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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS OF A NOVEL PHOSPHORUS-CONTAINING POLYMER BASED ON STYRENE-BUTADIENE RUBBER, INVESTIGATION OF ITS SORPTION PROPERTIES TOWARD HEAVY METAL IONS AND YEAST FUNGI

Speciality: 2304.01-Macromolecular chemistry

Field of science: Chemistry

Applicant: **Ellada Sabir Karimova**

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The work was performed at at the Department of “Chemistry of High Molecular Compounds” at Baku State University.

Scientific supervisor: Doctor of Chemical Sciences, Professor
Abdulsayid Abdulgamid Azizov

Doctor of Chemical Sciences, Professor
Rasim Mirali Alosmanov

Official opponents: Doctor of Chemical Sciences,
Associate Professor
Fizuli Akper Nasirov

Doctor of Philosophy in Chemistry,
Associate Professor
Samira Mirali Mammadova

Doctor of Philosophy in Chemistry
Farah Mammadali Mammadaliyeva

Dissertation council FD1.15 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the PLE “Institute of Chemistry” of the Ministry of Science and Education of the Republic of Azerbaijan

Chairman of the
Dissertation council
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Corresponding member of ANAS,
Doctor of Chemical Sciences, Professor
Mahammad Baba Babanly

Scientific secretary of the
Dissertation council
____
Doctor of Philosophy in
Chemistry, Associate Professor
Ulviyya Ahmed Mammadova

Chairman of the
scientific seminar
____
Doctor of Chemical Sciences,
Associate Professor
Kazim Gafar Guliyev

GENERAL CHARACTERISTICS OF THE WORK

Relevance of the topic and degree of development. Diene polymers occupy an important place among industrial polymers. Their importance for both researchers and engineers is associated with two factors: 1) they have good physical and physicomechanical properties; 2) the presence of olefin groups in macromolecules allows for various reactions of chemical modification of these polymers. Such reactions are mainly applied to the synthesis of polymers that cannot be obtained by standard polymerization and polycondensation methods but have the required physical and chemical properties. The specific chemical modification reaction is selected depending on the properties of the new polymers obtained and, accordingly, the area of application. For example, the role of new products synthesized by functionalization of diene polymers in hydrometallurgy, nanotechnology, the medical and electronics industries, and solving environmental problems is undeniable.[1,2,3.]

Polymers with phosphorus-containing groups occupy a special place among functional polymers. One interesting method for introducing phosphorus-containing groups into a polymer macromolecule is the oxidative chlorophosphorylation reaction. The presence of these functional groups in the modification products enables numerous chemical transformations and the synthesis of new polymers using them. These polymers, in turn, possess complex mechanical and physicochemical properties. Thus, the products obtained because of the modification of industrial polymers, including diene polymers, by

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[1] Chakraborti, S. Elastomers for Soft Electronics: A Review from the Material's Perspective / Chakraborti, S., Banerjee, P.S., Basu, D. [et al.] // Advanced Engineering Materials. 2025, 27, 9, https://doi.org/10.1002/adem.202402458

[2] Ansari, A. Design of Silyl Functionalized Emulsion SBR and its Application in Green Tire / Ansari, A., Mohanty, T.R., Ramakrishnan, S. [et al.] // *Industrial & Engineering Chemistry Research* 2025 *64* (7), 3795-3809, DOI: 10.1021/acs.iecr.4c04125

[3] Ansari, A. Epoxy modified styrene butadiene rubber (SBR) in green tire application / Mohanty, T.R., Sarkar, Sh. S. [et al.] // European Polymer Journal, Volume 213, 2024, 113069, https://doi.org/10.1016/j.eurpolymj.2024.113069.

the oxidative chlorophosphorylation reaction, can be used in various fields of engineering and technology due to their properties.

The object and subject of the research. The object of the research is a phosphorus-containing polymer (PhCP) synthesized based on styrene-butadiene rubber (SBR) with brand No: DSSK-2560/M27. The subject of the study is the investigation of the synthesis of PhCP by oxidative chlorophosphorylation reaction. Along with this, various investigation methods were used to confirm the structure and properties of the synthesized polymer. The sorption capacities of the synthesized polymer in relation to heavy metal ions and yeast fungi were also discussed in detail.

Aims and objectives of the research. The aim of this work is to synthesize a new polymer with certain properties by oxidative chlorophosphorization of industrial rubber in laboratory conditions and study the physicochemical characteristics of the sorption of zinc, chromium and iron ions and microorganisms in them. Thus, the specific tasks set in the work are as follows:

- Modification of industrial BSR by oxidative chlorophosphorylation and further hydrolysis of the intermediate product.
- Determination of the composition and structure of the synthesized polymer using modern analytical methods.
- Investigation of the sorption properties of the obtained polymer in relation to Zn (II), Fe (III), and Cr (III) ions and determination of optimal sorption conditions.
- Study of the kinetics and thermodynamics of the sorption process of Zn (II), Fe (III), and Cr (III) ions.
- Comparison of the sorption properties of the synthesized polymer and the industrial sorbent KU-1 in relation to zinc ions and study of the kinetics of the sorption process.
- Study of the effect of contact time, temperature, stirring speed, mass of the synthesized polymer, number of yeast cells, and pH of the medium on the sorption of yeast and the determination of optimal sorption conditions.

Research methods. Modern physicochemical analysis methods were used in this work to ensure a comprehensive approach to achieving the set goals. The PhCP was synthesized using oxidative chlorophosphorylation. To determine the structure and physicochemical properties of the synthesized PhCP, as well as qualitative and quantitative analysis, IR, UV, and electron spectroscopy, an ICP MS 7700e mass spectrometer, thermogravimetric (TG), and X-ray diffraction (XRD) analysis were used in the research work.

Reliability of results. The reliability of the results obtained was confirmed using modern methods of physicochemical analysis.

The main provisions for the defense:

1. Synthesis of a new PhCP based on industrial SBR;
2. Study of the structural and sorption characteristics of the synthesized polymer;
3. Data on the optimal conditions (pH, temperature, sorption time, metal ion concentration, sorbent weight) for the sorption of Zn^{2+}, Fe^{3+}, and Cr^{3+} ions from their individual solutions by PhCP synthesized based on SBR;
4. Kinetic characteristics of the sorption process of heavy metal ions (Zn^{2+}, Fe^{3+}, and Cr^{3+}) by PhCP synthesized based on SBR;
5. Description of isotherms and the analysis of possible mechanisms of sorption of Zn^{2+}, Fe^{3+}, and Cr^{3+} ions by PhCP synthesized based on SBR using known theoretical models;
6. Thermodynamics of the sorption process of heavy metal ions (Zn^{2+}, Fe^{3+}, and Cr^{3+}) by PhCP synthesized based on the SBR;
7. Comparative sorption of Zn^{2+} ions by KU-1 universal cationite and PhCP synthesized based on the SBR;
8. Data on simultaneous sorption of Zn^{2+}, Fe^{3+}, and Cr^{3+} ions from the mixed solution;
9. Data on the optimal conditions of the sorption of yeast fungus by the PhCP synthesized based on the SBR.

