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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS AND STUDY OF NEW COMPOUNDS BASED
ON (C₆ –C₁₂) ALKYL-, 2-ALLYL- AND 2-
PROPENYLPHENOLS**

Speciality: 2314.01 – Petrochemistry

Field of science: Chemistry

Applicant: **Zulfiyya Museyib Javadova**

Baku– 2022

Dissertasiyanın müdafiəsi "31" oktyabr 2022-ci il tarixində saat 10⁰⁰-da Azərbaycan Respublikası Elm və Təhsil Nazirliyinin Neft-Kimya Prosesləri İnstitutunun nəzdində fəaliyyət göstərən ED 1.16 Dissertasiya şurasının iclasında keçiriləcək.

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Dissertasiya ilə Azərbaycan Respublikası Elm və Təhsil Nazirliyinin Neft-Kimya Prosesləri İnstitutunun kitabxanasında tanış olmaq mümkündür.

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Avtoreferat "23" sentyabr 2022-ci il tarixində zəruri ünvanlara göndərilmişdir.

General description of the work

Relevance of the topic and degree of development. One of the urgent problems of petrochemicals in modern times is the synthesis of new types of corrosion inhibitors, surface-active and antimicrobial compounds based on phenols and alkylalkenylphenols and their application in various fields of technology¹.

By this way, purposeful chemical reactions are carried out in the world, and obtained polyfunctional compounds are used in the oil industry to increase oil production, and effective biologically active compounds used in synthesis of drugs and as reagents antipathogenic pathogenic microorganisms².

Literature reviews show that the field of chemistry of alkenylphenols and its functionally substituted derivatives is developing intensively.

Due to the fact that alkenylphenols contain several reactive fragments (double bond, OH- group and aromatic ring) at the same time, stabilizers, corrosion inhibitors, antimicrobial additives and other technically important compounds can be obtained on their basis.

From this point of view, the presented dissertation is dedicated to solving an actual problem - the production of alkenylalkylphenols, their quaternary ammonium salts based on phenol, α -olefins, allyl halides, which are considered as industrial products, determination of their physical and chemical characteristics and molecular structures, their anticorrosion, antioxidant, antimicrobial properties, surface activity, etc.

The object and subject of the research. New compounds synthesized on the basis of alkylalkenylphenols and their application fields

Research goals and objectives. The main goal of the work is the synthesis of new compounds based on alkylalkenylphenols and the investigation of their application fields.

¹ Magerramov, A.M. Alkenylphenols: production, transformation, application / A.M. Magerramov, M.R. Bayramov, M.A. Agayeva, I.G. Mamedov, G.M. Mekhtieva // Advances in Chemistry, - Moscow: - 2015. vol. 84, No. 2, - p. 1258-1278.

² Menshchikova, E.V. Phenolic antioxidants in biology and medicine / E.V. Menshchikov, V.Z. Lankin, N.V. Kandalintseva, - Saarbrücken: LAP Lambert Academic Publishing, - 2012. - 496 p.

To achieve the goal, the following tasks were performed:

- Alkylation of phenol with (C₆-C₁₂) α -olefins in the presence of KU-23 catalyst were studied and various 4-(C₆-C₁₂) alkylphenols were synthesized;
- As a result of Claisen rearrangement of allyl ethers of 4-alkylphenols obtained as a result of the condensation of those alkylphenols with allylbromide were synthesized 2-allyl-4-alkylphenols;
- 2-propenyl-4-alkylphenols were synthesized by isomerization of 2-allyl-4-alkylphenols;
- As a result of three-component condensation of alkenyl (allyl-, propenyl-) alkylphenols with formaldehyde, aliphatic and cyclic amines, aminomethyl derivatives were obtained;
- Hydrochlorides (quaternary ammonium salts) of aminomethyl derivatives of alkenyl (allyl-, propenyl-) alkylphenols were synthesized;
- The reaction of 2-propenylphenol with hexene-1 in the presence of Al containing complex catalyst was studied;
- Physico-chemical properties and structures of all synthesized compounds were determined by modern analytical methods;
- Aminomethyl derivatives of alkenylalkylphenols and their quaternary ammonium salts were tested as corrosion inhibitors, antioxidant additives, surfactants, and bactericides against microbes.

Research methods. Alkylation reactions of phenol with (C₆-C₁₂) α -olefins in the presence of catalyst, synthesis reactions of alkenylalkylphenols obtained on the basis of alkylphenols, their aminomethylation with various amines, and reactions for obtaining hydrochlorides of synthesized Mannich bases were carried out in the laboratory in intermittent units.

Main clauses defended. Catalytic alkylation reactions of phenol with (C₆-C₁₂) α -olefins, synthesis of alkenylalkylphenols based on obtained alkylphenols, aminomethylation reactions of the latter with various amines, preparation of quaternary ammonium salts of synthesized Mannich bases were carried out, antimicrobial, bactericidal and antioxidant properties, as well as tests as surface-

active substances of Mannich bases and their quaternary ammonium salts were tested.

Scientific novelty of the research. Here for the first time:

– The relationship between structures and properties of the synthesized functionally substituted phenolic compounds and their derivatives was studied.

– Optimization of the alkylation reaction of phenol with hexene-1 in the presence of KU-23 catalyst and the model of the process was drawn up and the alkylation of phenol with other α -olefins was studied under the found optimal conditions.

– Alkylation reaction of 2-propenylphenol with hexene-1 in the presence of a metal complex catalyst was studied and optimal conditions were found. The molecular structure and mass of the obtained reaction product were determined and it was found that it consists of an oligomer of alkylalkenylphenol.

– A number of new organic compounds containing allyl-, propenyl-, aminomethyl groups were synthesized on the basis of the obtained alkylphenols, and water-soluble quaternary ammonium salts were obtained on the basis of them. The structures of the synthesized compounds were confirmed by NMR-spectroscopy.

– Compounds containing various functional groups (alkenyl-, aminomethyl-, etc.) were tested as additives to oils and confirmed their polyfunctional properties.

– It was found that most of the hydrochlorides of aminomethyl derivatives of alkylalkenylphenols (quaternary ammonium salts in aqueous systems) have surface-active properties in water-air, kerosene-water environments at different temperatures, concentrations and systems.

– The quaternary ammonium salt of 2-Allyl-4-isooctyl-6-piperidinomethylphenol was studied as a corrosion inhibitor, and the activation energy (ΔE), enthalpy (ΔH) and entropy (ΔS) of process were calculated.

