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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

OBTAINING SORBENTS FOR THE REMOVAL OF RADIONUCLIDES AND HEAVY METALS FROM OILFIELD FORMATION WATERS AND OTHER AQUEOUS SYSTEMS

Speciality: 2314.01- Petrochemistry

Field of science: Chemistry

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GENERAL CHARACTERISTICS OF WORK

Relevance of the work and degree of development. As is known, in the modern era, a number of polyfunctional compounds are being developed on the basis of substances such as phenols, alkyl- and alkenylphenols, amines, aldehydes, organic acids, etc. These compounds include antioxidants, corrosion inhibitors, additives for lubricants and fuels, biocides, sorbents used in the removal of environmentally harmful heavy metals and other technical and special-purpose compounds. Intensive scientific study is being conducted in these areas.

Natural and synthetic sorbent materials are widely used for the purpose of cleaning the environment, including water systems, from heavy metals, radionuclides and other toxic substances[1,2]. In addition, other methods, such as chemical and electrochemical techniques, are also employed worldwide. In recent years, scientific and practical work has been carried out on the development of heteroatom-containing (N, S, P, O) polyfunctional sorbents with long-term working capabilities and the study of their chemistry.

These types of sorbents play an irreplaceable role in addressing the most important issues of "green chemistry," because it's known that natural sorption materials are not considered ecologically favorable for reuse in the long term, are limited in variety, have low selectivity and can lead to additional problems.

The sorbent materials, obtained by synthetic methods, such as eco-friendly "cross-linked" thermoresponsive polymers hold a special place. Generally, "cross-linked" polymers have found application in other areas as well, such as microelectronics, membranes, contact lenses, and other optical materials, computer technology, etc. Recently, these types of sorbents have been increasingly used to purify blood from toxic substances.

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[1] Ajmal, M. Amidoximated poly(acrylonitrile) particles for environmental applications: Removal of heavy metal ions, dyes, and herbicides from water with different sources / Muhammed Ajmal, Sahin Demirci, Mohammed Siddiq [et al.] // Journal of Applied Polymer Science, – 2016. v. 133, No. 7, – p. 430-432.

2 Guo, H. Carbon materials for extraction of uranium from seawater / Hang Guo, Peng Mei, Jingting Xiao [et al.] // Chemosphere, – 2021. v. 278, – p. 1304-1311

Scientific research in the direction of obtaining and studying various types of sorbent materials for the removal of heavy metals and radionuclides from aqueous systems is required to develop the synthesis of three-dimensional functionalized sorbents with chelating functional groups on the basis of functional oligomers meeting modern requirements.

The topic of the dissertation entitled "Obtaining Sorbents for the Removal of Radionuclides and Heavy Metals from Oilfield Formation Waters and Other Aqueous Systems is relevant and holds both scientific and practical significance.

Object and subject of work. The object of the study includes the use of alkenylphenols, maleic anhydride, styrene, various amines. The subject of the study is the development of new types of sorbents based on alkenylphenols and their application in the removal of heavy metals and radionuclides from various systems.

The purpose and objectives of the dissertation work. The goal of the study is synthesizing functional oligomers and thermoresponsive polyfunctional polymers with different structures based on phenol, 2-propenyl-, 4-isopropenylphenols, formaldehyde, acrylonitrile, maleic anhydride, styrene, amines, etc. The study includes the study of their structure, physicochemical properties and the process of uranyl ions and heavy metal sorption from model aqueous solutions, as well as studying the sorption process of elements present in oil field waters and providing relevant recommendations.

The goal was achieved through solving the following specific tasks:

- obtaining a three-component oligomer on the basis of 4-isopropenylphenol, phenol and formaldehyde and optimizing the modification process with maleic anhydride;
- obtaining a functionally-substituted sorbent by modifying the three-component oligomer synthesized from 4-isopropenylphenol, phenol and formaldehyde with acrylonitrile in the presence of a initiator;
- synthesis of a three-component oligomer containing a phosphorus atom on the basis of 4-isopropenylphenol, formaldehyde, and 4-(1-methyl-1-dimethoxyphosphorylethyl)

phenol in the presence of an alkaline catalyst, and modification with acrylonitrile to obtain a sorbent containing both nitrogen and phosphorus atoms;

- polycondensation of 2-propenylphenol with formaldehyde in the presence of an alkaline catalyst, and the interaction reaction with maleic anhydride (as a crosslinking agent) to obtain a polyfunctional "cross-linked" polymer;
- Conducting the hydrolysis process of synthesized "cross-linked" polymers for the purpose of obtaining a carboxylate-type sorbent;
- synthesis of cooligomers on the basis of 4-isopropenylphenol-maleic anhydride and modification (crosslinking) with ethylenediamine, diethylenetriamine, and ethylenediamine-formaldehyde-epoxy resin, followed by the study of the obtained modified product as a sorbent;
- obtaining a three-component oligomer on the basis of 4-isopropenylphenol, maleic anhydride, and styrene, and modification with ethylenediamine, ethylenediamine-formaldehyde and diethylenetriamine, followed by studying the obtained modified product as a sorbent;
- differential thermal analysis of the temperature resistance of the new polyfunctional sorbents synthesized on the basis of alkenylphenols;
- study of the sorption process of uranyl ions of all synthesized sorbents from aqueous solutions;
- study of the sorption properties of a polymer (XVI) on the basis of a composition containing 4-isopropenylphenol-maleic anhydride, formaldehyde, ethylenediamine, and epoxy resin, and the modified product obtained by the modification of cooligomer with formaldehyde and ethylenediamine on the basis of 4-isopropenylphenol, maleic anhydride, and styrene, in the removal of Pb^{2+} ions from model aqueous solutions;
- study of four sorbents, synthesized from the produced drilling fluids from two oil fields (Garadagh district, Oil and Gas

Extraction Department (OGED) Lokbatan Field No. 260 named after Amirov and "Bibihaybatneft" Oil and Gas Extraction Department (OGED) Field No. 3, operating at a depth of 625-800 meters on the third horizontal of well K748:

- a cooligomer obtained on the basis of 4-isopropenylphenol, formaldehyde and phenol, modified with maleic anhydride;
- a cooligomer of 4-isopropenylphenol and maleic anhydride, modified with ethylenediamine, formaldehyde and epoxy resin;
- a cooligomer obtained on the basis of 2-propenylphenol and formaldehyde modified with maleic anhydride;
- a cooligomer obtained on the basis of 4-isopropenylphenol-maleic anhydride-styrene, modified with formaldehyde and ethylenediamine.

- element analysis of the process using the known ICP-MS 7700e device and comparison of the results;
- the efficiency of the sorption process, as well as the regeneration of the sorbents using mineral acids and reuse of them.

Research methods. A variety of modern analysis methods have been employed in the execution of the scientific research work, including (IR and NMR spectroscopy, differential thermal analysis, X-ray fluorescence analysis, photocalorimetry, X-ray diffraction, scanning electron microscopy, ICP-MS, etc.).

The main provisions defended. Polyfunctional sorbents have been obtained by polycondensation reactions of alkylphenols (2-propenyl, 4-isopropenylphenols) with formaldehyde and amines, as well as the modification of the resulting functional oligomers with vinyl monomers, formaldehyde, and amines. The obtained polyfunctional sorbents were tested for removing radionuclides and heavy metals from model water systems and oilfield formation water.

Scientific novelty of the dissertation:

– the systematic studies on the modification and functionalization of alkylphenol-based oligomers (4-isopropenylphenol, 2-propenylphenol) with industrially produced compounds (maleic anhydride, acrylonitrile, styrene, ethylenediamine, diethylenetriamine, epoxy resins, formaldehyde) has been conducted. As a result of the studies, new types of functional

ecological, thermally stable, synthetic "cross-linked" polymer sorbents have been synthesized, containing active functional groups (-NH-, -CONH-, -OH, -COOH, =C=O) that can form complexes with metals in aqueous systems and participate in exchange reactions;

- the synthesized polyfunctional "cross-linked" polymers have been systematically studied as sorbents for uranyl and lead ions in model systems;
- the sorbents synthesized have been systematically tested for removing metals from oilfield formation waters obtained from various oil fields.

Theoretical and practical value of the work. The synthesized various structurally different thermoresponsive "cross-linked" polymers can be used in the purification of aqueous systems containing radionuclides, heavy metals, and other toxic substances., Several sorption centers in their composition allows participation in chemisorption process and results in effectiveness due to complex formation.

The sorbents obtained by modifying 4-isopropenylphenol-phenol-formaldehyde cooligomer with maleic anhydride (IV) and acrylonitrile (VI) showed optimal conditions for uranium ion adsorption in a model system, with an adsorption degree (R) of 94,0 % and static sorption capacity (SST) of ~ 75,2 mg/g for the IV sorbent, an R of ~ 93 % and SST of ~ 210 mg/g for the VI.

Effectiveness of the “cross-linked” polymer synthesized based on 4-isopropenylphenol – formaldehyde – 4-(1-methyl-1-dimethoxyphosphorylethyl) phenol as a sorbent of uranyl ions at pH 7 is R = 89%, SST = 229 mg/g.

The synthesized "cross-linked" polymers with different structures are thermally stable (up to 350-600°C), and their mechanical properties do not change during the sorption process. Adsorbed uranyl ions can be easily desorbed with mineral acids (H_2SO_4, HCl), and they are reusable in 10-12 times.

