interfacial tensions of surfactants, synthesized on the basis of palm oil and diethanolamine

Table 2. Colloid-chemical parameters of surfactants synthesized on the basis of palm oil and ethanolamines

Reagent	Average molecular weight, g/mol	Density ρ_{25}^{25} , g/cm ³	$C_{cmc} \times 10^3$, mol/l	$\text{Ln}(C_{cmc})$	σ_{cmc} , mN/m	π_{cmc} , mN/m	Q_c , mN/m	$\Gamma_m \times 10^{10}$, mol/cm ²	$A_g \times 10^3$, nm ²	d , nm	ΔG_{mic} , kJ/mol	ΔG_{nh} , kJ/mol
PM _I	402	1.24	4.35	-5.44	4.34	41.66	-9.29	3.79	43.8	1.23	-13.3	-24.3
PM _{II}	418	1.265	4.19	-5.48	4.86	41.14	-10.1	4.12	40.3	1.36	-13.4	-23.4
PM _{III}	397	1.2062	3.78	-5.58	6.87	39.13	-7.60	3.10	53.6	1.02	-13.7	-26.3
PM _{IV}	440	1.228	2.84	-5.86	3.1	42.9	-7.46	3.04	54.6	1.09	-14.4	-28.5
PM _V	484	1.231	2.58	-5.96	3.39	42.61	-8.85	3.61	46.0	1.42	-14.6	-26.4
PD _I	432	1.192	4.05	-5.51	4.12	41.88	-9.89	4.03	41.2	1.46	-13.5	-23.9
PD _{II}	448	1.195	3.91	-5.55	1.6	44.4	-11.32	4.62	36.0	1.73	-13.6	-23.2
PD _{III}	427	0.9987	3.51	-5.65	8.26	37.74	-6.59	2.69	61.8	1.15	-13.9	-27.9
PD _{IV}	470	1.162	3.19	-5.75	4.86	41.14	-7.94	3.24	51.3	1.31	-14.1	-26.8
PD _V	514	1.175	2.92	-5.84	7.28	38.72	-6.27	2.56	65.0	1.12	-14.3	-29.5

Table 3. Oil-collecting and oil-dispersing properties of 5% solutions of salts synthesized on the basis of palm oil and MEA

Reagent	Oil from "Pirallahy" oil-field				Balakhany oil				Naphthalane oil			
	Fresh water		Sea water		Fresh water		Sea water		Fresh water		Sea water	
	τ, h	K	τ, h	K	τ, h	K	τ, h	K	τ, h	K	τ, h	K
PM _I	0-24	11.1	0-24	16.7	0	11.1	0	11.1	0	4.9	0	6.3
	48	9.8	48-72	13.9	24	16.0	24	12.5	24	5.0	24	4.9
	72	8.7	96	13.4	48	13.7	48	10.4	48	5.3	48	4.3
	96	8.2			72	11.9	72	6.6	72	5.6	72	3.7
					96	11.1	96	6.3	96	5.7	96	3.3
PM _{II}	0	8.2	0	9.2	0	9.2	0	8.3	0	4.9	0	4.8
	24	11.1	0	11.1	24	25.0	24	25.0	24	2.8	24	3.2
	48	9.8	24-96	8.2	48	17.4	48	16.0	48	2.4	48	3.0
	72	8.7			72	9.8	72	12.8	72	2.2	72	2.6
	96	8.2			96	7.7	96	9.2	96	2.0	96	2.4
PM _{III}	0	11.1	0	13.3	0	9.5	0	89.7	0	8.3	0	8.3
	24	9.8	24	11.1	24	16.0	24	90.8	24	4.9	24	6.3
	48	9.2	48	5.9	48	14.8	48	4.2	48	4.2	48	4.0
	72	8.7	72	5.4	72	12.8	72	4.1	72	3.6	72	3.2
	96	8.2	96	4.9	96	11.1	96	4.0	96	3.3	96	2.8
PM _{IV}	0-24	8.2	0	93.4	0	8.2	0	93.5	0-24	6.3	0	11.1
	48	8.7	24	90.3	24	11.1	24	90.6	48	4.0	24	8.2
	72	9.8	48	4.0	48	9.8	48	83.7	72	2.8	48	5.7
	96	11.1	72	3.6	72	8.7	72	5.6	96	2.0	72	3.1
			96	3.3	96	8.2	96	4.2			96	2.0
PM _V	0	11.1	0	92.7	0	11.1	0	91.9	0	8.3	0	8.2
	24	16.7	24	90.8	24	16.0	24	92.5	24	11.1	24	11.1
	48	93.7	48	90.6	48	12.8	48	91.7	48	6.3	48	7.2
	72	92.7	72	89.6	72	9.8	72	90.6	72	4.0	72	4.2
	96	91.7	96	11.1	96	7.3	96	11.1	96	2.8	96	3.6