The teoretical and practical significance of research. A complex analysis of the structures of compounds containing hydroxyl, nitrogen, alkenyl and chlorine fragments in the synthesized composition and conducted research has shown that they can use as

antioxidant additives to oils, corrosion inhibitors, etc., in this regard the synthesized alkenyl(allyl-, propenyl)-(C₆-C₁₂)alkylphenols, either aminomethyl derivatives or quaternary ammonium salts can be used as antioxidant additives to oils, corrosion inhibitors, surfactants in the oil industry, biocide against *S. aureus* bacteria.

2 Azerbaijani and 1 Eurasian patents were obtained on the base of positive results of study.

Approve and application. The main provisions of the study and obtained results were reported and discussed at the following International and Republican conferences:

XIX Mendeleevskiy Congress of General and Applied Chemistry (Volgograd, September 25-30, 2011); VIII Baku International Mamedaliyev Conference on Petroleum Chemistry (Baku, October 3-6, 2012); Republican Scientific Conference on "Lubricants, fuels, special liquids, additives and reagents" dedicated to the 50th anniversary of the establishment of the Additives Chemistry Institute named after Academician A. Guliyev (Baku, October 13-14, 2015); VI All-Russian Conference dedicated to the 50th anniversary of the Chuvash State University. I.N. Ulyanova (Cheboksary, November 23-24, 2017); The 1st Republican Scientific Conference of students dedicated to the 96th anniversary of the birth of national leader H.A. Aliyev (Baku, BHO, April 15-19, 2019); The International Scientific Conference "Actual problems of modern chemistry" dedicated to the 90th anniversary of the Academician Y.H.Mammadaliyev Institute of Petrochemical Processes (Baku, October 2-4, 2019); The 7th International Scientific and Practical Conference (Tokyo, Japan, February 6-8, 2021); II International Scientific Conference of Students and Young Researchers dedicated to the 98th anniversary of the birth of national leader H.A. Aliyev (Baku, BHO, April 13-28, 2021).

The results of the dissertation were published in 12 articles (2 in Indexed Journals), 2 Azerbaijan and 1 Eurasian Patents and 9 theses.

The name of the institution where the dissertation work was performed. Study was carried out in the scientific-research laboratory "Alkenylphenols chemistry" operating of department of "Oil chemistry and chemical technology" of Baku State University.

The total volume of the dissertation with a sign indicating the volume of the structural sections of the dissertation separately. Dissertation work consists of introduction, 4 chapters, conclusion, bibliographic list of 162 names of used literature, 36 pictures and 34 tables.

In the introduction, the relevance of topic, the set goal, objects, methods, subject, methodological basis and model of the research conducted to achieve that goal, the scientific novelty and approval of the results obtained in the work, the structure and scope of the dissertation are presented as briefly and as concretely as possible.

The first chapter is literature review, which consists of two sub-chapters, provides an analysis of scientific research works conducted by world and Azerbaijani scientists on corrosion inhibitors, antioxidant additives, bactericides.

In the second chapter, methods of alkylation of phenol with α -olefins, preparation of allyl ethers as a result of condensation of alkylphenols with allyl bromide, alkylallylphenols as a result of Claisen rearrangement, aminomethylation of the latter with amines (according to the Mannich reaction), methods of preparation of quaternary ammonium salts of aminomethyl derivatives and NMR spectrums of synthesized compounds are given.

The third chapter discusses the results of the research. Including optimization of the alkylation process of phenol with α -olefins, the construction of a kinetic model, the discussion of the results related to the production of aminomethyl derivatives of alkylalkenylphenols and quaternary ammonium salts.

The fourth chapter presents the results of the research of the aminomethyl derivatives of 2-(allyl-, propenyl-)alkylphenols and their quaternary ammonium salts as antioxidant, corrosion inhibitor, antimicrobial, bactericidal properties against SRB, as surface-active compounds.

In conclusion, scientific innovations resulted in study presented and chemical point of view are explained.

At the end of the dissertation work, **a list of literature** (sources) compiled in accordance with the current requirements, used in its implementation and writing, is given.

There are 9462 marks in the introduction, 46021 marks in the first chapter, 30052 marks in the second chapter, 36179 marks in the third chapter, 47784 marks in the fourth chapter, and 4251 marks in the results. The total volume of the dissertation consists of 174032 chapters.

The applicant's personal contribution to the research conducted. While completing the dissertation, the author directly participated in the analysis of the recent world literature review on the subject, conducting experiments, analyzing the NMR spectra of reaction products, preparing articles, as well as solving other issues.

MAIN CONTENTS OF THE WORK

1. Primary raw materials, catalysts, rules of conducting experiments and methods of analysis of reaction products. Phenol, (C₆-C₁₂) α -olefins, allyl bromide, formaldehyde, diethylamine, piperidine, morpholine, benzene, toluene, acetone, K₂CO₃, HCl, H₂SO₄, KOH, CH₃OH, NaCl, C₂H₅Cl, AlCl₃ were taken as raw materials for the research.

KU-23 (ГОСТ 20298-74) and aluminum-containing complex catalysts were used in the alkylation reaction.

In order to prepare an aluminum-containing catalyst, 5 ml of toluene and 2.5 g of AlCl₃ were added into a flask equipped with a condenser and a thermometer and heated to 40⁰C (with intense stirring), then 8.5 ml (7.7 g) of ethyl chloride was added and the temperature of reaction increased to 55-60⁰C and stirred for 40 min. The obtained complex was used after cooling and keeping it in dark for 2-3 days.

Alkylation of phenol with (C₆-C₁₂) α -olefins was carried out at normal conditions in three-necked flask. When the reaction completed, the liquid part of mixture was separated from the catalyst, initial unreacted phenol and olefin were distilled in the atmosphere, and then the main reaction product (alkylphenol) in a vacuum.

Alkylalkenylphenols were obtained in normal conditions on the basis of synthesized alkylphenols.

Aminomethyl derivatives of alkylalkenylphenols were synthesized as a result of three-component condensation with formaldehyde, alkyl and cyclic amines.