Scientific novelty. For the first time:

- a new PhCP was obtained by oxidative chlorophosphorylation on the base of SBR with brand No. DSSK-2560/M27;
- the structure and structural-morphological characteristics of the synthesized PhCP were determined by using a complex method of FTIR, UV-Vis, EDS-spectroscopy, SEM, XRD, TGA and DSC;
- the sorption of heavy metal ions (Zn^{2+}, Fe^{3+}, and Cr^{3+}) by the synthesized PhCP were carried out by systematic studies;

- comparative sorption of Zn^{2+} ions was carried out using synthesized PhCP and industrial cation exchanger KU-1;
- sorption of microorganisms was carried out using synthesized PhCP on the example of yeast fungus, and the influence of temperature, amount of sorbent, pH of the medium, and number of microorganism cells on the sorption processes was studied.

Theoretical and practical significance of the research: The regularities identified in the research work can be applied to the chemical modification of various copolymers, as well as polymer wastes obtained from them. The approaches taken to study the synthesized new polymer can be used to determine the structural properties of various types of polymers and composites. The practical significance of our study is to obtain information about the PhCP synthesized for the first time based on SBR and to study the sorption of heavy metal ions and microorganisms. The obtained PhCP can be recommended for wastewater treatment processes. The range of such sorbents will expand in the future to solve environmental problems.

Approbation and implementation: The most important results of the dissertation were reported at international and Republican conferences: Materials of the XI Republican Scientific Conference "Actual Problems of Chemistry" dedicated to the 94th anniversary of the birth of national leader Heydar Aliyev (Azerbaijan, Baku, 2017); III International Scientific Conference on “Ecology: Problems of Nature and Society” dedicated to the 110th anniversary of Academician Hasan Aliyev (Azerbaijan, Baku, 2017); “Coordination Compound Chemistry: Current Problems of Analytical Chemistry” dedicated to the 85th anniversary of Academician Rafiga Alirza Aliyeva (Azerbaijan, Baku, 2017); International Scientific and Technical Conference “Petrochemical Synthesis and Catalysis in Complex Condensed Systems”, dedicated to the 100th anniversary of Academician B.K. Zeynalov (Azerbaijan, Baku, 2017); XII International Scientific Conference of PhD students, masters and young researchers “Current Problems of Chemistry” dedicated to the 95th anniversary of the birth of National Leader Heydar Aliyev (Azerbaijan, Baku, 2018); International Scientific Conference of Master’s students and young researchers “Problems of Physics and Astronomy” (Azerbaijan, Baku, 2018); Abstracts of reports XXXIV Russian youth scientific conference with international participation dedicated to the 190th anniversary of the birth of D. I. Mendeleev "Problems of theoretical and experimental chemistry" (Russian Federation, Yekaterinburg, 2024).

Based on the results of the dissertation, the author has 14 scientific publications, including 7 original articles (3 of which are single-authored) in journals listed by the Higher Attestation Commission, and 7 abstracts of reports at international and Republican conferences.

Name of the organization where the dissertation has been performed: The dissertation work was completed at Baku State University, Department of Chemistry, Macromolecular Compounds subdepartment.

Structure and scope of the dissertation. The total length of the dissertation is 165 pages. The dissertation consists of an introduction (9488 characters), four chapters (151126 characters), conclusions (2236 characters), a list of 160 references, 27 tables, and 30 figures, for a total of 162850 symbols.

Personal contribution of the author. Analysis of literature data and justification of the tasks set, selection of research methods and their application in solving problems, systematization of experimental results were carried out directly by the author herself in consultation with scientific supervisors. The analysis of the experimental and spectral data obtained, the discussion of the results in scientific conferences, and the publication of scientific articles were carried out by the author herself with the assistance of various specialists.

The introduction substantiates the relevance and importance of the dissertation work, the goals and objectives of the research, the scientific novelty, practical significance and application of the obtained results.

The first chapter provides an overview of the available literature on polymer modification, the synthesis of polymer sorbents, the sorption of heavy metal ions, and the adsorption of microorganisms.

The second chapter describes the starting materials, equipment, methods, and reaction parameters used in the synthesis of PhCP, sorption of metal ions and yeast. The principle of operation of the instrumental methods used, the brands of instruments, the level of sensitivity, and the software are described in detail. The influence of various factors on the sorption capacity of PhCP was also studied.

Synthesis method of PhCP. Synthesis of PhCP was carried out by oxidative chlorophosphorylation of SBR with PCl_3 in the presence of oxygen. After the reaction is complete, the liquid phase (by-product) from the reaction medium is removed with a water jet pump. As a result, the solid phase remains in the reaction flask, which is subsequently subjected to hydrolysis.

Sorption of metal ions by PhCP. 10^{-1} M aqueous solutions of $FeCl_3{\cdot}6H_2O$, $ZnSO_4{\cdot}7H_2O$, and $Cr(NO_3)_3$ salts were prepared and diluted for usin in all stages of the subsequent study. The concentrations of Zn^{2+}, Fe^{3+}, and Cr^{3+} ions in the solution were measured using an ICP MS 7700e mass spectrometer and a photocolorimetric method. Experiments were conducted to study the sorption capacity of PhCP under the influence of various factors: sorbent amount, initial ion concentration, pH of the medium, sorption time, and temperature.

Sorption of yeast fungi by PhCP. For the experiment, yeast biomass was taken, and the number of yeast cells determined in advance. The amount of yeast in the solution was determined using a photoelectric colorimeter (photoelectric colorimeter type KF77 ZALIMP) at a wavelength of λ=290 nm.

Equipment and research methods used in the study. ICP MS 7700e mass spectrometer, KF77 ZALIMP photoelectric colorimeter; Perkin Elmer STA 6000 analyzer; Varian 640 – IR IR spectrometer; UV-vis (ultraviolet spectrometer); Rigaku Mini Flex 600 diffractometer; *Candida guillermondii* yeast fungi from BDU 217; JEOLJSM-6610LV scanning electron microscope were used in research work.

The third chapter is devoted to the study of PhCP, which was obtained by oxidative chlorophosphorylation reaction with subsequent hydrolysis of the intermediate modifier. The following methods were used in the study: IR, UV-Vis, EDS spectroscopy, SEM, XRD, TGA and DSC.

Study of the structure of PhCP by IR spectroscopy.

To interpret the results obtained by IR spectroscopy, the spectra of carbon tetrachloride, SBR and PhCP were considered (Figures 1-3).