Additionally, the synthesized "cross-linked" polymers have demonstrated high effectiveness in not only solid solutions but also in very dilute solutions (e.g., in oil-field drilling waters, capturing trace

amounts of various cations). The highest sorption capacity was demonstrated by the cooligomer based on 4-isopropenylphenol-maleic anhydride modified with ethylenediamine, formaldehyde, and epoxy resin (XVI). Thus, the efficiency of the sorption processes is almost up to ~ 98-100 %.

These types of synthesized "cross-linked" polymers provide the purification of the environment from other toxic substances and contribute to the solutions of the issues set by "green chemistry".

Approbation and publication. The innovations and results of the dissertation work were presented and discussed at various international and local conferences, as well as published in indexed journals, including:

- The XIII International Scientific Conference on "Current Problems of Chemistry" dedicated to the 96th anniversary of the birth of national leader Heydar Aliyev (Baku, 2019);
- The International Scientific on "Current Problems of Modern Chemistry" Conference for master's students and young researchers dedicated to the 90th anniversary of academician Y.H. Mammadaliyev (Baku, 2019);
- The I International Scientific Conference for PhD students, master's students and young researchers dedicated to the 97th anniversary of the birth of national leader Heydar Aliyev (Baku, 2020);
- The 4th International Novruz Conference on Scientific Research (Baku, 2021);
- The II International Scientific Conference for students and young researchers dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev (Baku, 2021);
- The XIV International Scientific Conference on "Current Problems of Chemistry" for PhD students, master's students and young researchers dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev (BSU, 2021);
- the III International Scientific Conference for students and young researchers dedicated to the 99th anniversary of the birth of national leader Heydar Aliyev (BHOS, 2022);

- "Chemistry and Chemical Technology" Republican Scientific Conference dedicated to the 99th anniversary of the birth of national leader Heydar Aliyev (BSU, 2022);
- The Proceedings of the 8th International Scientific and Practical Conference "Global and Regional Aspects of Sustainable Development" (Copenhagen, Denmark, 2023);
- The Proceedings of the 2nd International Scientific and Practical Conference "Innovative Development in the Global Science" (Boston, USA, 2023);
- The 6th International Scientific and Practical Conference "Scientific Community: Interdisciplinary Research" (Hamburg, Germany, 2023);
- The II International Scientific and Practical Conference "Theoretical and Practical Perspectives of Modern Science" (Stockholm, Sweden, 2023);
- Vysshaya shkola: Nauchnye issledovaniya (Higher School: Scientific Research), the Interuniversity International Congress "Higher School: Scientific Research" (2023);
- The Republican Scientific Conference "Modern Approaches in Chemistry and Chemical Technology" dedicated to the 80th anniversary of the Department of Petroleum Chemistry and Chemical Technology (Baku, 2023).

29 scientific works were published on the subject of the dissertation, including 8 articles and 21 conference papers.

The name of the institution where the dissertation work was performed. The dissertation work was carried out in the "Alkenylphenol Chemistry" Scientific Research Laboratory, operating under the Department of Petroleum Chemistry and Chemical Technology at Baku State University, and in the research laboratory under the Nuclear Research Department of the Innovation and Digital Development Agency.

The total volume of the dissertation indicating the volume of structural sections. The dissertation work is 200 pages long, consists of introduction, 4 chapters, conclusion, 174 references and abbreviations. The dissertation is 190444 characters long (excluding figures, tables, graphs and a list of references). Introduction – 13490,

chapter I – 49870, chapter II – 32540, chapter III – 73152, chapter IV – 18033, conclusion – 3359 characters. The dissertation contains 44 tables and 75 figures that present the results.

The first chapter (Literature Review) is dedicated to the synthesis of sorption materials based on phenols, alkenylphenols, maleic anhydride, acrylonitrile, styrene, and other reagents in recent years, study of their functional properties, and the removal of heavy metals and radioactive substances from aqueous systems.

The systematically presented literature materials confirm the relevance of the dissertation topic.

The second chapter describes the characteristics of the reagents, solvents, initiators, and the devices used in the studies, as well as the purification and preparation of substances. It also presents the methodology for obtaining binary and ternary cooligomers and "cross-linked" polymer-sorbents.

The third chapter focuses on the synthesis of functionalized cooligomers containing double bonds and various heteroatoms, studying of some properties and modification products in the sorption of uranyl and lead ions from aqueous systems.

The fourth chapter describes removal of microelements from the oilfield formation water containing specific microelements using four synthesized sorbents IV, XII, XVI and XVIII.

The applicant's personal contribution to the research conducted. The main leading role belongs to the author when setting the issue in front of the dissertation topic, analyzing the recent world literature on the subject, conducting experiments, testing the reaction products as sorbents, preparing papers, and solving other issues set for the research.

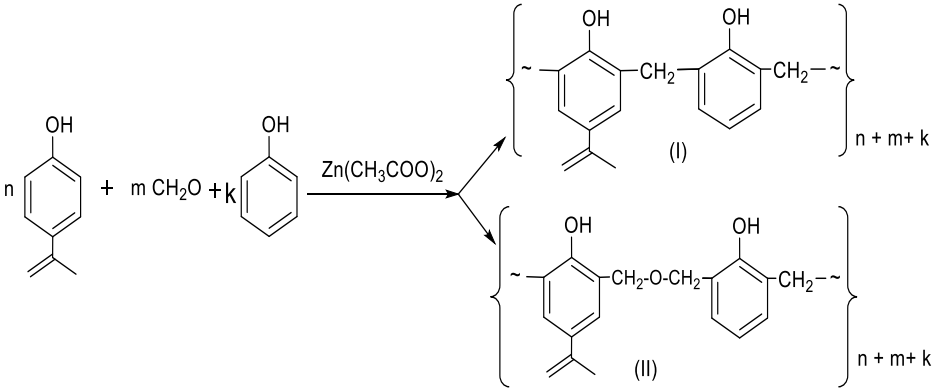
THE MAIN CONTENT OF THE WORK

Natural, hybrid and synthetic polymer sorbents are used in the fight against global environmental issues, processing of industrial waste, mineral raw materials, separation of uranium from ore as nuclear fuel, initial concentration of several radionuclides from technogenic waste and removal of heavy metals and radionuclides from aqueous solutions of various systems. However, in recent years, synthetic polymer sorbents are given more attention due to certain advantages. Synthesis of eco-friendly and economically viable, thermoresponsive polymer sorbents is particularly significant.

Polycondensation of 4-isopropenylphenol with phenol and formaldehyde (I), synthesis of carboxylate-type sorbent on the basis of modification of the resulting oligomer with maleic anhydride (IV), and study of the reaction product as a sorbent

Polycondensation of 4-isopropenylphenol with phenol and formaldehyde (I) was carried out in the presence of a zinc acetate catalyst. The reaction results in obtaining a novolak-type (linear structure) unsaturated phenol-formaldehyde copolymer with a yield of 96.0-96.5% in the compositions (I, II).

Reaction scheme is as follows:

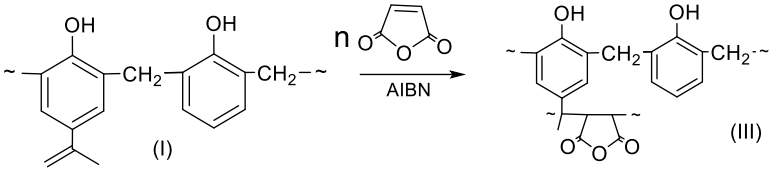


The molecular weight and molecular weight distribution (MWD) of the synthesized cooligomer were determined by gel chromatography. The analysis results reveal that the average

molecular weight (M_w) of the cooligomer was 740, the number-average molecular weight (M_n) – 475, and the M_w/M_n ratio – 1,56. The maximum retention volume (V_R) is in the range of 8.0-10, where a cooligomer with a relatively high average molecular weight (~5000) was also obtained.

The synthesis of the I cooligomer was modified with maleic anhydride via a free radical mechanism.

The reaction scheme is as follows:



Modification process was systematically studied by examining various factors, including temperature, the nature of the initiators, reaction time, and the effect of temperature (thermal) without using an initiator.

The optimal conditions were determined as follows on the basis of the obtained results: 1 % concentration of AIBN initiator, 80°C temperature, 4-5 hours reaction time; and 0,5 % concentration of DTBP initiator, 140°C temperature, 4-5 hours reaction time.

The results from the study prove that modification of the cooligomer can be carried out thermally at 140°C for 5 hours without the use of any initiators.

For the purpose of clarifying the chemistry of crosslinking process, the cooligomer (I) was mixed with maleic anhydride and cross-linked in the presence of AIBN initiator. The process was studied using a derivatographic method, heating the mixture up to 130°C. According to the results, after the mixture reaches 130°C, a characteristic peak indicating the occurrence of an exothermic reaction (polymerization) was observed. This confirms the participation of the initiator and formation of radical chains between the maleic anhydride and the double bonds in the copolymer, as well as the radicals generated from the breakdown of methylene ether groups. Therefore,

the derivatographic study confirms the results of the laboratory experiments.

One of the most important requirements for synthetic polymer sorbents is their thermal stability and environmental safety. Therefore, thermal stability and weight stability of the "cross-linked" polymer as a sorbent were determined through derivatographic analysis.