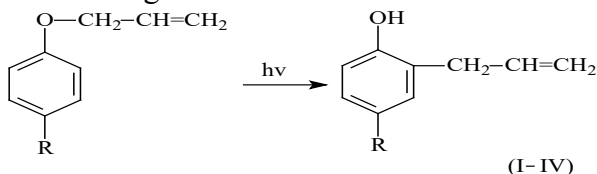
For obtaining quaternary ammonium salts of aminomethyl derivatives of alkylalkenylphenols, HCl (gas) was released from benzenesolution for 4-5 hours at room temperature.

The density of the synthesized compounds was determined by pycnometric method, the refraction coefficients were determined by the refractometric method on the apparatus "IRF-22" (Russia), and their structures were confirmed by NMR-spectroscopy. NMR-analysis was carried out on a Bruker-300 spectrometer (operating frequency 300 and 75 MHz), made in Germany, using CDCl_3 (chloroform) and D_2O as solvents.

Molecular weight of the oligomer was determined in a chromat-mass spectrometer manufactured by the German company "Bruker" (HPLS-MS Agilen 1200-Esquire-6000).

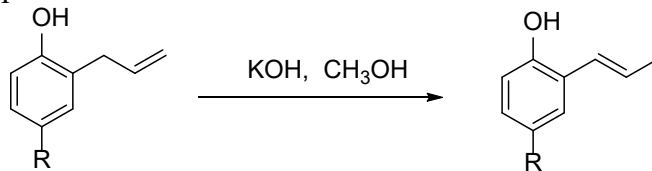
German-made DSA 30 for determining the surface tension of quaternary ammonium salts of Mannix bases; Krüss Scientific was used.

2. Preparation of 2-Allylalkylphenols. Allylalkylphenols were prepared by well-known method - allyl ethers of alkylphenols were heated in a microwave oven for 7-10 minutes. It was obtained as a result of Claisen rearrangement:



here, $\text{R}=(\text{C}_6\text{-C}_{12}) \alpha\text{-olefin}$.

3. Preparation of 2-propenylalkylphenols. 2-Propenylalkylphenols were obtained by known method - by isomerization of 2-allylalkylphenols:



here, $\text{R} = \text{C}_6\text{H}_{13}\text{- (V)}; \text{C}_8\text{H}_{17}\text{- (VI)}; \text{C}_{10}\text{H}_{21}\text{- (VII)}$.

2-Allylalkylphenol was added to saturated solution of KOH in

methyl alcohol in 1:3 ratio. A portion of solvent is separated until the temperature reaches 110°C, then the remainder was boiled at 110°C for 6 hours in a flask equipped with a condenser.

When the reaction completed, the product is purified by washing with distilled water, dried, and the residue was distilled under vacuum.

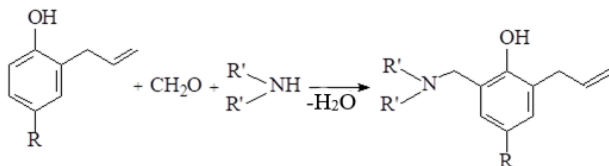
4. Study of alkylation reaction of alkenylphenols with hexene-

1. Developing more efficient method of alkylation of alkenylphenols, optimization of the alkylation reaction of phenol with hexene-1 was studied as a model reaction. KU-23 was used as a catalyst. Optimal conditions for the reaction were determined (temperature – 80°C, reaction time - 4 s, mole ratio of components - phenol:hexene-1-2:1, amount of catalyst - 10% (based on phenol)). Alkylation reactions of phenol with other α -olefins were carried out under the found optimal conditions, and the yield was 70,1-57%. The results obtained from the optimization were satisfied to the experimental results.

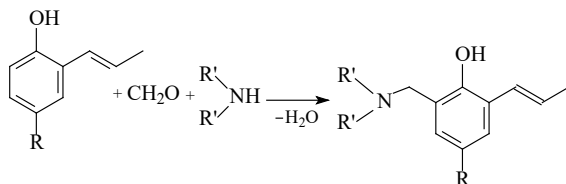
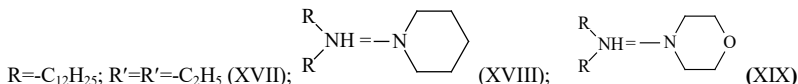
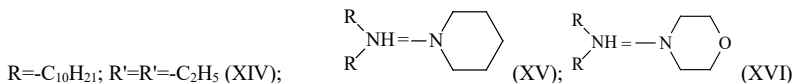
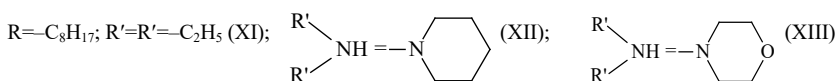
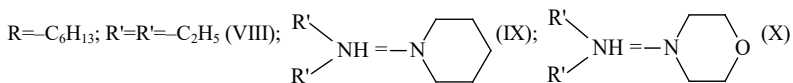
Then, the alkylation reactions of 2-propenyl- and 2-allylphenols were also carried out under the found optimal conditions. However, under those conditions, the oligomer of 2-propenylphenol, and a small amount of dimer and trimer of 2-allylphenol obtained, not alkenylalkylphenol, as the targeted reaction product. Considering this, a metal complex ($\text{AlCl}_3 + \text{C}_2\text{H}_5\text{Cl} + \text{toluene}$) was used as a catalyst. The effect of various factors on the process (temperature, 2-propenylphenol: hexene-1 mole ratio, amount of catalyst, reaction time and other factors) was studied and optimal reaction conditions were determined (temperature 120°C, mole ratio of components 1:1, reaction time 1,5 hours, the amount of catalyst is 0,5%). The reaction product consists of oligoalkylation product (yield 72%), its structure is NMR-, IR-spectroscopy, and its molecular mass (2500-2700) is determined by liquid mass chromatography method is defined.

Despite the fact that the oligomerization product of alkenylalkylphenol obtained under the studied reaction conditions has high functional properties, the research was continued on the previously mentioned reaction schemes (allylation of synthesized alkylphenols, rearrangement, etc.) to solve the problem, that is, to obtain individual alkylalkenylphenols.