When analyzing the IR spectrum of carbon tetrachloride, absorption bands are visible in the 795-760 cm^{-1} range, which corresponds to chlorine atoms bonded to carbon (Figure 1). The IR spectrum of SBR (Figure 2) shows absorption bands in the range of 3030 cm^{-1} and 1600-1500 cm^{-1}, which indicates the presence of an aromatic group, i.e., a benzene ring. The nature of the substitution being determined by strong absorption below 900 cm^{-1}. The absorption band in the 1490-1460 cm^{-1} region indicates the presence of a double bond in the polymer macromolecule.

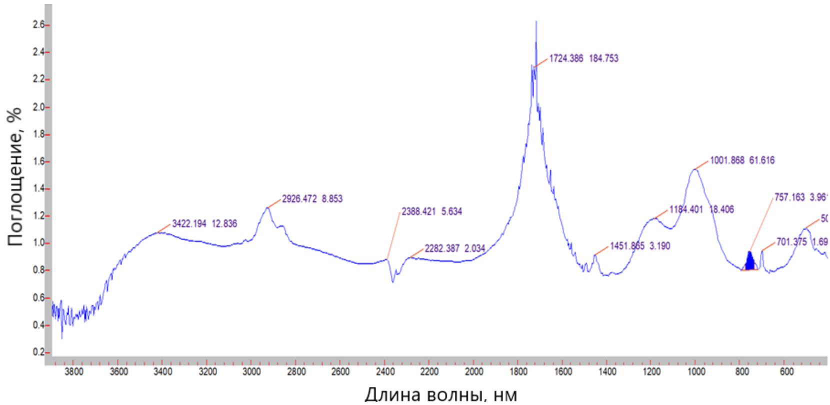

Figure 1. IR spectrum of carbon tetrachloride – CCl_4 [1, 11]

Figure 2. IR spectrum of SBR [1, 11]

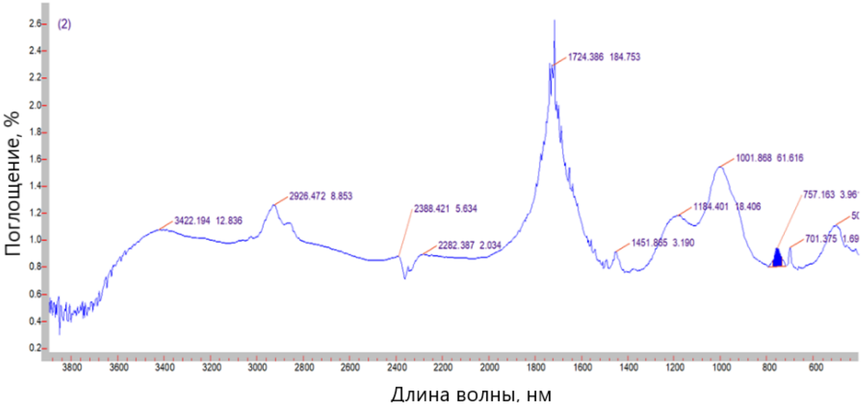

Figure 3. IR spectrum of PhCP [1, 11]

Figure 3 shows that the absorption bands in the 750-700 cm^{-1} range correspond to C–Cl bonds. During modification, chlorine radicals attach to the carbon atom according to the corresponding reaction.

In the IR spectrum of the modification, the absorption bands at 1050-1030 cm^{-1} indicate the presence of a phosphorus atom bonded to the alkyl group of the polymer via oxygen. In polymers, a bound of OH^{-} group gives a signal in the 3400-3200 cm^{-1} absorption region. It has been established that there is only one broad band for solids. The absorption band at 1720 cm^{-1} indicates the presence of a -CO-O- group attached to the aromatic part of the polymer, to the parts of the polymer containing double bonds. According to the literature, the valence vibrations of the OH^{-} group do not depend on the nature of the atom bonded to oxygen (carbon or phosphorus). However, in phosphorus compounds containing the P-OH group and having acidic properties, the hydrogen bond is more effective than in carboxylic acids. In PO(OH)$_2$ - groups, the valence vibrations of the OH^{-}- group at a wavelength of 2300 cm^{-1} can be attributed to hydrogen bonds. Asymmetric and symmetric vibrations of the POO- group correspond to lower wavelengths (1000 and 1100 cm^{-1}) [1, 11].

Study of the structure of PhCPs using UV spectroscopy. Figures 4 shows the UV spectrum of PhCP.

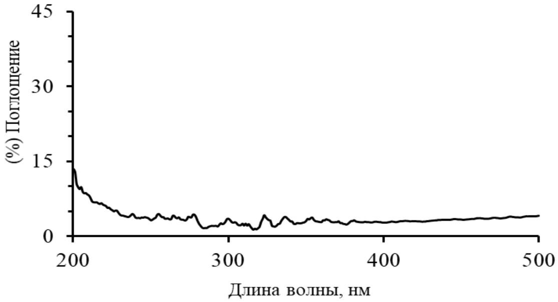

Figure 4. UV spectra of PhCP [10, 12]

Comparison of this spectrum with literature data indicates that the initial rubber undergoes modification and the hydrolysis product contains phosphorus-containing acid groups.[4]

Thermogravimetric analysis of PhCP. In this study, the thermogravimetric analysis of PhCP was conducted in an argon atmosphere at a rate of 5 K/min, heating from 25 to 1000 °C. For comparison and interpretation of the data obtained, the initial rubber was also analyzed under similar conditions. The results in the form of TG/DTG and DSC are shown in Figure 5.

As can be seen in Figure 5a, the TG and DTG curves do not change until a temperature of about 300 °C. However, a tendency toward an exothermic effect is noticeable on the DSC curve from the the beginning of the process. It should be noted that several processes occur at this time. First, as the temperature rises, the macromolecules of the original rubber become more mobile. The reason for this is the melting process. In fact, the melting of polymers is an endothermic process. However, as the macromolecules move in the original rubber, the density of the polymer packing increases. This is an exothermic process. Since the latter process is more

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[4] Ellingford, C. Structure and Dielectric Properties of Electroactive Tetraaniline Grafted Non-Polar Elastomers / Ellingford, C., Pengchaicharoen, A., Wemyss. *J. Compos. Sci.* - 2020, *4*, -p. 25. https://doi.org/10.3390/jcs4010025

dominant, the total Δ*H* is less than zero. After a temperature of about 300 °C, the rubber begins to lose mass. The process continues up to a temperature of ~ 520 °C. If we consider all three curves, the processes occurring in the temperature range 350-520 °C can be divided into two parts.