The optimal conditions for the modification of the I cooligomer with maleic anhydride were determined using MATLAB-6 computer program, based on the laboratory research results to ensure maximum efficiency.

The results proved that in crosslinking process in the presence of DTBP, the maximum yield of the polymer at a temperature 140°C for 5 hours at ~ 97,0 % , in the presence of AIBN, the maximum yield was obtained at a temperature 80°C for 5 hours at ~ 96,2.

The obtained "cross-linked" polymer was studied as a carboxylate-type sorbent (polymer IV) by performing hydrolysis with water at 100°C using a known method. The structure of the IV polymer was studied using IR spectroscopy.

Further, the "cross-linked" polymer was studied as a sorbent for uranyl ions in model systems. The effect of the environment's pH

on the sorption parameters was studied, and it was determined that the maximum sorption efficiency (94 %) occurs at pH = 5-6.

The maxima and stationary regions observed at pH 8 during the sorption process are explained by the sorption of $(UO_2)_3(OH)_5^+$ and $(UO_2)_3(OH)_7^+$ ions by the sorbent. At pH > 10, electron-acceptor centers are present on the surface of the sorbent.

The equilibrium between the uranyl ions and the sorbent can be clearly represented using the Freundlich isotherm.

$$lgq_e = lgK_F + \frac{1}{n} lgC_e$$

Here, $C_e -$ represents the uranium concentration after sorption, mg/l; $q_e -$ is the amount of uranium sorbed per unit mass of sorbent, mg/g; $K_F -$is the sorption capacity constant.

The dependence of the sorption capacity (q_e) on Ce can be explained using the Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{a_L}{K_L} C_e, \qquad q_e = \frac{Q_{max} a_L C_e}{1 + a_L C_e}.$$

where: $K_L -$ is the sorption constant of the sorbent, l/mg; , $a_L -$ is a constant dependent on the sorption energy, l/mg; $Q_{max} -$ is the maximum sorption capacity of the sorbent, mg/g.

The obtained results are set into table 1.

Table 1

Results of calculating the Freundlich isotherm

A_0, Bk/l	A, Bk/l	C_0, mg/l	C_e, mg/l	lg C_e	q_e, mg/g	lgq_e	C_e/q_e
1,2	0,06	2,4	0,12	–0,92	2,28	0,36	0,053
2,4	0,21	4,8	0,42	–0,38	4,38	0,64	0,096
3,6	0,4	7,2	0,8	–0,10	6,4	0,81	0,125
14,4	3,2	28,8	6,4	0,81	22,4	1,35	0,286
28,8	6,9	57,6	13,8	1,14	43,8	1,64	0,315
43,2	12,6	86,4	25,2	1,40	61,2	1,79	0,412
57,6	19,5	115,2	39	1,59	76,2	1,88	0,512
86,4	39,4	172,8	78,8	1,90	94	1,97	0,838
115,2	64,7	230,4	129,4	2,11	101	2,00	1,281

The Freundlich and Langmuir isotherms are shown in Figures 1 and 2.

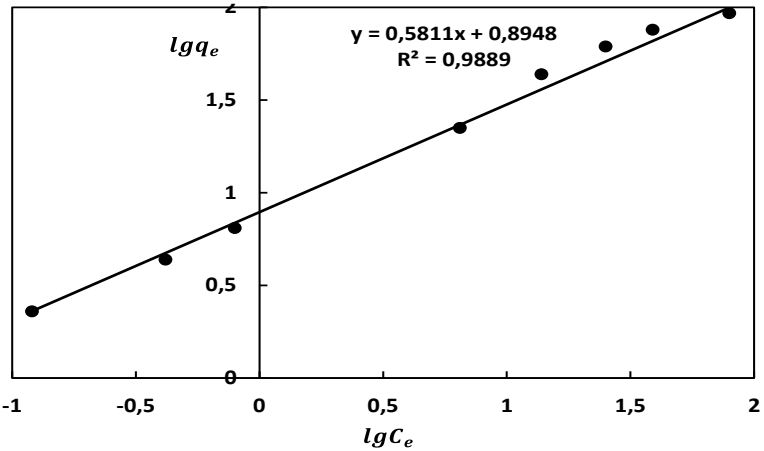


Figure 1. Freundlich isotherm of the sorption process of polymer IV

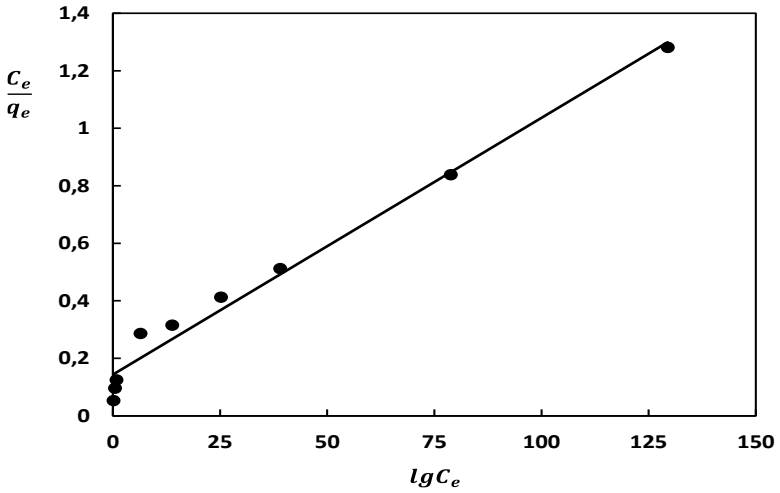


Figure 2. Langmuir isotherm of the sorption process of polymer IV

For the purpose of determining the efficiency of the sorbent, the distribution constant (R_L) was calculated using the following formula:

$$R_L = \frac{1}{1 + bC_0}$$

Where:

b (a_L) is the Langmuir constant; C_0 – is the initial concentration of uranyl ions in the solution, mg/l.

Table 2 presents Langmuir and Freundlich isotherm constants.

Table 2

Langmuir and Freundlich isotherms constants

Freundlich			**Langmuir**			
n	K_F	R^2	K_L, l/g	a_L, l/mg	R_L	Q_{max}, mg/g
1,72	7,8	0,989	6,96	0,06	0,07	101

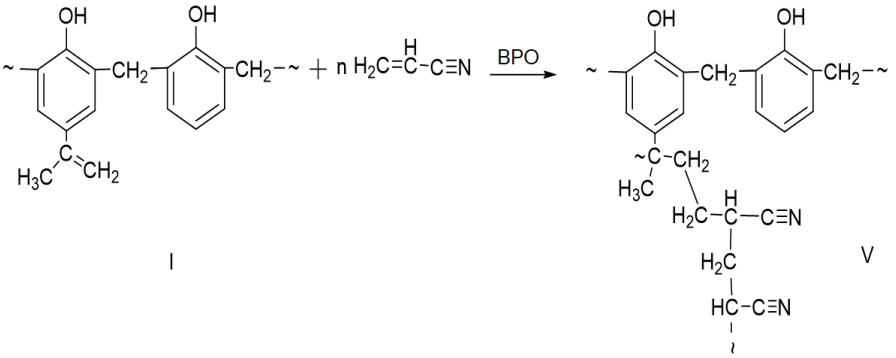
As is evident from table 2, the value of R_L is within the range of 0-1, indicating the presence of the sorption process. Additionally, 1/n = 0,5829 within the 0-1 range proves that the surface of the sorbent is sufficiently heterogeneous.

To achieve the maximum retention of uranyl ions, tests were carried out at high concentrations (300 mg/l) and various amounts of sorbent (0,2-2 g/l). When the concentration of the sorbent in the liquid phase was increased from 0,2 to 10 g/l, the sorption degree of uranyl ions increased. Sorption stabilized in the 5-10 g/l range.

Modification of 4-isopropenylphenol, phenol and formaldehyde (I) cooligomer with acrylonitrile (V) and study of the reaction product as a sorbent

First, the influence of various factors, including temperature and time, on the modification of the sorbent I with acrylonitrile was studied. Based on the results obtained, the optimal conditions for the reaction of the sorbent I with acrylonitrile in the presence of BPO were found to be a temperature of 90°C, a reaction time of 5 hours, and a initiator concentration of 1,2 % by total mixture weight.

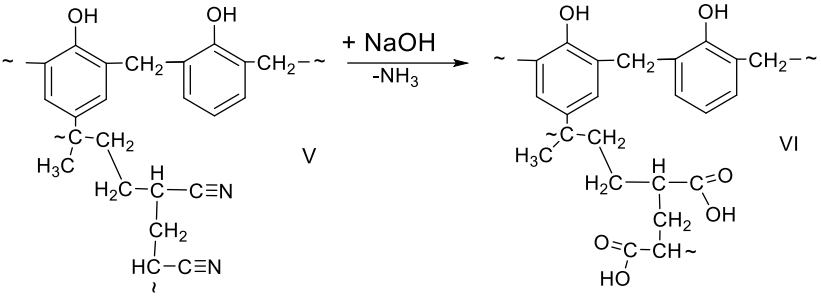
The reaction scheme is as follows:



The structure of the obtained "cross-linked" polymer was studied using IR spectroscopy (Figure 3).

For the purpose of obtaining a carboxylate-type sorbent, its hydrolysis was carried out in the presence of an alkali.