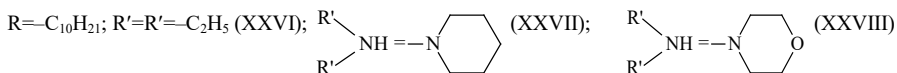
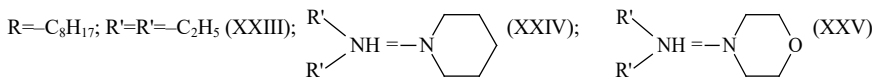
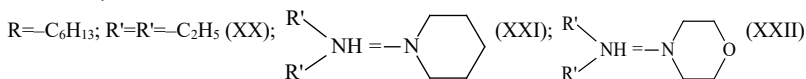
5. Study of aminomethylation reactions of alkenylalkylphenols. Triple condensation of 4-(C₆-C₁₂)alkyl-2-allyl-(2-propenyl)phenols with binary amines of different structures and formaldehyde (Mannich reaction) at 70-75°C, for 4-5 hours, in 1:1:1 molar ratio of reagents was carried out according to the following scheme in benzene medium:



here,

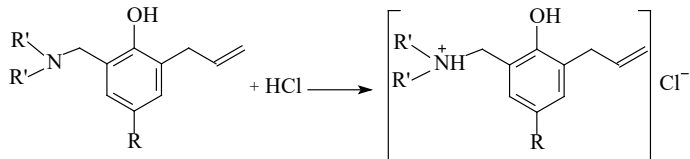


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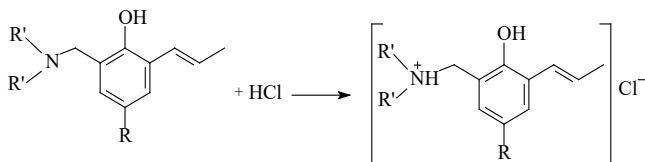
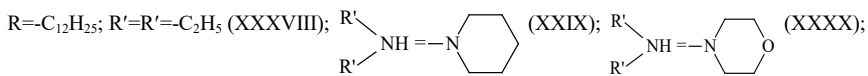
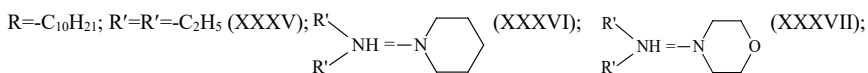
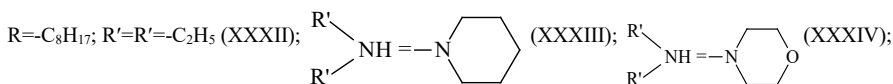
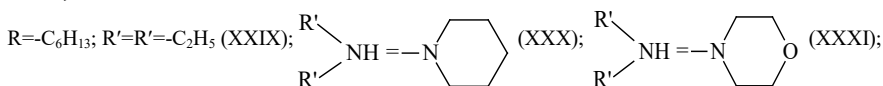


6. Synthesis of hydrochlorides of aminomethyl derivatives of alkenylalkylphenols. The water-soluble hydrochlorides of the

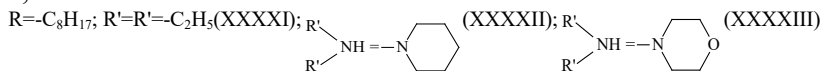
aminomethyl derivatives of the synthesized alkenylalkylphenols were obtained based on the interaction of their solution in benzene with hydrogen chloride according to the following reaction schemes:



here,



here,



The structures of the synthesized compounds (both aminomethyl derivatives and their hydrochlorides) were confirmed by NMR spectroscopy.

Study of the fields of application of synthesized compounds

1. Study of quaternary ammonium salts of aminomethyl derivatives of 2-Allyl-4-(C₆-C₁₂)alkylphenols as inhibitors of metal corrosion in 3% NaCl systems. Quaternary ammonium salts of aminomethyl derivatives of 2-allyl-4-(C₆-C₁₂)alkylphenols were synthesized and studied as corrosion inhibitors by gravimetric method for 5 hours, at 25, 35, 45°C, in a 3% NaCl+1:9 by volume

kerosene-water system using a known method.

For comparison, ANP-2 inhibitors used in industry were used.

The results obtained from the conducted studies showed that when the concentration of synthesized compounds increased from 50 mg/l to 150 mg/l, St. The anticorrosion effect of steel 3 varies in a wide range (35-99%) depending on the temperature, the nature of the aminomethyl fragment and the number of carbon atoms in the alkyl group.

Thus, the effect of metal corrosion protection increases by increasing of the concentration of all synthesized compounds from 50 mg/l to 150 mg/l (35.03-99.00%), but the temperature increasing (from 25°C to 45°C- up to) decreases the efficiency in all concentrations of inhibitors (from 95% to 35%). In addition, as the number of carbon atoms in the alkyl group increases (up to a certain limit - C₉), the anti-corrosion protection effect increases. Also, the effect of the nature of the amine group on corrosion was studied and the change of the corrosion effect in the following order was found:

diethylamine < morpholine < piperidine

Thus, it was determined that compound XXXIII showed the highest inhibitory effect (99,0%). Therefore, the following studies were mainly conducted with those salts.

2. Investigation of 2-Allyl-4-isooctyl-6-aminomethylphenols hydrochloride (quaternary ammonium salts in water) as a corrosion inhibitor in H₂S medium. The test of the synthesized compounds as a corrosion inhibitor in a hydrogen-sulfide (more aggressive) medium was also studied.

It was determined that compounds XXXII-XXXIV synthesized in H₂S-saturated medium give high results as corrosion inhibitors. However, the corrosion effect in the 3% NaCl-water+kerosene system is slightly higher than the results obtained in the H₂S medium. So, for example, while the efficiency of compound XXXIII in 3% NaCl-water + kerosene medium at 100 and 150 mg/l concentrations at 25°C temperature is 97,7 and 99,0%, respectively, the result in H₂S medium is slightly lower (94,3 and 96,5 %).

3. Investigation of 2-Allyl-4-isooctyl-6-aminomethylphenols hydrochloride (quaternary ammonium salts in water) as a

corrosion inhibitor in CO₂ medium. Hydrochlorides of the aminomethyl derivative obtained on the basis of 2-allyl-4-isoctylphenol were studied as corrosion inhibitors in the 3% NaCl-water + kerosene + CO₂ system.

It was determined that when the corrosion test was carried out in the salt water-kerosene and CO₂ gas system at 100, 150 mg/l concentrations at 25°C, the protective effect of the XXXIII compound was 95.9 and 97.9%, slightly different from the results of the test conducted in the H₂S medium under those conditions.