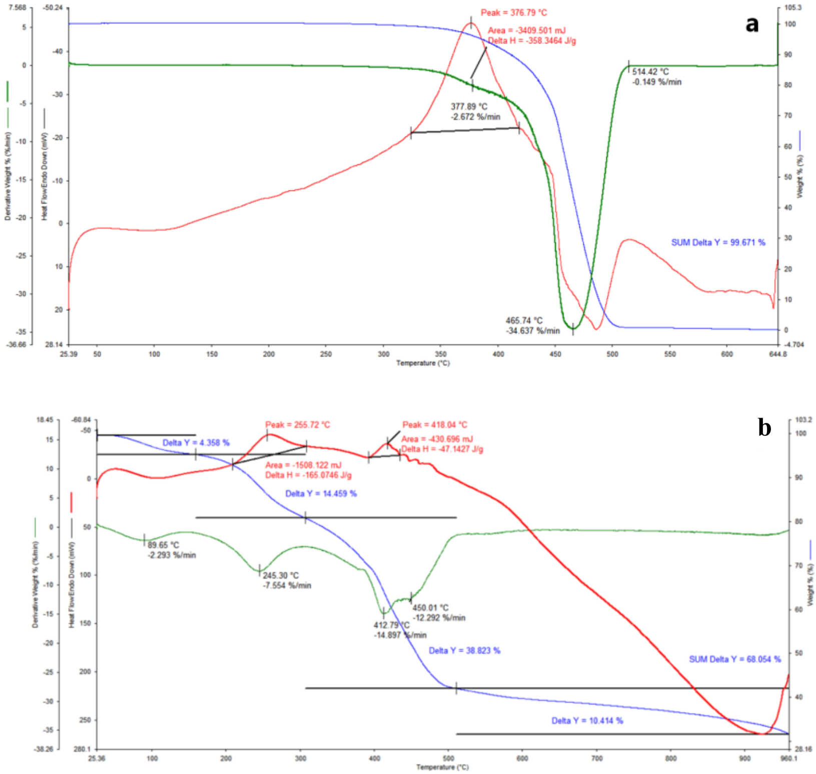


Figure 5. TG/DTG and DSC curves of the original rubber (a) and its modified form (b)

The first interval is 350–430 °C. An intense exothermic process occurs in this interval. According to the DSC curve, the maximum temperature corresponds to ~377 °C and the Δ*H* is -358 J/g. According to the TG curve, there is ~10% mass loss, with the maximum mass loss occurring at ~378 °C. Thus, the processes occurring in the first interval can be explained as follows. Since the temperature is relatively high, some compounds are removed from the sample. This creates voids between the macromolecules. As a result, the packing density of the macromolecules increases even more.

The second interval is 430-530°C. In this case, the original rubber is completely decomposed. As can be seen from the DSC curve, an intense endothermic process occurs. The total mass loss is ~99.7%. As a result, the residue is very small, only 0.4%.

As can be seen from the figure, the thermal destruction of PhCP is quite complex compared to the thermal destruction of the initial rubber. First of all, it should be noted that according to the TG/TGA curve, the resulting polymer decomposes in three stages. The first stage begins at a temperature of 25°C and continues up to a temperature of about 150°C. The mass loss is ~4.4%.

It should also be noted that an exothermic peak is observed on the DSC curve at the first stage. This peak is relatively noticeable up to 100 °C. In general, this trend continues up to 150 °C. It should be taken into account that due to the evaporation of water from the sample, voids are created between the macromolecules in the polymer matrix and the packing density increases due to the mobility of the macromolecules. Thus, in the first interval (25-150 °C on the DTA curve), two processes occur in parallel. The first, i.e., water evaporation, is an endothermic process, and the second, an increase in the packing density of macromolecules, is an exothermic process. Overall, the process is exothermic.

The second stage of the DTA curve begins at 150–170 °C and continues to approximately 320 °C. As can be seen from the TG curve, the mass loss at this stage is 14.5%. In general, the second stage of decomposition is characterized by relatively higher mass loss values than in the first stage. At this stage, mass loss is the result of the transformation/decomposition of functional groups. The process begins with the dehydration reaction of phosphonate and phosphate groups in polymers, and the DTA curves have a slightly more complex shape. As can be seen from the figure, the peak on the DTG curve is recorded at a temperature of about 245 °C. Near this maximum, there is a peak at 255.72 °C on the DSC curve in the temperature range of 210-310 °C. The change in enthalpy characterizing the exothermic process in the specified interval is -165 J/g. This process indicates that the dehydration of phosphonate and phosphate groups is accompanied by other processes: the formation of polyphosphate fragments with the formation of new bonds after dehydration; a change in the packing density of macromolecules.

The third stage of thermal degradation of the synthesized polymer begins at a temperature of 320 °C. As can be seen from the figure, this stage can be divided into two parts. The first part lasts up to a temperature of approximately 510 °C. According to the TG/TGA curve, the mass initially decreases slowly during the process, but after 380 °C, the mass decreases rapidly (up to 430 °C), which is visible as an intense peak (413 °C) on the TGA curve. In the temperature range of 380-430 °C, an intense exothermic effect is recorded on the DSC curve (the peak corresponds to a temperature of 418 °C). The enthalpy change is -47 J/g. A new peak also appears on the DTG curve at a temperature of 450 °C. Overall, the mass loss in the temperature range 320-510 °C is 38%. At a temperature of 510 °C, the second stage of decomposition begins, which continues until the end. According to the DSC and TG/DTA curves, this stage is characterized by equal destruction. The mass loss is about 10%, and due to the thermal effect, the process is endothermic. Thus, the third stage is quite complex. A series of sequential-parallel reactions occur, leading to the complete destruction of the polymer matrix: in the second stage, fragments formed as a result of the transformation of functional groups separate from the total mass, the main chain is destroyed, etc.

According to the results obtained, the total mass loss during thermal destruction of PhCP in an argon environment is approximately 68.0%. It can be seen that the thermal degradation of PhCP, formed as a result of the oxidative chlorophosphorylation reaction and hydrolysis of the intermediate product, differs significantly from the thermal degradation of the initial rubber.

XRD analysis of PhCP. Figure 6 shows the PhCP diffractogram.

As can be seen from the figure, the polymer diffractogram corresponds to the amorphous phase. It is known that rubbers, like a number of industrial polymers, have an amorphous structure. The SBR used in our study also has an amorphous structure. However, during the modification process, the functional groups introduced into the polymer matrix can form a new phase. The results obtained show that the synthesized PhCP, has an amorphous structure like the original rubber. No additional peaks are observed on the diffractogram, which indicates that the system has a single phase. That is, the functional groups in the polymer are not found as a separate phase, but are evenly distributed throughout the matrix and together with the polymer represent a single phase, while the polymer retains the general amorphous structure characteristic of industrial rubbers.