Scheme of the hydrolysis reaction:



The structure of the hydrolyzed product was also studied using IR spectroscopy (Figure 3).

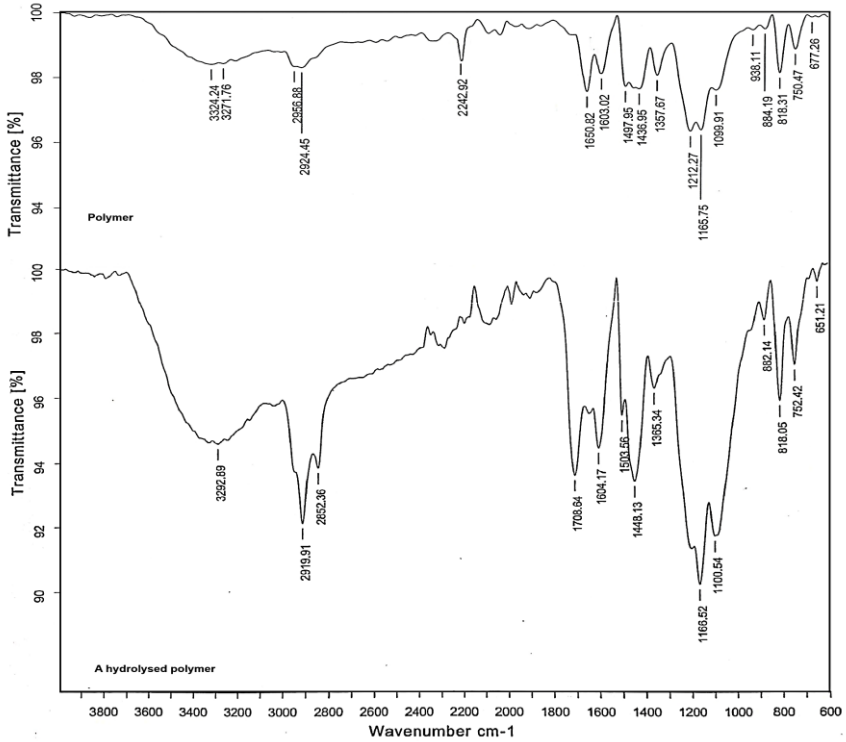


Figure 3. IR spectrum of polymer V before (V) and after hydrolysis (VI)

The obtained "cross-linked" polymer was studied as a sorbent for uranyl ions in a model system. To determine the sorption capacity of the nitrile group (−C≡N), the effect of pH on the sorption parameters of UO_2^{2+} ions was studied under static conditions (temperature 25°C, sorption time 24 hours, initial concentration of UO_2^{2+} ions 134,5 mg/l). It was determined that at pH 7, R = 64,4% and SST = 155,7 mg/g.

The hydrolyzed VI polymer has been investigated as a sorbent. The effect of the pH of the medium on the sorption process was studied, and the obtained results are shown in fig. 4. The results show that the sorption of UO_2^{2+} ions from the aqueous systems (~ 93-90%) is achieved when the pH of the medium is 7 or 9. At this point, the static sorption capacity is approximately ~ 210-202 mg/g.

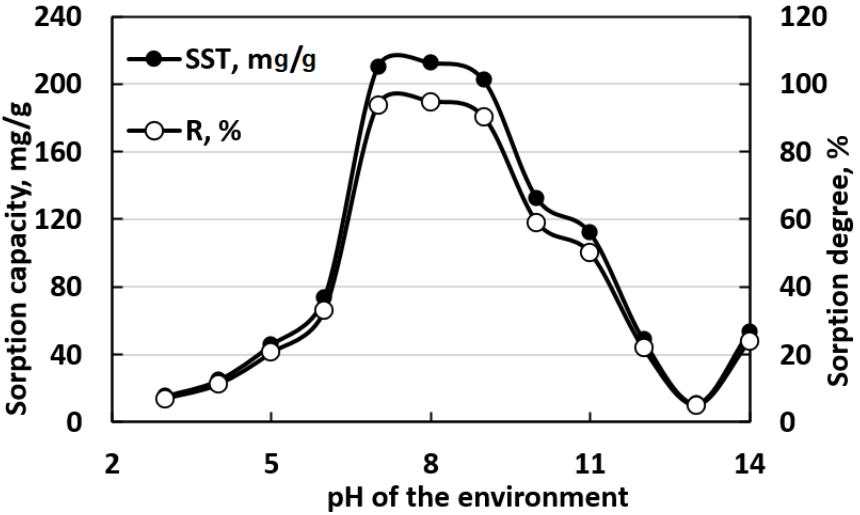


Figure 4. Dependence graph of the sorbent (VI) sorption degree and sorption capacity on pH of solution

Therefore, it becomes clear that the lower sorption parameters of the hydrolyzed sorbent and the higher ones after hydrolysis are related to the presence of carboxyl groups in the "cross-linked" polymer.

Under static conditions, the effect of sorbent sorption time on the sorption process of ions at pH 7 was studied. As a result of the

studies it was determined that the process occurs in two phases. In the first 60 minutes, the sorption process is fast, which can mainly be explained by the chemisorption process. As the time increases, a slower diffusion process occurs. After 24 hours, the sorption degree reaches its maximum value of ~ 93,5%, and the sorption capacity is ~ 209,6 mg/g.

First-order, pseudo-second-order, and second-order kinetic models were applied to determine the kinetics of the sorption process. The sorption of uranyl ions from an aqueous solution onto the "cross-linked" polymer in two stages is clearly shown in fig. 5. This indicates that the sorption follows a second-order kinetic model. In the second-order kinetic dependence, the fast first phase - chemisorption has a regression coefficient (R^2)=0,96, while the slower diffusion phase is R^2=0,98. The first-order sorption kinetic graph was plotted for the 0÷60 minute time interval and accurately reflects this time interval only (Figure 5).

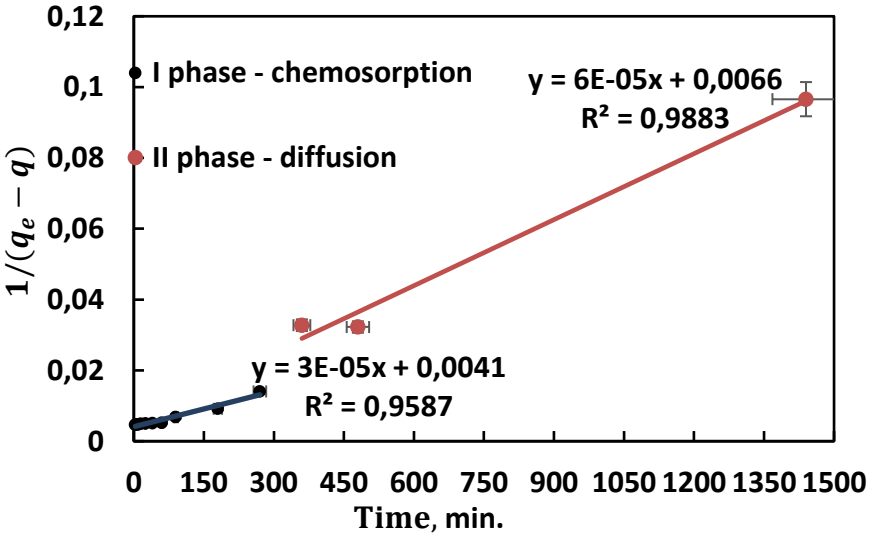


Figure 5. Graph of the first-order degree constant for uranium sorption with polymer VI

The kinetics of uranium sorption is more clearly reflected due to the second-order and pseudo-second-order kinetic graphs covering the entire time interval and providing information about the sorption mechanism. Thus, the most suitable model for representing uranium sorption by the sorbent has been identified as the pseudo-second-order degree constant model (Figure 6).

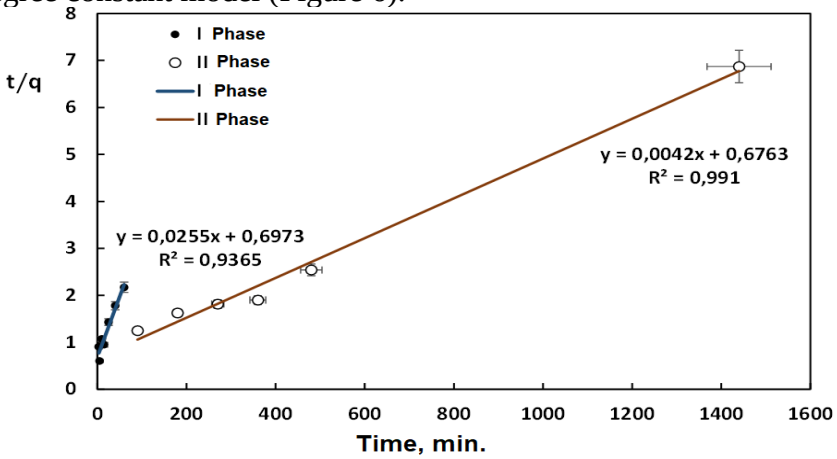


Figure 6. Graph of the pseudo-second-order degree constant for uranium sorption with polymer VI

The effect of the initial concentration uranyl ions on the sorption degree and sorption capacity (R, % and SSC, mg/g) has also been studied (table 3). The best results in terms of the sorption degree of uranyl ions from the aqueous solution were obtained at uranyl ion concentrations of 74,4–140,6 mg/l (R = 90-93 %, SSC = 116–211 mg/g).