4. Testing of quaternary ammonium salts of synthesized aminomethyl derivatives of 2-propenylalkylphenols as a corrosion inhibitor of steel St. 3. Tests of synthesized quaternary ammonium salts of aminomethyl derivatives of 2-propenylalkylphenols as corrosion inhibitors were carried out in the above-mentioned conditions - NaCl + hydrocarbon (kerosene) medium, and it was determined that at concentrations 100 and 150 mg/l compound XXXXII protects 94,9; 97,7%; compound XXXIII 97,7; 99,0% from corrosion.

At the same time, quaternary ammonium salts were tested as corrosion inhibitors in the solution saturated with H₂S (500 mg/l) of that system. In H₂S medium, the corrosion efficiency of compound XXXXII at concentrations of 100 and 150 mg/l at 25°C is 87,0 and 90,3% (vs. 94,9 and 97,7%).

Those compounds were tested as corrosion inhibitors in CO₂-saturated medium under the conditions mentioned above. It was determined that the inhibitory efficiency of compound XXXXII is relatively higher (89,1 and 93,6%) in CO₂ medium instead of H₂S. As in previous tests, corrosion efficiency decreases with increasing temperature.

5. Determination of some thermodynamic parameters of quaternary ammonium salt of 2-Allyl-4-isoctyl-6-piperidinomethylphenol. During the study of synthesized quaternary ammonium salt of 2-allyl-4-isoctyl-6-piperidinomethylphenol as corrosion inhibitor in 3% NaCl-water + kerosene (9:1 volume) medium, some physical-chemical quantities were calculated and characteristics were investigated.

At different temperatures (25, 35 and 45°C) and concentrations (0; 0.025; 0.050; 0.10; 0.15 g/l) the rate (K_C), efficiency (Z) of the corrosion were studied and the results were summarized in table 1.

The corrosion rate constant is calculated according to Arrenius equation:

$$K_C = A e^{\frac{-E_a}{RT}}$$

Where, E_a is the probable activation energy of the corrosion process,

A -exponential constant, frequency factor.

As expected, as the temperature increases, the rate of corrosion increases and effectiveness decreases (table 1).

Table 1

Kinetic parameters of corrosion of steel St. 3 in salt-water medium

$C_{inh.}$, g/l	Corrosion rate (K_C), g/m ² ·h			Protection efficiency from corrosion, %		
	298K	308K	318K	298K	308K	318K
0	0,15441	0,23917	0,32565	-	-	-
0,025	0,08431	0,12559	0,18247	86,68	84,92	81,94
0,050	0,01181	0,01864	0,02626	92,35	88,72	84,64
0,100	0,005453	0,010034	0,018137	96,47	93,36	89,36
0,150	0,001896	0,004579	0,013386	99,00	97,23	92,17

This is probably related to the structure of the inhibitor molecules and their desorption from the surface of the metal.

The relatively weak decrease in inhibition efficiency according to temperature increasing (2-4% per 10⁰C) indicates that the inhibitor molecules are probably adsorbed onto the metal surface by partial chemisorption mechanism. The activated chemisorption process can be compensated by desorption of inhibitor molecules to a certain extent and increase with increasing temperature. Therefore, formation of protective coating due to adsorption of inhibitor molecules on the metal surface occurs by a mixed mechanism.

Eyring's equation was used to calculate the enthalpy (ΔH) and entropy (ΔS) of the corrosion process:

$$K_C = \frac{RT}{hN_A} e^{\frac{\Delta S}{R}} e^{-\frac{\Delta H}{RT}}$$

In the equation, the translation coefficient is assumed to be equal to unity.

Here, ΔH and ΔS are the activation enthalpy and entropy of the corrosion process, respectively,

R-universal gas constant,

N_A -Avogadro's number,

h-Plank's constant.

The activation energy can also be found as:

$$E_a = \Delta H + RT$$

The value of the expression $E_a - \Delta H$ is given in table 2, and this value is close to the value of the RT product, which indicates that the corrosion process obeys the first order kinetic equation.

As can be seen from the table, the corrosion process of steel proceeds with a significant decrease in entropy ($\Delta S < 0$) in the salt-water system.

As mentioned earlier, the corrosion process of metals is close to a neutral environment ($6 \leq \text{pH} \leq 8$), including in a salt-water solution, as in oxygen corrosion, a solid corrosion product is formed here, and the corrosion process is multi-stage. Most likely, the corrosion process formation and disintegration of the active complex causes the value of entropy ($\Delta S < 0$) to be negative.

Table 2

Values of the thermodynamic parameters of the corrosion process of steel St. 3 without inhibitor and in different concentrations of inhibitor

$C \cdot 10^{-3}$, g/l	E_a , kC/mole	ΔH , kC/mole	$E_a - \Delta H$, kC/mole	ΔS , C/mole	$E_{\text{calc.}}$, kC/mole
0	29,43	26,87	2,56	-170,2	29,43
25	30,52	27,95	2,57	-171,4	30,50
50	31,81	28,97	2,84	-184,5	33,96
100	47,61	45,04	2,57	-137,2	48,63
150	77,44	74,89	2,55	-46,3	74,27

6. Study of cooligomers of 2-propenylphenol with α -olefins as antioxidants. The antioxidant properties of oligomers of 2-propenylphenol with α -olefins were first compared with the indicators of the studied individual compounds, as well as taking into account the recent interest in these types of compounds in the literature.

For comparison, first the autoxidation process of cumene (110°C, without any antioxidant) and then its oxidation in the presence of polyphenol-type sooligomers synthesized by us (8%) were carried

out. During the process, the induction times of the samples were determined. The obtained results are given in table 3.

It is clear from the results that sooligomers increase the induction time from 0 to 220-280 min.

To demonstrate the process path more clearly, the oxidation of cumene was carried out in the presence of initiator (activator). Studies were carried out in the laboratory in a monometric device: temperature 60°C, amount of sooligomers 8%.

Actionmechanismof these types of compounds synthesized by us as antioxidants is explained by the interaction of phenol fragments in the large molecule (framework) with peroxide radicals one after the other.

Table 3

Auto-oxidation of cumene and its oxidation in addition of sooligomers

Compound	Inductionperiod, min.	Induction time of the reaction with umylperoxide radicals, min.
Cumol (withoutadditives)	20	0
Cumol+8% soolyqomer of 2-propenylphenol-heptene-1 (I)	130	280
Cumol+8% soolyqomer of 2-propenylphenol-decene-1 (II)	90	220

7. Testing of the synthesized aminomethyl derivatives as antioxidant additives. Testing of the synthesized compounds as antioxidants increasing the oxidation stability of M-8 motor oil was carried out in the APCM-1M apparatus (DÜST 981-75).