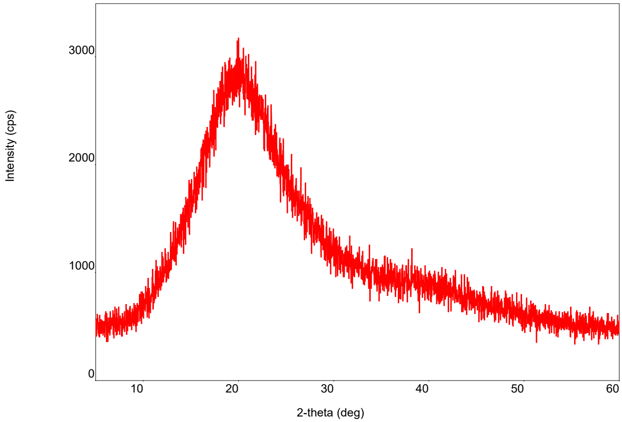


Figure 6. Diffractograms of PhCP

Investigation of PhCP using SEM with EDX. The PhCP was studied by electron spectroscopy. SEM images were magnified 1900 times and pore sizes were determined. SEM images of the sorbent are shown in Figure 7. As can be seen in Figures 7a and 7b, the synthesized PhCP has a porous structure (based on the scale of the image, it can be said that the pores present are meso-, macro-, and micro-sized), which cannot be said about the original rubber. At the same time, the percentage content of the elements in the new polymer has been determined (the corresponding spectrum is shown in

Figure 7c and Table 1.

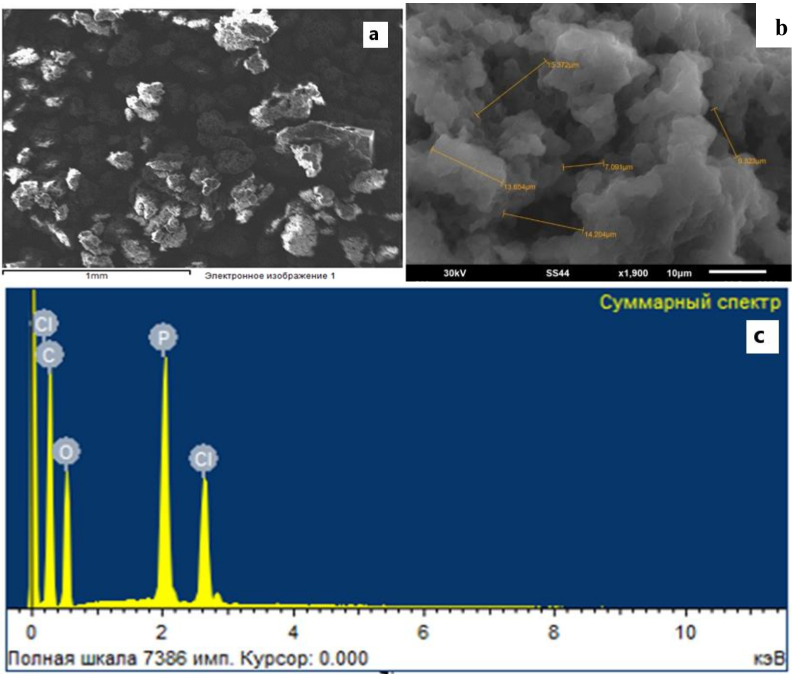


Figure 7. SEM images and energy dispersive X-ray spectra of PhCP

Table 1

Percentage content of elements in the composition of the new polymer

Element	Weight, %	Atomic, %
C	59.68	69.96
O	28.17	24.79
P	7.40	3.37
Cl	4.75	1.89

As can be seen, the PhCP sample contains phosphorus, chlorine, and oxygen atoms in addition to carbon atoms. Considering the elemental composition of the synthesized polymer, it can be concluded that 2 out of 10 elementary units are modified with phosphorus-containing groups. In addition, one elementary unit contains a chlorine atom. Since the amount of oxygen is more than equimolar to phosphorus, it can be assumed that the macromolecules are linked together by oxygen, i.e., they have C-O-C bonds.

Furthermore, given the relatively large number of butadiene elementary units in the SBR macromolecule, as well as the shielding of styrene elementary units as a result of conformational changes, modification by oxidative chlorophosphorylation proceeds mainly in three directions:

- oxidation;
- phosphochlorination (the intermediate modifier contains fragments with phosphonyl and phosphoryl dichloride groups; as a result of hydrolysis of the intermediate product, the phosphonyl and phosphoryl dichloride groups are converted into phosphonic and phosphate groups; the presence of chlorine atoms in PhCP indicates that the reaction proceeds in the direction of double bond formation; the release of hydrogen chloride from the reaction flask during the phosphochlorination reaction indicates that the reaction also proceeds in the direction of substitution;
- cross-linking (PhCP is insoluble in the corresponding organic solvents, which indicates that it is a polymer with a three-dimensional structure).

The fourth chapter is devoted to the study of the sorption of Zn(II), Fe (III), Cr (III) ions and yeast on the synthesized FSP. It also includes a comparative study of the sorption properties of FSP for zinc ions with the industrial sorbent cation exchange resin KU-1, as well as kinetic and thermodynamic studies.

Study of the sorption properties of PhCP. In this chapter, the sorption capacity of the obtained polymer for Zn (II), Fe (III) and Cr(III) ions was studied. In order to clarify the sorption mechanism of metal ions by FSP and to determine the changes occurring in the polymer matrix during sorption, polymer samples containing metal ions were studied using UV, TG/DTG, DSC, X-ray diffractometry and SEM.

Study of metal ion sorption. The initial concentration of metal ions affects their distribution between the solid (in our case, PhCP) and liquid phases (metal ion solution). As the initial concentration of metal ions in the solution increases, the active centers of the polymer become filled. Table 2 shows the effect of the initial concentration of Zn (II), Fe (II), and Cr (III) ions in the solution on the sorption capacity (sorption capacity and degree of sorption) of PhCP.

Table 2.

Effect of initial concentration of metal ions on the sorption process [3, 9, 11]

Zn (II)	C_0, M	0,34 $\times10^{-3}$	0,58 $\times10^{-3}$	0,958 $\times10^{-3}$	5,03 $\times10^{-3}$	6,36 $\times10^{-3}$	14,03 $\times10^{-3}$
	CE, mg/g	6,63	11,31	15,76	15,8	15,9	16
	R, %	100	100	84,34	68,2	54,2	2,3
Fe (III)	C_0, M	0,9$\times$ 10^{-3}	1,1 $\times10^{-3}$	1,5 $\times10^{-3}$	1,7 $\times10^{-3}$	2,2 $\times10^{-3}$	2,6 $\times10^{-3}$
	CE, mg/g	1,2	2,1	7,2	6,8	4,7	3,8
	R, %	22,2	36,4	90,7	76,5	41	26,9
Cr (III)	C_0, mg/l	402,6	2459,4	5094,3	7159,6	9856,3	11197,5
	CE, mg/g	12	171	935	948	980	990
	R, %	9.6	23.2	61.2	44.1	33.1	29,5

As can be seen from the table, the sorption capacity increases with increase in the initial concentration of metal ions in the solution. The probability of their collision with the sorption centers of the polymer increases as the concentration of metal ions in the solution increases. Along with this, the driving force of mass transfer is better, which contributes to a decrease in resistance to mass transfer and an increase in the adsorption capacity of PhCP.