Table 3

Effect of uranyl ion concentration on R, % and SSC, mg/g (pH 7)

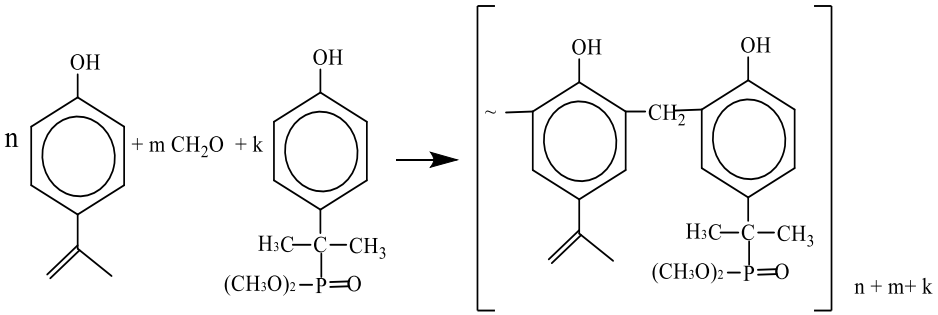
A_0 , Bk/l	A, Bk/l	C_0, mg/l	C_e, mg/l	q_e, mg/g	R, %
22,6	9,3138	38,0	10,1	46,5	73,4
27,1	10,529	45,6	8,9	61,2	80,5
44,3	11,001	74,4	4,8	116,0	93,5
83,6	13,363	140,6	13,8	211,3	90,2
117,4	31,114	197,4	21,1	293,8	89,3
154,8	54,803	260,1	70,2	316,5	73,0

Nitric and hydrochloric acids were used to for the desorption of uranyl ions. The study determined that as the concentration of both acids increased, the desorption of uranyl ions from the sorbent increased. Specifically, desorption with nitric acid reached 96,1%, while with hydrochloric acid, it was 92,2 %.

Obtaining 4-isopropenylphenol, formaldehyde, and 4-(1-methyl-1-dimethoxyphosphorylethyl)phenol cooligomer (VII) and Its modification with acrylonitrile (VIII) and and study of the reaction product as a sorbent

Polycondensation reaction of 4-(1-methyl-1-dimethoxyphosphorylethyl) phenol with formaldehyde and 4-isopropenylphenol was carried out, resulting in a three-component oligomer. The structure of the cooligomer was determined by ^{1}H NMR and IR spectroscopy methods. Then, the molecular weight and molecular weight distribution of the reaction product were studied using gel chromatography (M_W = 2225, M_n = 750).

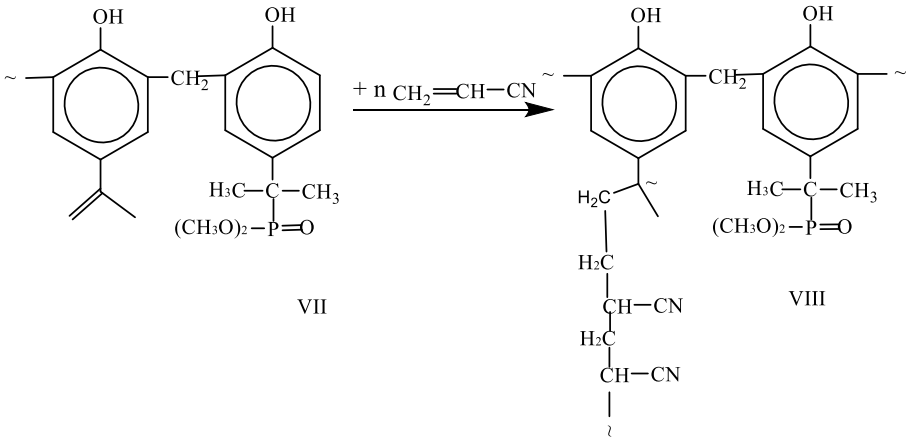
Reaction scheme is as follows:



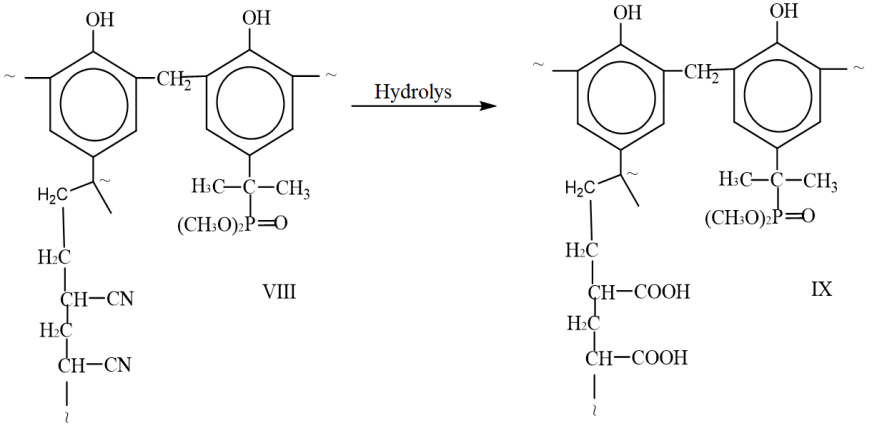
VII

The obtained cooligomer was modified (crosslinking) with acrylonitrile.

Reaction scheme is as follows:



A new type of sorbent was obtained by hydrolysis of polymer VIII in the presence of alkali:



For the purpose of studying the sorption properties of the IX polymer, its sorption process was studied in aqueous systems containing uranyl ions at various concentrations for 24 hours at room temperature.

The effect of various factors on sorption parameters was studied. As is evident from Figure 7, the sorption parameters reveal the highest results at pH 7 (R = 89 %, SST = 229 mg/g). The effect of time on the found optimal pH was studied and the optimal sorption time was determined to be 24 hours.

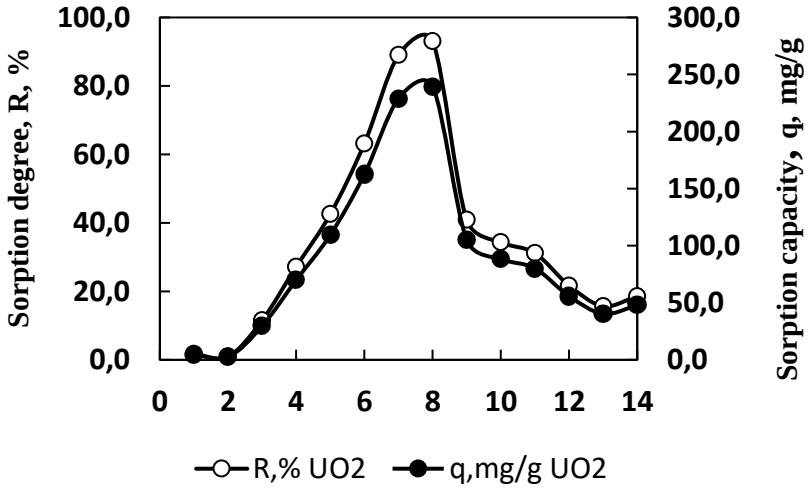


Figure 7. Graphical representation of the effect of pH on the sorption degree and sorption capacity of the IX polymer

Synthesis of carboxylate-type sorbent (XII) on the basis of 2-propenylphenol formaldehyde two-component polycondensation product (X) and and study of the reaction product as a sorbent

There are two approaches for obtaining a polymer sorbent based on 2-propenylphenol, formaldehyde and maleic anhydride:

a) the first approach involves the synthesis of cooligomer of 2-propenylphenol and maleic anhydride, followed by polymerization (condensation) of the obtained cooligomer with formaldehyde;

b) in the second approach, 2-propenylphenol undergoes polycondensation with formaldehyde in the presence of an alkaline medium, and the resulting polycondensate (oligomer) is modified with maleic anhydride in the presence of a catalyst.

Ecological, technological and scientific analysis of both approaches shows that the second approach might be more beneficial. Specifically, the removal of unreacted formaldehyde from the obtained cooligomer through polycondensation of 2-propenylphenol with formaldehyde in the presence of an alkaline is relatively easier. On the other hand, the obtained novolac can also exhibit sorbent

properties. Additionally, the process of modifying the obtained cooligomer with maleic anhydride in the presence of AIBN initiator (60-70°C) is more technologically feasible.

Considering the advantages of the second variant, the first stage of the study involved the polycondensation of 2-propenylphenol with formaldehyde. The structure of the obtained cooligomer was studied using IR spectroscopy.

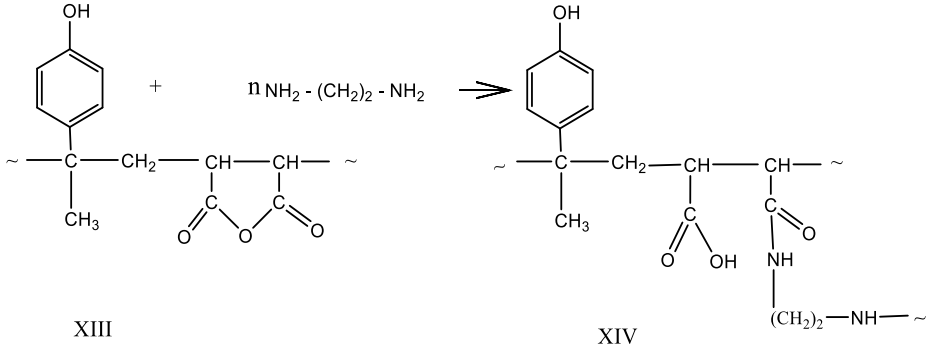
In order to obtain a carboxylate-type sorbent, its reaction with maleic anhydride (crosslinking) was carried out and followed by hydrolysis.