Calculated amounts of synthesized compounds are added to the oil, heated to 120-140°C and pumped through it at 200 ml/min. rapid oxygenation (30 hours).

In the same conditions, a control test is also conducted. After the research is completed, the main indicators are the acid number of the oxidized oil (K), the acid number of volatile low-molecular acids separated from the oil as a result of oxidation (K'), and the amount of participate (X).

It was determined that when adding compounds XI-XIII (at 2% concentration) to M-8 base oil, the amount of participate is 0.1046; 0.2232; 0.1635%. Thus, compound XI shows the best results. This compound differs from compounds XII and XIII only by the

aminomethyl group. The presence of diethylaminomethyl fragment and phenolic hydroxyl in its structure provides high antioxidant property, which is related to the synergism effect.

Study of antimicrobial properties of synthesized compounds

1. Investigation of the antimicrobial properties of sooligomers of 2-propenylphenol with heptene-1 and decene-1. As known, various chemical compounds are used to protect metal structures and other materials from biocorrosion. Antimicrobial properties of 2-propenylphenol and α -olefins in I-12A oil were studied.

The results of the tests showed that the studied sooligomers show high fungicidal properties at concentrations of 0.25 and 0.5% and surpass 8-hydroxyquinoline taken as a standard at the same concentrations due to their effectiveness. While 0.5% hepten-1 sooligomer together with I-12 A oil has a fungal destruction zone of 2.5-3 cm, for the same concentration of 8-hydroxyquinoline with I-12 A oil, this indicator is only 1.1 cm.

2. Study of antimicrobial properties of aminomethyl derivatives of 2-allyl-4-isoctylphenol. The study conducted with compounds XI, XII, XIII. The experiments were carried out in the postgate environment at a temperature of 30-32°C for 15 days, and SRB separated from the formation waters of the "Neft-daslari" field was used.

The amount of SRB is 104-106 cv/ml, and the amount of the tested compound is 100 and 200 mg/l. In addition, control experiments (without combination) were also conducted. The SRB destruction rate (Z) of the compounds was calculated based on the amount of hydrogen sulphide released during the test.

The results of the conducted tests showed that the synthesized compounds have high bactericidal activity against SRB. The highest bactericidal result is shown by compound XIII (the rate of destruction of SRB at a concentration of 200 mg/l is 90%).

Synthesized compounds XXXII-XXXIV were also tested. Of these, compound XXXIV shows the best result, so that at concentration of 200 mg/l, its bacteria destruction rate reaches 93.5%.

Synthesized compounds XXXII, XXXIII, XXXIV were tested as

an antimicrobial additive in oil-cooling fluids. It was found that the salts containing the cyclic amine fragment perform better as compounds with mainly fungicidal properties. They kill only fungi, not bacteria, and zone of fungal destruction of salts XXXIII and XXXIV at 0.5% concentration is 3.6-3.8 cm happens.

3. Study of the antimicrobial activity of quaternary ammonium salt of 2-Allyl-4-isodecyl-6-diethylaminomethylphenol against disease-causing bacteria. The antimicrobial activity of synthesized quaternary ammonium salt of 2-allyl-4-isodecyl-6-diethylaminomethylphenol against disease-causing *Acinetobacter baumannii* BDU32, *Escherichia coli* BDU12, *Klebsiella pneumoniae* BDU44, *Pseudomonas aeruginosa* BDU49 gram-negative and *Staphylococcus aureus* BDU23 gram-positive bacteria was studied.

In order to ensure that the studied substance has high effectiveness, its minimum inhibitory concentration was determined. For this purpose, a liquid (agar-free) nutrient medium was used. The initial concentration of the substance was taken as 1000 µg/ml, by diluting each concentration twice, concentrations of 500 µg/ml, 250 µg/ml, 125 µg/ml, 62.5 µg/ml and 31.25 µg/ml were obtained. The substance in each concentration was incubated with the microbial suspension and the lowest concentration inhibiting the development of bacteria was determined to be 62.5 µg/ml. Experience has shown that the *Staphylococcus aureus* BDU23 bacterium is highly sensitive to the studied substance. The sensitivity of *Acinetobacter baumannii* BDU32, *Escherichia coli* BDU12, *Klebsiella pneumoniae* BDU44, *Pseudomonas aeruginosa* BDU49 bacteria to this substance was 8, 16 and 32 times lower than the sensitivity of *S. aureus* BDU23 bacteria, respectively.

In order to study the antimicrobial effect of the allyl group in the structure of the investigated compound against bacteria, the antimicrobial property of the quaternary ammonium salt of 4-isodecyl-6-diethylaminomethylphenol, which does not contain an allyl group, was studied under those conditions.

It was determined that the quaternary ammonium salt of 4-isodecyl-6-diethylaminomethylphenol, which does not contain an allyl group, shows a very slight sensitivity only against the

pathogenic bacterium *Staphylococcus aureus*.

It should be noted that while the quaternary ammonium salt of 2-allyl-4-isodecyl-6-diethylaminomethylphenol showed antimicrobial properties at a concentration of only 62.5 $\mu\text{g/ml}$, the quaternary ammonium salt of 4-isodecyl-6-diethylaminomethylphenol without an allyl group showed antimicrobial properties at 2000 $\mu\text{g/ml}$ (3.2 times more) shows antimicrobial properties.

Thus, as a result of the conducted studies, it was determined that the quaternary ammonium salt of 2-allyl-4-isodecyl-6-diethylaminomethylphenol has a specific and very strong antimicrobial effect only against *Staphylococcus aureus* pathogenic bacteria at a minimum inhibitory concentration of 62.5 $\mu\text{g/ml}$.

Study of the surface tension of quaternary ammonium salts of synthesized aminomethyl derivatives of allylalkylphenols at the water-air and water-hydrocarbon (kerosene) boundaries

Surfactants are used to increase the productivity of oil wells. The surface tension of aqueous solutions of synthesized compounds XXXII-XXXIV (in the form of quaternary ammonium salts) at the water-air interface at a temperature of 298.15 K was studied. Figure 1 shows the surface tension isotherm (curve 1) and adsorption (curve 2) of various concentrations of the XXXIII compound aqueous solutions at the water-air boundary.