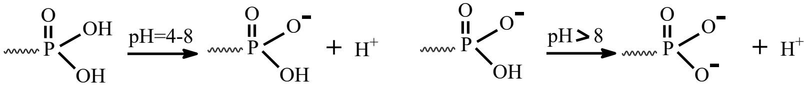
In addition, the driving force for mass transfer increases, which reduces the mass transfer resistance and increases the adsorption capacity of the PhCP.

The effect of the PhCP mass on the sorption of metal ions was studied in this work. According to the results of the study, an increase in the polymer mass increases the sorption rate, while the sorption capacity decreases. Initially, this phenomenon can be explained as follows: the increased polymer mass increases the surface area of the solid phase in the "solid phase - metal ion solution" system. This leads to an increase in the number of active sorption sites involved in the sorption process.

However, the decrease in sorption capacity with an increase in the polymer mass can be explained as follows: although the adsorbed surface area increases with an increase in the polymer mass, the adsorbed amount of metal ions does not increase proportionally (linearly) with an increase in the polymer mass, so the ratio between the adsorbate mass (mg) and the polymer mass (sorption capacity) decreases. When the polymer and metal ions in the solution come into contact, adsorption occurs rapidly and in large quantities.

The effect of pH on the sorption capacity of PhCP for Zn(II), Fe(III) and Cr(III) ions was studied. It was found that increasing the pH of the solution from 1 to 6 led to an increase in the R% sorption efficiency from 27% to 82.2%, while increasing the pH of the solution from 6 to 11 led to a decrease in the R% sorption efficiency from 82.2% to 30% (for Zn ions). The highest removal efficiency was achieved in weakly acidic and neutral solutions (pH 6-7). This is due to the protonation of the functional groups of the polymer at low pH values ($pH < 3$). The data show that in a more acidic environment, when the pH of the solution is in the range of 1-3, a relatively low adsorption efficiency is observed. This can be explained by the fact that Zn ions are in the form of aqua complexes in this pH range: $[Zn(H_2O)n]^{2+}$. Adsorption occurs primarily as a result of coordination of ions with $-P = O$ groups. However, these groups also act as active centers for protons due to the formation of hydrogen bonds $- P = O------H^+$, which displace metal ions. On the other hand, PhCP exhibits a low swelling rate in this pH range and the number of active sites available for the adsorption of Zn ions is small. As the acidity of the solution decreases in the pH range of 4-6, the adsorption rate of metal ions gradually increases and reaches a maximum at pH 6.

Then, as the pH increases towards a more alkaline environment, the adsorption rate decreases continuously. As the pH of the environment increases, PhCP gradually ionizes. Specifically, the polymer acquires the following structures: ~P(O)(OH)(O-) (in the pH range of 4-8) and ~P(O)(O-)(O-) (at pH>8).



In this case, sorption occurs due to ionic coordination and/or ionic interaction, partial transformation of existing aqua complexes ($[Zn(OH)(H_2O)_{n-1}]^+$) into hydro complexes. In this regard, at pH>3, it is possible to form both ammonium and acetate complexes of various compositions, structures, and stability. Thus, metal ions in solution at pH >3 will be present in the form of aqua, hydroxo, and ammonium complexes. In particular, at a pH of 6, which corresponds to the maximum sorption capacity, zinc ions are predominantly present in the form of an ammonium complex of the following type: $[Zn(NH_3)_4]^{2+}$. When the pH increases, their amount in the solution decreases. Such types of complexes are easily adsorbed by a fully ionized polymer (~P(O)(O-)(O-)). The final increase in ammonia concentration between pH 10 and 11 resulted in the formation of a relatively unstable $[Zn(NH_3)_6]^{2+}$ complex. As a result, an initial pH of 6.0 was chosen for subsequent sorption experiments.

For Fe(III) ions, increasing the pH of the solution from 1 to 6 led to an increase in the R% from 23% to 90.7%, and a further increase in the pH of the solution from 6 to 11 led to a decrease in R% from 90.7% to 19%. The highest removal efficiency values were obtained in slightly acidic solutions (pH 6). For Cr (III) ions, an increase in the pH of the solution from 1 to 6 led to an increase in the R% value from 18.4% to 61.2%, and a further increase in the pH of the solution from 6 to 11 led to a decrease in R% from 61.2 to 30%. The highest removal efficiency values were obtained in slightly acidic solutions (pH 6).

One of the important parameters for optimizing sorption processes and their application in industry is the contact time between the liquid (metal ion solution) and solid phases (PhCP), i.e., the time required to establish equilibrium in the system. To this result, the dependence of sorption parameters on time was studied. The results of the experiments showed that the sorption of Zn (II) and Cr (III) ions occurs rapidly in the first 5 minutes and slows down near equilibrium. For the sorption of Fe (III) ions, this parameter is 10 minutes.

One of the main factors affecting sorption processes is temperature. To study the influence of this factor, sorption of metal ions by PhCP was carried out at different temperatures (25, 35, and 50 °C). It was found that an increase in temperature only reduces the sorption time of metal ions. This is due to the fact that the initial concentration of metal ions is not high. According to the results obtained, when sorption of zinc ions is carried out at a temperature of 25 °C, equilibrium in the system is reached within 25 minutes, and the sorption efficiency is 100%. This sorption efficiency equilibrium is achieved in 10 minutes at 35 °C and in 7 minutes at 50 °C. A similar trend was also observed for Fe (III) and Cr (III) ions. Thus, for Fe(III) ions, equilibrium is reached within 70 (25 °C), 65 (35 °C), and 55 minutes (50 °C). When the temperature increases during the sorption of Cr (III) ions, equilibrium is reached in 35 (25 °C), 30 (35°C), and 25 minutes (50 °C).

To determine the effect of temperature, samples (i.e., solutions of metal ions with higher concentrations) were taken in advance and the effect of temperature on the sorption properties of the polymer was established as follows:

- the degree of sorption and the sorption capacity of the polymer increase for all three metal ions;
- equilibrium in the "polymer-metal ion solution" system is established in a shorter period of time (if the concentration of metal ion solutions is not high).

This result can be explained as follows:

- the mobility of metal ions increases with increasing temperature;
- with increasing temperature, conformational changes in the macromolecule occur faster, which leads to the involvement of a larger number of active centers in the sorption process;

- sorption is chemical in nature.

Determination of kinetic parameters of the sorption of Zn(II), Fe(III) and Cr(III) ions

Our research has identified the limiting stage in the study of sorption processes. The dependence of the sorption process on time was used as experimental results.