The sorption tests of the XII polymer as a uranyl ion sorbent were performed, and the effect of pH of the medium, initial solution hardness and process duration on the sorption parameters was studied. In this case, with an initial concentration of uranyl ions of 155.2 mg/l in a pH 6 medium, at room temperature, and for 24 hours, R=93%, SSC=240,7 mg/g was obtained.

Modification of 4-isopropenylphenol-maleic anhydride (XIII) based cooligomer with ethylenediamine (XIV) and study of the reaction product as a sorbent

The cooligomer (XIII) of 4-isopropenylphenol with maleic anhydride was synthesized by a known method. Then, the modification of the XIII cooligomer with ethylenediamine was carried out to obtain a "cross-linked" polymer. The structure of the obtained polymer was studied using IR spectroscopy.

Reaction scheme is as follows:



XIII XIV

The "cross-linked" polymer was studied as a sorbent for the removal of uranyl ions from model aqueous solutions. The sorption parameters showed that the maximum result was achieved at pH 7 (R = 76,9%, SSC = 197,7 mg/g). The effect of time at the optimal pH was studied and sorption equilibrium was determined to be reached within 6-8 hours.

Modification of 4-isopropenylphenol-maleic anhydride (XIII) based cooligomer with formaldehyde and diethylenetriamine (XV) and study of the reaction product as a sorbent

Modification of the 4-isopropenylphenol-maleic anhydride cooligomer with formaldehyde and diethylenetriamine resulted in a new type of polymer-sorbent containing several functional groups capable of coordinating with metals. The structure of the obtained polymer was studied using IR spectroscopy. The synthesized "cross-linked" polymer was studied as a sorbent for the removal of uranyl ions from model aqueous systems. The effects of pH of the medium, sorption time, and concentration of uranyl ions on the process were studied.

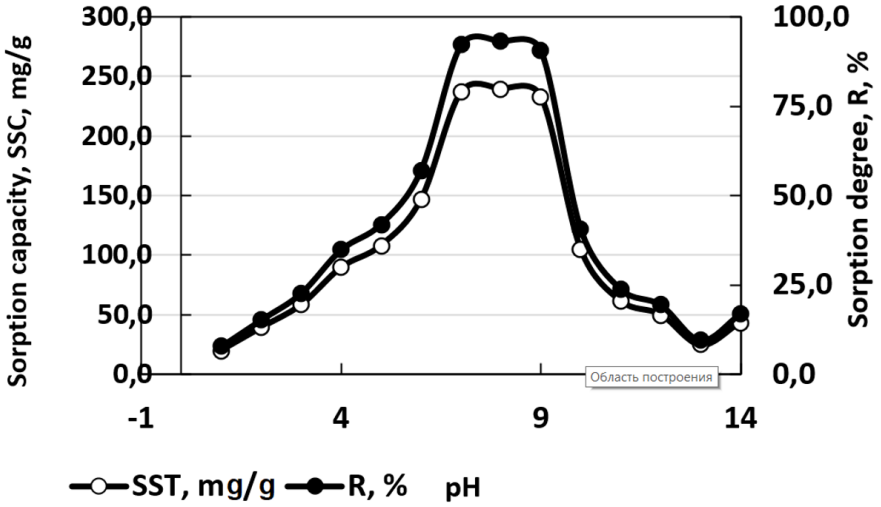


Figure 8: Effect of pH of the medium on sorption parameters of the sorbent (XV)

As shown in fig. 8, the sorption degree was 92,2% and 90,7% at pH 7 and 9, respectively. The sorption capacity was 237,0 mg/g at pH 7 and 233,3 mg/g at pH 9.

The optimal sorption time for the process at the found optimal pH was determined to be 4-6 hours. The effect of the initial concentration of UO_2^{2+} ions on the process was studied. The XV sorbent showed high efficiency at both low and high concentrations of UO_2^{2+}ions.

4-Isopropenylphenol-maleic anhydride (XIII) based composite sorbent (formaldehyde, ethylenediamine, epoxy resin) (XVI) and and study of the reaction product as a sorbent

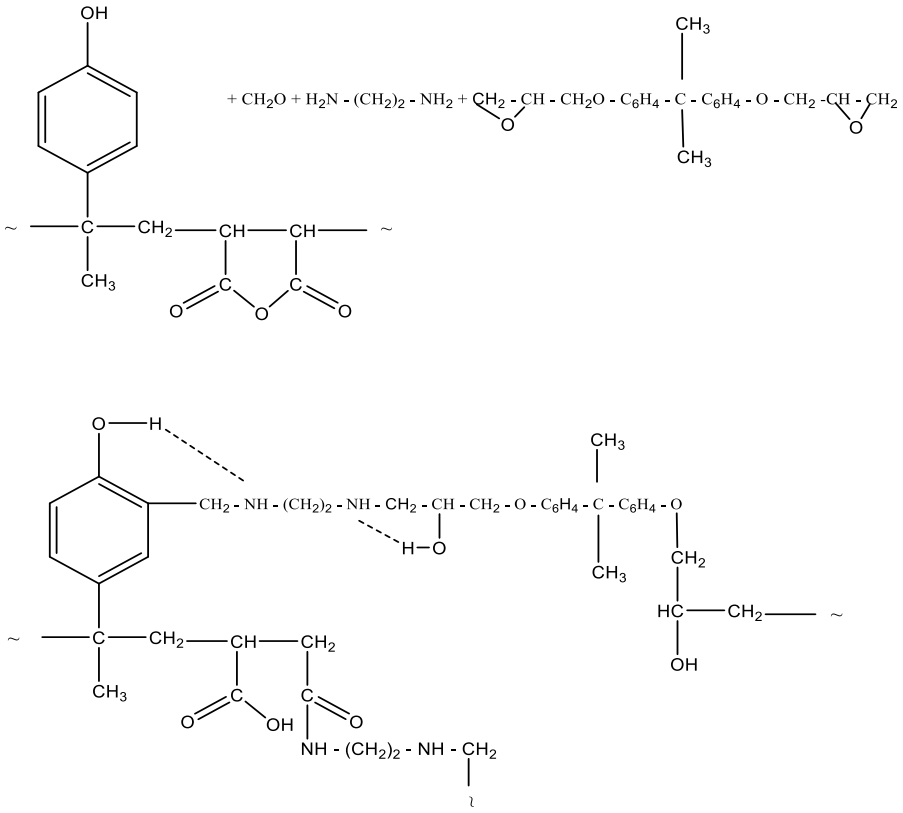
Previously synthesized sorbents containing carboxyl, amine, amide and hydroxyl active centers in their structure were modified to introduce quasi-aromatic and five-membered intramolecular hydrogen bonding fragments into the polymer molecule via hydrogen bonding. As a result, a new polyfunctional, thermally stable polymer sorbent was synthesized. The polyfunctional polymer sorbent was obtained by modifying the 4-isopropenylphenol-maleic anhydride cooligomer with formaldehyde, ethylenediamine, and epoxy resin. The structure of the obtained polymer was studied by IR spectroscopy.

A composition-based polyfunctional polymer was studied as a sorbent for uranyl ions. The effect of environmental pH and sorption time on sorption parameters was studied. The optimal pH for the sorbent was found to be 7 and 9. The effect of time on the sorption process at the identified optimal pH was studied and maximum results were obtained in a very short time. Specifically, after 0,25 hours, the sorption degree was 98,1%, and the sorption capacity was 252,8 mg/g.

The results obtained under the influence of the initial concentration of uranyl ions were obtained above ~ 80% even at the lower and higher concentrations taken (38 – 260 mg/g).

Thus, unlike the previously studied sorbents, sorbent XVI has high performance in liquid and solid uranyl ion solutions in a very short time.

Reaction scheme is as follows:



Modification of 4-isopropenylphenol-maleic anhydride-styrene (XVII) based cooligomer with ethylenediamine (XVIII) and study of the reaction product as a sorbent

Taking into account the requirements of the day, a polyfunctional"cross-linked" polymer sorbent containing active groups was synthesized by the radical polymerization of 4-isopropenylphenol, styrene and maleic anhydride using a known method. The structure of the synthesized cooligomer was studied using IR spectroscopy.

The synthesized cooligomer was modified with ethylenediamine and the structure of the obtained polymer was studied using IR spectroscopy.

The obtained polymer was tested as a sorbent for removing uranyl ions from model aqueous solutions, and the optimal sorption parameters of the XVIII sorbent were determined as follows: R = 84,2%, SSC = 216,5 mg/g. The optimal sorption time has been determined to be 4-6 hours.

The IR spectrum of the sorbent sample was recorded after sorption and compared with the spectrum before sorption. The changes observed in the absorption bands indicate the presence of various types of interactions (complexation, exchange) between carbonyl, amide, amino groups, five- and six-membered intramolecular hydrogen bonds and uranyl ions as a result of sorption.