Table 4. Oil-collecting and oil-dispersing properties of 5% solutions of salts synthesized on the basis of palm oil and DEA

Reagent	Oil from "Pirallahy" oil field						Balakhany oil						Naphthalane oil										
	Fresh water			Sea water			Fresh water			Sea water			Fresh water			Sea water							
	τ, h	K		τ, h	K		τ, h	K		τ, h	K		τ, h	K		τ, h	K						
PD _I	0	11.1		0	10.3		0-24	16.0		0	16.0		0	8.2		0	6.7		0	8.2			
	24	20.7		24	11.3		48	18.9		24-48	17.4		24	2.8		24	2.6		24	20.7		24	8.3
	48-72	25.0		48	10.0		72	20.7		72-96	18.9		48	2.4		48	2.2		48	11.1		48	5.5
	96	20.7		96	3.5		96	22.7			18.9		96	9.2		96	1.8		96	12.8		96	1.6
PD _{II}	0-24	8.2		0	11.1		0-24	8.2		0	8.7		0	4.0		0	3.3		0	4.0		0	3.3
	48	6.3		24	9.8		48	11.1		24	25.0		24	2.8		24	2.6		24	2.8		24	2.6
	72	5.7		48-72	8.2		72	9.8		48	12.8		48	2.4		48	2.2		48	2.4		48	2.2
	96	4.9		96	6.3		96	8.2		96	11.1		96	1.9		96	1.8		96	2.2		96	2.0
PD _{III}	0	25.0		0	11.4		0	25.0		0	9.1		0	17.4		0	7.5		0	17.4		0	7.5
	24	30.9		24	15.1		24	39.0		24	10.7		24	20.7		24	10.0		24	20.7		24	10.0
	48-72	25.0		48	18.5		48	25.0		48	12.5		48	16.0		48	10.5		48	16.0		48	10.5
	96	18.9		96	15.9		96	17.4		96	13.2		96	14.8		96	8.7		96	14.8		96	8.7
PD _{IV}	0	14.8		0	11.1		0	17.4		0	16.7		0	8.2		0	13.9		0	8.2		0	13.9
	24	25.0		24	18.1		24	16.0		24	20.2		24	16.0		24	16.4		24	16.0		24	16.4
	48	17.4		48	16.7		48	11.1		48	14.3		48	11.1		48	14.3		48	11.1		48	14.3
	72-96	11.1		72	11.1		72	10.0		72	12.8		72	9.8		72	8.2		72	9.8		72	8.2
PD _V	0	14.8		0	12.5		0	16.0		0	11.8		0	6.2		0	9.5		0	6.2		0	9.5
	24	18.9		24	16.3		24	25.0		24	8.2		24	11.4		24	11.5		24	11.4		24	11.5
	48	14.8		48	22.2		48	18.9		48	16.7		48	11.1		48	5.8		48	11.1		48	5.8
	72-96	12.8		72	16.7		72	12.8		72	15.2		72	9.8		72	5.7		72	9.8		72	5.7
	96	11.1		96	15.2		96	11.9		96	8.7		96	8.7		96	4.9		96	8.7		96	4.9

Of great interest is the analysis of oil-collecting and oil-dispersing properties of synthesized salts of thin oil film on the surface of fresh and sea water. Laboratory tests went to show that synthesized salts of sulphate-derivatives of higher carboxylic acids display higher activity as oil-collecting and oil-dispersing reagents on the surface of sea and fresh water (Table 3 and 4). PD_{III} ($K_{max.} = 30.9$, $\tau = 24$ h) demonstrates maximum collecting effect of oil film from "Pirallahy" oil-field in fresh water through the use of 5% solutions of the reagents, true, in the sea water. PD_V ($K_{max.} = 22.2$, $\tau = 48$ h).

Among the reagents of mixed-type effect, dispersion is replaced by collecting which is observed in PM_{IV} ($K_d = 93.4\%$, $\tau = 0$ h; $K_d = 90.3\%$, $\tau = 24$ h; $K = 4.0$, $\tau = 48$ h; $K = 3.6$, $\tau = 72$ h; $K = 3.3$, $\tau = 96$ h) and PM_V ($K_d = 92.7\%$, $\tau = 0$ h; $K_d = 90.8\%$, $\tau = 24$ h; $K_d = 90.6\%$, $\tau = 48$ h; $K_d = 89.6\%$, $\tau = 72$ hours; $K = 11.1$, $\tau = 96$ h) in the sea water medium as well as mixed-type collecting effect which is replaced by dispersion at PM_V ($K = 11.1$, $\tau = 0$ h; $K = 16.7$, $\tau = 24$ h; $K_d = 93.7\%$, $\tau = 48$ h; $K_d = 92.7\%$, $\tau = 72$ h; $K_d = 91.7\%$, $\tau = 96$ h) in the fresh water medium.