The contribution of external diffusion to the ion sorption process on the polymer can be described by equation (1):

$$-ln(1-F) = -\gamma \cdot t, \qquad (1)$$

where F – s the equilibrium attainment rate, calculated using the formula $F = q_t / q_e$ (q_t – is the sorption value (mg/g at time t, q_e – is the sorption value at equilibrium (mg/g));

γ – is a constant value for the current conditions;

t – is the sorption time (min)

A graph of the $-ln(1-F)$ versus $f(t)$ was constructed. From the data obtained (for Zn (II) ions: $ln(1-F)=-0.3881 \cdot t$; correlation coefficient – 0.8866; for Fe (III) ions: $ln(1-F)=-0.0312 \cdot t$; correlation coefficient – 0.9136; for Cr (III) ions: $ln(1-F)=-0.1256 \cdot t$; correlation coefficient – 0.8265), the experimental kinetic data does not obey the external diffusion model (since the correlation coefficients are not high). In general, only in the initial stages is a linear dependence of the function $-\ln(1- F) = f(t)$, and the kinetic curves do not describe only the external diffusion mechanism throughout the entire process. The process proceeds with a decrease in the influence of the external diffusion factor and an increase in the influence of the internal diffusion factor. Thus, this process proceeds in a mixed diffusion mode, namely, with diffusion in the solution film and diffusion in the functional polymer grain.

To estimate the contribution of internal diffusion to the sorption process, the empirical equation (2) is used:

$$q_t = k_d \cdot t^{1/2}, \qquad (2)$$

where q_t – is the amount of sorbed ion per unit mass of polymer in time t, mg/g;

k_d – is the internal diffusion rate constant, $mg \cdot g^{-1} \cdot min^{-0,5}$;

t – is the sorption time, min.

If internal diffusion is the limiting stage in the sorption process, then the experimental data should obey a linear relationship in the q_t – $t^{1/2}$ coordinates. Based on the data obtained, parameters for internal diffusion were found (for Zn (II) ions: $q_t = 0.1993 \cdot t^{1/2} + 4.7925$; correlation coefficient – 0.7118; for Fe (III) ions: $q_t = 0.8152 \cdot t^{1/2} + 0.3879$; correlation coefficient – 0.9520; for Cr (III) ions: $q_t = 136.59 \cdot t^{1/2} + 731.41$; correlation coefficient – 0.9880).

In most cases, this dependence is multilinear and characterized by 2-3 sections, and is described by the equation (3):

$$q_t = k_d \cdot t^{1/2} + A, \qquad (3)$$

where A is the segment cut off on the dependence $q_t = f\,(t^{1/2})$, on the ordinate axis.

The value A in equation (3) characterizes the thickness of the boundary layer. The initial section (external diffusion kinetics) shows the diffusion of sorbate (metal ions) through the solution layer to the surface of the functional polymer. The internal diffusion process describes the second section, from the slope of which the internal diffusion rate constant is determined. In all cases, mixed kinetics occur. The external and internal diffusion rate constants are presented in Table 3.

Table 3

External and internal diffusion rate constants [8, 12]

Ion	External diffusion rate constants		Internal diffusion rate constants		
	γ	R^2	k_d, mmol·g^{-1} min$^{-0,5}$	A	R^2
Zn(II)	0,3881	0,8866	0,1993	4,7925	0,7118
Fe(III)	0,0312	0,9136	0,8152	0,3879	0,952
Cr(III)	0,1256	0,8265	136,59	731,41	0,988

R – correlation coefficient.

It is assumed that the sorption stage itself can make a significant contribution to the kinetics of the entire process. Therefore, to describe the regularity of this kinetic stage, pseudo-first-order and pseudo-second-order reaction models were used, which are expressed in linear form by equations (4) and (5):

$$ln(a_e - a_t) = lna_e - k_1 \cdot t, \qquad (4)$$

$$t/a_t = 1/(k_2 \cdot a_e^2) + (1/a_e) \cdot t, \qquad (5)$$

where a_e и a_t – are the equilibrium sorption value and the sorption value at time t, respectively, mmol/g;

k_1, k_2 – are the sorption rate constants of the pseudo-first-order (min^{-1}) and pseudo-second-order (g/mmol·min) reactions, respectively.

The values of k_1 и a_e, calculated from these dependencies are presented in Table 4.

Table 4

Rate constants of the pseudo-first and pseudo-second order models [8, 12]

Ion	Pseudo-first-order model			Pseudo-second-order model		
	a_e	k_1	R^2	a_e	k_2	R^2
Zn(II)	13,7816	0,3259	0,9308	6,0168	0,2562	0,9991
Fe(III)	6,7727	0,0278	0,9302	8,8809	0,0052	0,8918
Cr(III)	907,96	0,0999	0,9177	1666,7	0,0002	0,9933

The kinetic parameters of Zn(II), Fe(III), and Cr(III) ion sorption at temperatures of 35 °C and 50 °C were calculated using a similar method.

In addition, the studies showed that the sorption of zinc ions by industrial cation exchangers also occurs via a mixed diffusion mechanism and follows a pseudo-second-order model.

Adsorption isotherms

Adsorption isotherms determine the equilibrium relationship between the adsorbent and the adsorbate. The results obtained in this study were analyzed using the Langmuir, Freundlich, and Dubinin-Radushkevich isotherm equations. Linear forms of these models were used to calculate the corresponding parameters. It was found that for all three metal ions, the process is best described by the Langmuir model. This indicates that sorption occurs at energetically homogeneous active sites with the formation of a monomolecular layer on the surface of the functional polymer. At the same time, chemical interactions occur between metal ions and active sites.

Thermodynamic studies

Thermodynamic parameters such as enthalpy ($\varDelta H°$), entropy change ($\varDelta S°$) and Gibbs free energy change ($\varDelta G°$) were calculated to determine the thermodynamic feasibility and spontaneous nature of adsorption.

The values of $\varDelta H°$ and $\varDelta S°$ were calculated from the slope and intercept of the linear graph of $\ln K_d$ versus 1/T for the sorption of Zn(II), Fe (III), and Cr (III), respectively. The values of $\varDelta G°$ at 293, 309 and 323 K were determined using the following equation:

$$\varDelta G° = \varDelta H° - \mathrm{T}\varDelta S° \qquad (6)$$

The calculated values of $\varDelta H°$, $\varDelta S°$ and $\varDelta G$ or the sorption of Zn, Fe, and Cr are presented in Table 5.