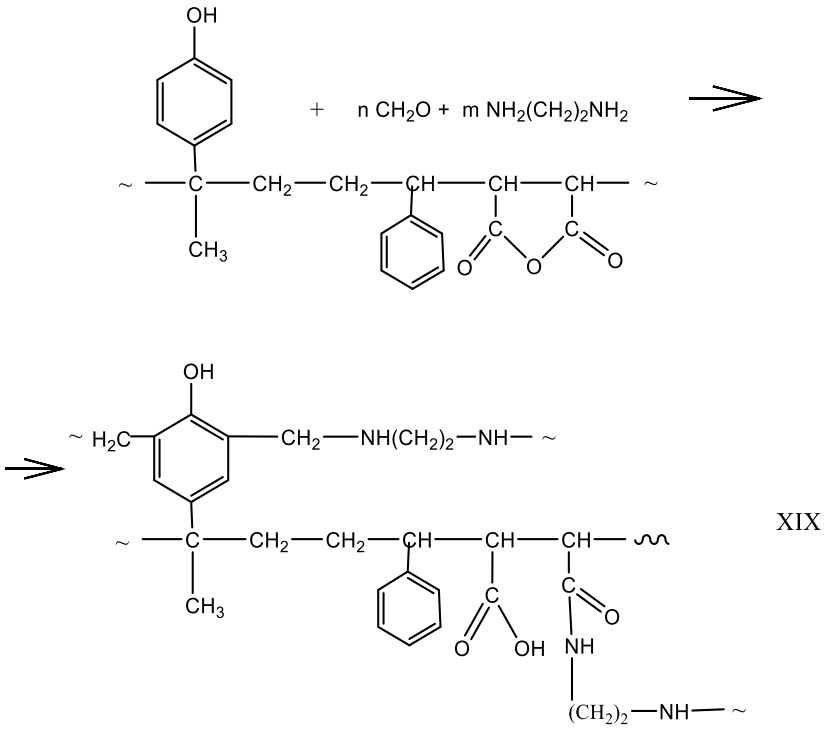
Modification of the 4-isopropenylphenol-maleic anhydride-styrene (XVII) based cooligomer with formaldehyde and ethylenediamine (XIX) and study of the reaction product as a sorbent

The modification of the XVII cooligomer with formaldehyde and ethylenediamine was carried out, and the resulting modification was studied as a sorbent. The XVII cooligomer was modified with formaldehyde and ethylenediamine and studied as a sorbent. The structure of the obtained polymer was studied using IR spectroscopy.

The effect of various factors on the sorption parameters was studied, and the optimal pH of 7 and a time of 1 hour resulted in a sorption degree (R) of 98% and a sorption capacity (SSC) of 252,0 mg/g.

After the study, the IR spectrum of the sorbent was recorded and compared with its IR spectrum before sorption. The changes observed in the absorption bands characterizing functional groups, suggest that the sorbent forms a complex with uranyl ions during the process.

Reaction scheme is as follows:



XIX

Modification of 4-isopropenylphenol-maleic anhydride-styrene-(XVII) based cooligomer with formaldehyde and diethylenetriamine (XXI) and study of the reaction product as a sorbent

The number of functional groups in the "crosslinking" product (XXI) obtained by the modification of the XVII cooligomer with formaldehyde and diethylenetriamine, increases due to the presence of amine groups. The structure of the obtained polymer was studied using IR spectroscopy.

The XXI polymer has been studied as a sorbent of uranyl ions. The effect of various factors on the sorption parameters was study, and the optimal conditions for the process were determined to be pH 7 and 9, with a sorption time of 1 hour.

The results of the studies proved the highest efficiency of the sorbent XVI among the synthesized sorbents. However, other sorbents

are also quite effective. Taking this into account, the studied sorbents may be recommended for application.

Study of Pb^{2+} ion of polymers obtained by the modification of 4-isopropenylphenol-maleic anhydride-based composition (XVI) and 4-isopropenylphenol-maleic anhydride-styrene-based cooligomer with formaldehyde and ethylenediamine (XIX)

The effect of pH on the sorption of Pb^{2+} ions by the XVI sorbent was studied. The highest result was obtained in a neutral environment (pH 7). The sorption degree was 90,5% at pH 7.

Next, the effect of Pb^{2+} ion concentration (ranging from (2-80)·10^{-4} M) on the sorption process using XVI sorbent was studied. The highest sorption capacity occurred at both 60·10^{-4} and 80·10^{-4} concentrations of the metal ion. Specifically, at these concentrations, the sorption capacity of the XVI sorbent was 236,1-237,1 mg/g.

Optimal pH values were found 7-8 for the sorption process using the XIX sorbent. Under these conditions, the sorption degree was 85,9 % at pH 7 and 85,1 % at pH 8.

After determining the effect of pH on the sorption process, the effect of Pb^{2+} ion concentration (ranging from (2-80)·10^{-4} M on the sorption process was studied. It was found that the highest sorption capacity occurred at a concentration of 60·10^{-4} M. The sorption capacity at this concentration was 222,9 mg/g.

Results prove effectiveness of both sorbents for the sorption of Pb^{2+} ions.

Sorption process in oilfield formation waters and discussion of the results

The synthesized polyfunctional thermoresponsive synthetic polymer sorbents were used for the characterization, quantitative determination, and purification of radionuclides and heavy metals present in oilfield formation waters. The studies were carried out using drilling waters extracted together with oil from two different oil fields. The waters used for the study were taken from the Lokbatan Field No.

260 named after Amirov (Garadagh district, Oil and Gas Extraction Department (OGED)) and “Bibihaybatneft” Oil and Gas Extraction Department (OGED) Field No. 3. The IV and XVI – for the A oilfield formation water and the IV, XII, XVI, XVIII "cross-linked" polymers were studied as sorbents for the B oilfield formation water.

The results obtained from the studies were analyzed using scanning electron microscopy (SEM), X-ray diffraction and elemental analysis methods.

The analysis conducted using the scanning electron microscope allows for both visual inspection of the sorbent and the spectrum of the absorbed elements. After the sorption process using the IV sorbent in the A oilfield formation water, the results obtained from the analysis using SEM are presented in table 4 and figures 9 and 10.

Table 4 presents the mass and atomic percentages of the elements, and figure 8 presents the overall spectrum taken by the electron microscope.

Figure 9 illustrates the distribution morphology (map) of the elements in the complex formed by the IV sorbent with the microelements in the A oilfield formation water.

Table 4

Results of the scanning electron microscope study of the sorption process using the iv sorbent in oilfield formation water

Element	Mass, %	Atom %
C	93,83	97,66
Mg	0,18	0,09
Na	1,25	0,68
Si	1,62	0,72
Cl	0,46	0,16
Ca	0,99	0,31
Fe	1,68	0,38
Cəm:	100,00	

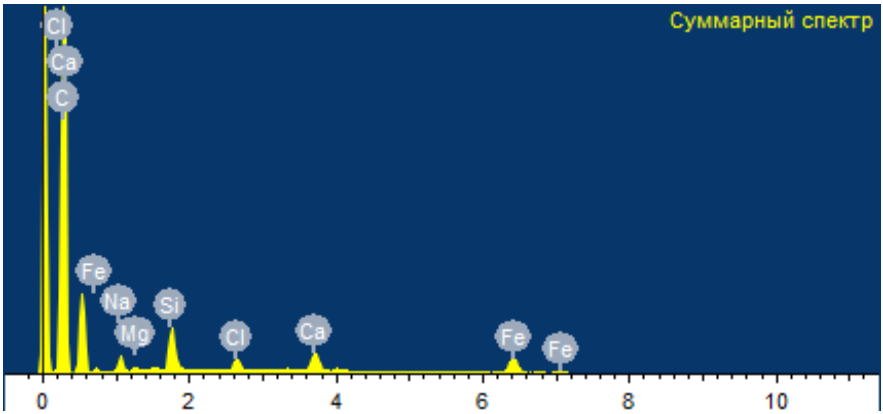


Figure 9. Overall spectrum of the sorption process using the IV sorbent in A oilfield formation water, taken by scanning electron microscope

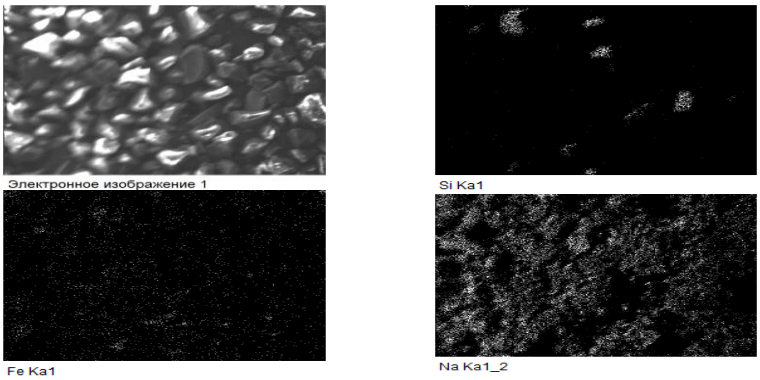


Figure 10. Distribution map of elements in the complex formed by the IV sorbent with the microelements in A oilfield formation water

Subsequently, a more precise and sensitive analysis device – the ICP-MS 7300e was used to study the microelement composition for the purpose of determining the amount of microelements sorbed by the sorbent.