PD_{III} reagent ($K_{max.} = 39.0$, $\tau = 24$ h) is also an effective collector of Balakhany oil film in the fresh water medium. It should be noted

that in the sea water medium maximum multiplicity of collection presents PM_{II} ($K_{max.} = 25.0$, $\tau = 24$ h) and PD_{II} ($K_{max.} = 25.0$, $\tau = 24$ h) reagents. Three reagents are synthesized on the basis of monoethanolamine - PM_{III} ($K_d = 89.7\%$, $\tau = 0$ h; $K_d = 90.8\%$, $\tau = 24$ h; $K = 4.2$, $\tau = 48$ h; $K = 4.1$, $\tau = 72$ h; $K = 4.0$, $\tau = 96$ h), PM_{IV} ($K_d = 93.5\%$, $\tau = 0$ h; $K_d = 90.6\%$, $\tau = 24$ h; $K_d = 93.7\%$, $\tau = 48$ h; $K = 5.6$, $\tau = 72$ h; $K = 4.2$, $\tau = 96$ h) and PM_V ($K_d = 91.2\%$, $\tau = 0$ h; $K_d = 92.5\%$, $\tau = 24$ h; $K_d = 91.7\%$, $\tau = 48$ h; $K_d = 90.6\%$, $\tau = 72$ h; $K = 11.1$, $\tau = 96$ h) firstly by dispersion which is followed by collecting of Balakhany oil film show mixed-type effects.

All of the reagents synthesized on the basis of palm oil have no dispersibility on Naphthalane oil both in fresh and sea water. Maximum multiplicity of collecting oil film is observed at PD_{III} reagents ($K_{max.} = 20.7$, $\tau = 24$ h) in the fresh water medium and PD_{IV} ($K_{max.} = 16.4$, $\tau = 24$ h) in the sea water medium.

Following the analysis results, one can conclude that, in the main, all of the reagents display sufficient high oil-collecting and oil-dispersing activity in removing off thin oil film from sea and fresh water surfaces. In some cases, a mixed-type effect is observed - collecting is replaced by dispersing and vice versa.

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СИНТЕЗ И ИССЛЕДОВАНИЕ КОЛЛОИДНО-ХИМИЧЕСКИХ СВОЙСТВ ПАВ НА ОСНОВЕ ПАЛЬМОВОГО МАСЛА И ЭТАНОЛАМИНОВ

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Синтезированы анионные поверхностно-активные Na^+ , K^+ , NH_4^+ , $-\text{NH}_2^+-\text{CH}_2-\text{CH}_2-\text{OH}$ и $-\text{NH}^+(\text{CH}_2-\text{CH}_2-\text{OH})_2$ соли сульфатпроизводных высших карбоновых кислот. Сульфатпроизводные получены взаимодействием пальмового масла с этаноламинами (моноэтаноламин и диэтаноламин) при мольном соотношении равным 1:2 с последующим модифицированием серной кислотой продуктов реакции. Определены основные коллоидно-химические параметры синтезированных солей и исследована их нефтесобирающая и нефтедиспергирующая способность на поверхности вод с различной степенью минерализации.

Ключевые слова: *пальмовое масло, этаноламины, поверхностное натяжение, критическая концентрация мицеллообразования, свободная энергия адсорбции.*

Поступила в редакцию 21.03.2017.

**PALMA YAĞI VƏ ETANOLAMİNLƏR ƏSASINDA SAM-ların SİNTEZİ VƏ
KOLLOİD-KİMYƏVİ XASSƏLƏRİNİN ÖYRƏNİLMƏSİ**

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Ali karbon turşularının sulfat törəmələrinin anion səthi aktiv Na^+ , K^+ , NH_4^+ , $-NH_2^+-CH_2-CH_2-OH$ və $-NH^+-(CH_2-CH_2-OH)_2$ duzları sintez olunmuşdur. Sulfat törəmələr, palma yağının etanolaminlərlə (monoetanolamin və dietanolamin) 1:2 mol nisbətində qarşılıqlı təsir reaksiya məhsullarının sulfat turşusu ilə modifikasiyası ilə alınmışdır. Sintez olunmuş duzların əsas kolloid-kimyəvi parametrləri təyin olunmuş, fərqli minerallıqlı suların səthində neftiğicilik və neftdispersləmə qabiliyyətləri öyrənilmişdir.

Açar sözlər: palma yağı, etanolaminlər, səthi gərginlik, kritik misella əmələgətirmə qatılığı, adsorbsiya enerjisi

Redaksiyaya daxil olub 28.03.2017.