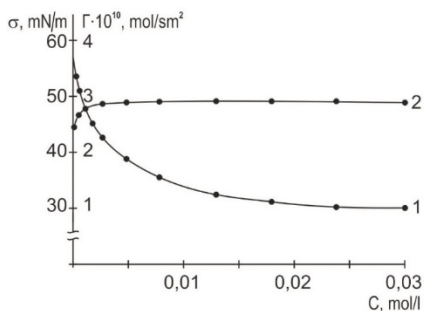


Figure 1. Surface tension isotherm of aqueous solutions of XXXIII compound (curve 1) and its adsorption at the water-air interface at a temperature of 298.15 K (curve 2)

As can be seen from the isotherm of salt of compound, it has appreciable surface activity.

The analysis of the experimental results allows finding the constants in the Shishkovsky and Langmuir equations. The values of constants found for aqueous solutions of XXXIII compound are as follows:

$A=6.237 \cdot 10^{-5}$, $b=7.346$; $K=16033$, $G_{\max}=2.963 \cdot 10^{-10}$ mol/cm² ($2.963 \cdot 10^{-6}$ mol/m²).

The maximum value of the adsorption of the compound at the water-air interface (G_{\max}) allows determining the effective cross section of the molecule in the dense monomolecular adsorption layer (ω_0) of SAB as a result of monomolecular layer formation.

It should be noted that since the structures of XXXIII and XXXIV compounds are close to each other (they differ by the piperidine and morpholine rings), the areas occupied by their molecules in the adsorbed dense monomolecular layer (ω_0) should also be close to each other. Figure 2 shows the surface tension isotherm (curve 1) and adsorption (curve 2) of compound XXXIV at the water-air interface.

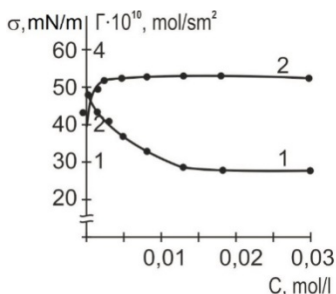


Figure 2. Surface tension isotherm of an aqueous solution of compound XXXIV (curve 1) and its adsorption at a temperature of 298.15 K at the water-air interface (curve 2)

As can be seen from the surface tension isotherm (curve 1) in Figure 2, surface tension of the compound decreases significantly when the thickness of the compound is low. This indicates that this compound has high surface activity.

Based on the values of K and G_{\max} in the Langmuir equation:

$$G = G_{\max} \frac{KC}{1 + KC}$$

here, $K=1/A$ - equilibrium constant of adsorption,

$G_{\max}=b/RT$.

Adsorption of the corresponding SAB (XXXII-XXXIV) molecules was calculated using this equation.

Based on the results obtained from the experiments, the constants of Shishkovsky and Langmuir equations of compound XXXIV were found:

$A=5.34 \cdot 10^{-5}$; $B=7,934$; $K=18732$, $G_{\max}=3.201 \cdot 10^{-10}$ mol/cm² ($3.201 \cdot 10^{-6}$ mol/m²), $\omega_{\min}=50.37 \text{ \AA}^2$ (0.504 nm^2).

By comparing the results obtained for compounds XXXIII and XXXIV, it was determined that the areas occupied by SAB molecules in the monomolecular adsorption layer are close to each other and equal to 56.03 and 50.37 Å², respectively.

Taking into account that the densities of salts XXXIII and XXXIV are equal to 953 kg/m³ and 979 kg/m³ in the initial approximation, the thickness (h) of the monomolecular layers, i.e. the effective length of the SAB molecule, can be calculated according to the following expression:

$$h = \frac{G_{max} \cdot M}{\rho}$$

here, M-molecular mass of SAB,
 ρ-density.

h=11.8 Å (1.18 nm) and h=12.5 Å (1.25 nm) for compounds XXXIII and XXXIV, respectively. Given that the structures of these two compounds are close to each other, the obtained values could be expected to be such.

Figure 3 shows the results of the surface tension of the aqueous solution of compound XXXII at the water-air interface:

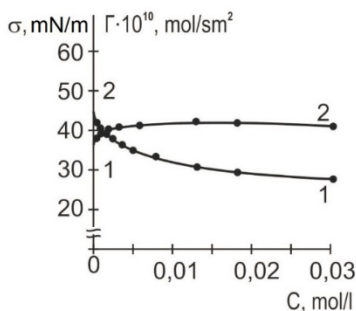


Figure 3. Surface tension isotherm (curve 1), adsorption (curve 2) of compound XXXII at a temperature of 298.15 K at the water-air interface

It should be noted that the quaternary ammonium salt of 2-allyl-4-isoctyl-6-diethylaminomethylphenol differs from compounds XXXIII and XXXIV by the area occupied by the aminomethyl group.

Therefore, the area occupied by this molecule must be different from the area occupied by the molecules of the other two compounds (XXXIII and XXXIV).

As a result of research, the values of the following parameters were found for article XXXII:

$A=5.14 \cdot 10^{-7}$; $b=4.022$; $K=2308249$; $G_{\max}=1.662 \cdot 10^{-10}$ mol/cm² ($1.662 \cdot 10^{-6}$ mol/m²); $\omega_{\min}=102.3 \text{ \AA}^2$.

As can be seen from the obtained results, the area occupied by the molecule of compound XXXII in the dense monomolecular adsorption layer is almost twice as large as the areas occupied by compounds XXXIII and XXXIV ($\omega_{\text{XXXIII}}=56.03$; $\omega_{\text{XXXIV}}=50.37 \text{ \AA}$), and therefore the maximum adsorption value (G_{\max}) of this compound is relatively low.

As for the calculated monomolecular dense adsorption layer thickness (h) (the effective length of the adsorbed molecule) for compound XXXII, $h=6.53 \text{ \AA}$, which is similar to that for compounds XXXIII and XXXIV ($h=11.8 \text{ \AA}$ and $h=12.5 \text{ \AA}$) is much lower than the result. The fact that the length of the molecule (h) is even lower than the true value is probably due to the non-perpendicular orientation of the molecule to the surface in the adsorption layer.

We also studied the dependence of the surface tension on the water phase density at the water-kerosene boundary.