The results obtained from the sorption process carried out on sample A with sorbent IV are given in table 5. The table presents the quantities (ppm and %) of metals found in A oilfield formation water both before and after sorption, as well as the sorption degrees.

Table 5

Results of the sorption process using the IV sorbent in A oilfield formation water

Microelements	The amount of microelements in A oilfield formation water, ppm and %		Sorption degree, %
	Before sorption	After sorption	
K	28027,929 (37,2%)	15321,225 (20,3%)	45,3
Na	31291,362 (41,5%)	13541,654 (18%)	56,7
Ca	8160,880 (10,8%)	0	100
Ba	1076,850 (1,4%)	375,433 (0,5%)	65,1
Zn	38,1760 (0,05%)	0	~100
Cu	83,7480 (0,1%)	0,3040 (0,0003%)	99,6
Hg	14,9210 (0,01%)	0,3380 (0,002%)	97,7
Fe	5998,198 (8%)	0	~100
Cr	505,3240 (0,7%)	0,0190 (0,00002%)	~100
Mn	87,7140 (0,1%)	0,1820 (0,0002%)	99,8
As	20,3640 (0,03%)	0,338 (0,0004%)	98,3
Se	63,4280 (0,08%)	0,1820 (0,0002%)	99,7

The description and quantity of metals in B oilfield formation water were analyzed in the same manner, and the corresponding results were obtained.

The IR spectra of the sorbents were recorded after sorption and compared with the pre-sorption IR spectra. The changes in the sorption bands confirm the occurrence of sorption process.

The results from the research indicate that by using the synthesized sorbents as multifunctional, thermoresponsive sorbents, it's possible to purify oilfield formation waters from microelements (Na, K, Ca, Ba, Zn, Cu, Mn, Hg, As, Se, Cd, Fe, Cr, Pb, etc.) by ~91-100%.

Given the economic viability, ecological safety and high activity of the synthesized sorbents in removing metals from aqueous systems, their application in purifying microelements from oilfield formation waters is recommended.

Conclusion

1. Modification of the unsaturated phenol-formaldehyde cooligomer obtained by the polycondensation of homogeneous catalytic degradation products – phenol and 4-isopropenylphenol derived from diphenylpropane (bisphenol A) with maleic anhydride systematically studied and resulted in obtaining a "cross-linked" polyfunctionalpolymer sorbent containing carboxyl and phenolic hydroxyl groups. The sorption process of uranyl ions in model systems was systematically studied and the optimal conditions for the process were identified, the maximum sorption degree and static sorption capacity were found to be R=94,0 % and SSC=75,2 mg/g, respectively [6,9,10].

2. The modification product of the synthesized unsaturated phenol-formaldehyde cooligomer modified with acrylonitrile was hydrolyzed with aqueous alkaline solution and resulted in obtaining a sorbent with the resistance to high temperatures. The sorbent's performance in removing uranyl ions from aqueous systems was studied and the sorption degrees were 93% and 90% at pH 7 and 9, static sorption capacities were 210-202 mg/g, respectively [26].

3. A new type of phosphorus-containing sorbent was synthesized on the basis of the cooligomer obtained by the polycondensation reaction of 4-(1-methyl-1-dimethoxyphosphorylethyl)phenol with formaldehyde and 4-isopropenylphenol and was tested as a

sorbent for uranyl ions. The sorption parameters revealed maximum results at pH 7, where R=89% and SSC=229 mg/g after 24 hours [15, 17].

4. The composite material (six-membered intramolecular hydrogen-bonding) obtained by modification of 4-isopropenylphenol-maleic anhydride cooligomer with ethylenediamine, formaldehyde and epoxy resin and a new type of polyfunctional sorbent containing the fragments forming active metal complexes such as phenolic hydroxyl groups. The sorbent's performance in removing uranyl ions from aqueous systems was systematically studied and optimal conditions were found to be at pH 7 and pH 9. Under the found pH: R=98%, SSC=285 mg/g.
5. The new "cross-linked" product obtained from the polycondensation of 2-propenylphenol with formaldehyde and modification with maleic anhydride was tested as a sorbent for separation of uranyl ions in model systems. The sorbent was found to have a high sorption capacity (R=93%, SSC=212 mg/g) [16].
6. Two synthesized “cross-linked” polymers (XVI and XIX) were studied as sorbents of lead ions in a model system and high results were obtained. Thus, the sorbent XVI have ~ 90,5%, and sorbent XIX ~ 85,9% of sorption degrees under optimal conditions [25].
7. Four synthesized sorbents were used to study the sorption of metals from drilling fluids extracted from two oil fields. It was found that the composite material obtained by modifying the 4-isopropenylphenol-maleic anhydride cooligomer with ethylenediamine, formaldehyde, and epoxy resin provided the highest sorption results. The tested sorbents were able to sorb metals (K^+, Na^+, Ca^{2+}, Zn^{2+}, etc.) up to 100% even in clear solutions.
8. The gel-chromatographic analysis method was used to determine the molecular weight distribution and molecular weights of the cooligomers used in the synthesis of the sorbents. The structures of the sorbents before and after sorption were determined using IR spectroscopy, and differential thermal analysis was performed.

Based on the results of the comprehensive research, it's concluded, that the synthesized polyfunctional synthetic polymers, which are thermally and mechanically durable and possess high sorption capacity, meet the requirements set for modern sorbents from both an economic and ecological standpoint and may be recommended for practical application.

The main results of the dissertation were expressed in the following publications:

1. Bayramov, M.R., Mehdiyeva, G.M., Guliyeva, Sh.J., Gasanova, G.M., Madinov, I.M. Purification of oil drilling waters from uranyl ions using crosslinked copolymer // Dedicated to the 96th anniversary of the national leader Heydar Aliyev, I Republic Scientific Conferences for Students, Baku: April 15-19, 2019, p. 39-40.
2. Bayramov, M.R., Mehdiyeva, G.M., Guliyeva, Sh.J., Naghiyev, C.A., Sadigov, N.M., Hasanova, G.M., Madinov, I.M. Synthesis of new types of sorbents for cleaning water systems from heavy metals // Dedicated to the 96th anniversary of national leader Heydar Aliyev, XIII International Scientific Conference on Actual Problems of Chemistry for PhD candidates, Master's students, and young researchers, Baku: May 15-16, 2019, p. 172-173.
3. Bayramov, M.R., Mehdiyeva, G.M., Guliyeva, Sh.J., Naghiyev, C.A., Madinov, I.M. Synthesis and study of phosphorus-containing unsaturated phenol-formaldehyde oligomer // International Scientific Conference on Actual Problems of Modern Chemistry, dedicated to the 90th anniversary of academician Y.H. Mammadaliyev Institute of Petrochemical Processes, Baku: October 2-4, 2019, p. 232.
4. Farzaliyev, V.M., Mehdiyeva, G.M., Agayeva, M.A., Madinov, I.M., Guliyeva, Sh.J. Synthesis and investigation of

sorbents based on 4-isopropenylphenol-phenol-formaldehyde // Dedicated to the 97th anniversary of national leader Heydar Aliyev, I International Scientific Conference for Students and Young Researchers, Baku: April 6-16, 2020, p. 208-209.

5. Guliyeva, Sh.J., Mehdiyeva, G.M., Bayramov, M.R. Sorbents based on alkenylphenol derivatives // 1st International Nowruz Conference on Scientific Research, Karabakh, Azerbaijan, March 18-21, 2021, p. 428.

6. Bayramov, M.R. Studying the sorption of uranyl ions from aqueous solutions with the structured 4-isopropenylphenol-phenol-formaldehyde copolymer / Musa Bayramov, Gunay Mehdiyeva, Jalal Nagiev, Mahira Agayeva, Shahla Guliyeva // Journal of Physical Chemistry A, 2021, v. 95, No. 4, p. 769-774.

7. Guliyeva, Sh.J., Mehdiyeva, G.M., Gasımova, Sh.Z. Polymer sorbents of uranium ions based on 4-isopropenylphenol // International Conference of Students and Young Researchers Dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev, - Baku: April 13-28, 2021, - p. 54-55.

8. Bayramov, M.R., Mehdiyeva, G.M., Guliyeva, Sh.J., Agayeva, M.A., Maharramov, A.M. Synthesis and study of oligomer based on 2-propenylphenol // XIV International Scientific Conference on the Current Problems of Chemistry Dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev, - Baku: May 25-26, 2021, - p. 124-125.

9. Bayramov, M.R. Synthesis study of triple cooligomers of 4-isopropenylphenol, phenol and formaldehyde structured by maleic anhydride / Musa Bayramov, Abel Maharramov, Gunay Mehdiyeva, Shahla Guliyeva, Mahira Agaeva // Processes of Petrochemistry and Oil Refining, – 2021. v. 22, № 4, – p. 476-485.

10. Bayramov, M.R. Optimization of the structuring process of 4-

isopropenylphenol, phenol and formaldehyde cooligomer with maleic anhydride / Musa Bayramov, Natig Zeynalov, Gunay Mehdiyeva, Mahira Agaeva, Shahla Guliyeva, Misir Javadov, Gulnara Hasanova // Oil Refining and Petrochemicals, – 2021. № 11, – p. 38-42.

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