Based on the experimental results, the value of the constants of the Shishkovsky and Langmuir equations and the parameters of the molecules were found in the dense monomolecular adsorption layer of SAB molecules at the water-kerosene boundary:

For compound XXXII: $A=4,319 \cdot 10^{-6}$; $b=5.25$; $K=228335$; $G_{\max}=2.118 \cdot 10^{-10}$ mol/cm²; $\omega_{\min}=78.39 \text{ \AA}^2$ (102.3 \AA^2); $h=8.53 \text{ \AA}$ (6.53 \AA) (values in parentheses are values at the water-air boundary).

Based on the obtained results, it can be said that the deviation of the SAB XXXII molecule from the perpendicular orientation is greater in adsorption at the water-air boundary. This causes an increase in the effective cross-section of the monomolecular adsorption layer ($\omega_{\min}=102.3 \text{ \AA}^2$). This fact is probably due to the presence of two ethyl radicals in the quaternary ammonium salt of 2-allyl-4-isooctyl-6-diethylaminomethylphenol. This, in turn, leads to a change in the hydrophobic-hydrophilic balance in the molecule of compound XXXII, unlike the other two compounds.

Thus, as a result of the research, it was determined that the synthesized compounds can be used as surface-active compounds to increase oil production in oil recovery.

CONCLUSION

1. As a simple model reaction for obtaining (C₆-C₁₂) alkylphenols, the optimization and kinetic model of the alkylation reaction of phenol with hexene-1 in the presence of KU-23 cationite catalyst was drawn up and the optimal conditions were established: temperature 80⁰C, reaction time 4 hours, the molar ratio of components 2:1, the amount of catalyst is 10% by phenol. The kinetic model of the alkylation reaction according to the probable mechanism was described, rate constants and activation energies were calculated. The calculated results did not differ more than 4-6% from the experimental results. This gives reason to accept the adequacy of proposed kinetic model.

Under the established optimal conditions, alkyl derivatives of phenol with other (C₆-C₁₂) α-olefins were obtained [12].

2. The alkylation of 2-propenylphenol with hexene-1 was studied in the presence of metal complex catalyst. The influence of various factors on the process was studied and optimal reaction conditions were found (120⁰C, mole ratio of components 1:1, reaction time 1.5 hours, amount of catalyst 0.5%). The structure and molecular weight of the obtained reaction product were studied by modern physical-chemical methods and it was confirmed that it consists of an oligomer of the alkylation product of 2-propenylphenol with hexene-1 [1, 2, 3].

3. Taking into account the useful functional properties of the oligomer obtained during the alkylation of 2-propenylphenol with hexene-1, technological conditions that provide maximum indicators for the process (120° C, reaction time 1.5 hours, ratio of propenylphenol:olefin ratio 1:1, amount of catalyst 0.5%, yield 71.3%) was found and it was determined that the viscosity index of the product obtained under these conditions is 87.9. The kinetics of the process was also investigated. The maximum deviation of the calculated results from the experimental results was less than 3-5%, which proves the adequacy of the prepared kinetic model [4, 7].

4. Amino(diethylamino-, piperidino-, morpholino-)methyl derivatives of 2-allyl- and 2-propenyl-4-(C₆-C₁₂)alkylphenols and their hydrochlorides (quaternary ammonium in water salts), as well

as the oligomer of the alkylation product of 2-propenylphenol with hexene-1 was synthesized. The structures of the synthesized compounds were confirmed by modern physicochemical analysis (^1H -, ^{13}C - NMR-, IR- and liquid mass spectroscopy) [4, 8].

5. In order to reveal the suitability of the aminomethyl derivatives of the synthesized 2-allyl(propenyl)-4-alkylphenols as antioxidant additives, autoxidation in the presence of cumene activator as a model reaction was studied, and the kinetic parameters of their interaction with peroxide radicals (K_7) and the induction number is defined. The stoichiometric coefficients of investigated compounds range from 0.72 to 2.04, and the rate constants range from 1.69 to $4.02 \times 10^{-4} \text{ l} \times \text{mol}^{-1} \times \text{s}^{-1}$. Among synthesized compounds, 2-allyl-4-isooctyl-6-morpholinomethyl shows better results (the induction period is 30 minutes higher than that of ionol) [6, 9, 11].

6. Hydrochlorides of synthesized 2-alkenyl-4-alkyl-6-aminomethylphenols (quaternary ammonium salts in water) at different temperatures (25°C , 35°C , 45°C) and concentrations (50, 100, 150 mg/l) for 5 hours in 3% NaCl- In the water+kerosene (9:1 volume ratio) system, Ct. 3 brand steel was studied in corrosion protection. It was determined that the protective effect of the studied compounds varies widely (35-99%) depending on the molecular mass, the nature of the aminomethyl group, density and temperature, medium (H_2S , CO_2). Kinetic and thermodynamic parameters (corrosion activation energy, ΔH , ΔE , etc.) of the quaternary ammonium salt of 2-allyl-4-isooctyl-6-piperidinomethylphenol of synthesized compounds were calculated [14, 17].

7. Corrosion of propenyl-4-isooctyl-6-aminomethylphenols quaternary ammonium chlorides in 3% water solution of NaCl + kerosene (9:1 by volume), in H_2S and CO_2 environments, at different temperatures and concentrations (50, 100, 150 mg/l), for 5 hours were studied as inhibitors and it was established that they have high efficiency [17].

8. The surface tension of quaternary ammonium salts of 2-allyl-4-isooctyl-6-amino(piperidino-, diethylamino-, morpholino-) methylphenols at water-air, water-hydrocarbon boundaries was studied. Surface tension and adsorption isotherms (curves) were constructed

at both boundaries.

The constants of Shishkovsky and Langmuir equations were found on the base of $\sigma = f(\ln C)$

$$A=6.237 \times 10^{-5}; B=7,346; K=16033; G_{\max}=2.963 \times 10^{-10} \text{ mol/cm}^2 \text{ (I)}$$

$$A=5.14 \times 10^{-7}; B=4,022; K=2308249; G_{\max}=1.662 \times 10^{-10} \text{ mol/cm}^2 \text{ (II)}$$

$$A=5.34 \times 10^{-5}; B=7,934; K=18732; G_{\max}=3.201 \times 10^{-10} \text{ mol/cm}^2 \text{ (III)}$$

So, it was determined that the investigated compounds have high surface activity, and they are close to each other in terms of micelle formation properties, and the critical micelle formation density is approximately 0.018 mol/l [15, 16, 21, 22, 24].

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