Table 5

Values of $\varDelta H°$, $\varDelta S°$ and $\varDelta G°$ for the sorption of Zn, Fe, and Cr ions by a PhCP sorbent at different temperatures [14]

Metal ion	T (K)	$\Delta S°$ (kJ/mol·K)	$\Delta H°$ (kJ/mol)	$\Delta G°$ (kJ/mol)
Zn^{2+}	298	+0,2169	+53,447	-11,189
	308			-13,358
	323			-16,612
Fe^{3+}	298	+0,2153	+49,290	-14,869
	308			-17,022
	323			-20,252
Cr^{3+}	298	+0,1107	+31,415	-1,574
	308			-2,681
	323			-4,341

As can be seen in Table 5, the ΔG° values are negative in the temperature range from 293 to 323 K, which indicates the feasibility and spontaneity of the adsorption process at these temperatures. The positive value of ΔS° shows that the sorption process involves a dissociative mechanism and reflects the affinity of the sorbent to the sorbate.

Simultaneous sorption of Zn(II), Fe(III), and Cr(III) ions. The results of the study on the simultaneous sorption of metal ions showed the following. In the first 25 minutes, Zn (II) ions are extracted to the greatest extent, while the remaining ions are absorbed much less during this period of time. This result can be explained by the law of sorption: the ability of ions to be sorbed depends on the ionic radius—the smaller the radius, the lower the sorption capacity.

Study of functional polymers containing metal ions. The structural characteristics of functional polymers containing metal ions were studied using various research methods.

Comparing UV spectra with the spectrum of the functional polymer, a significant difference can be observed (especially in the 200-400 nm range). This difference can be explained by the formation of corresponding compounds in the polymer matrix after the sorption of metal ions.

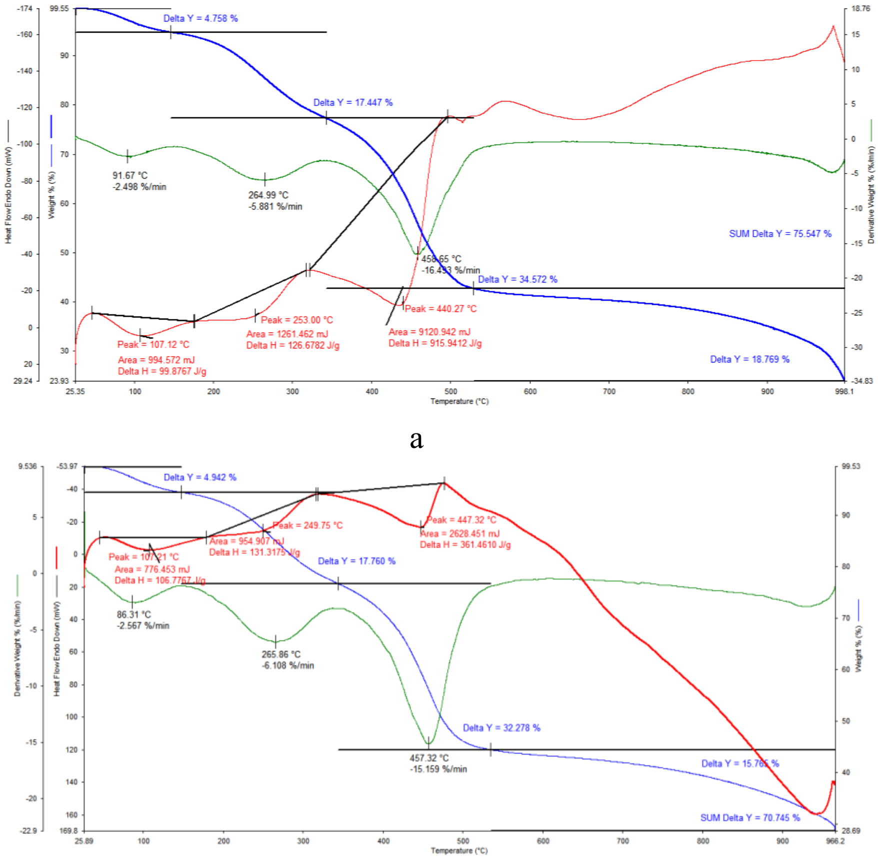
Polymers containing metal ions were also studied using thermogravimetric analysis. The results are shown in Figure 8.

According to the results obtained, the thermal destruction of samples containing metal ions has the following features:

- according to the TG and DTG curves, the destruction of samples occurs in three stages;
- depending on the nature of the metal ions, the amount of mass loss at each of the three stages differs from the amount of mass loss of the initial functional polymer at the corresponding stages. This can be explained both by the number of metal ions in the polymer matrix and by the different structure of the compounds that metal ions form in the polymer matrix;
- The DSC curve is quite complex and differs significantly from the DSC curve of the functional polymer.

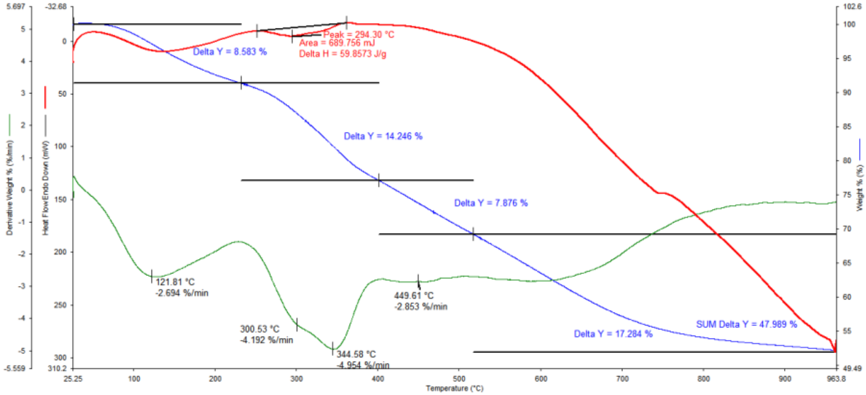
Finally, the residual amounts of polymers were compared. For other polymers, the values were moderate and similar. In general, the residual values ranged from 25 to 50%, which is typical for cross-linked functional polymers.

Metal-containing polymers were also studied using XRD. It was found that after sorption of metal ions, PhCP retains its amorphous nature. This means that the sorption of metal ions by polymers occurs throughout the volume and/or on the surface.



a

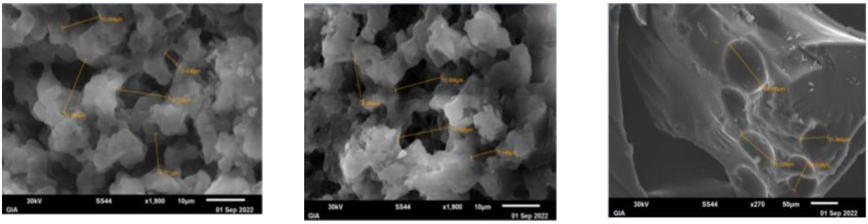
b



c

Figure 8. TG/TGA and DSC curves of PhCP with metal ions: a – with Zn (II) ions, b – with Fe (III) ions, c – with Cr (III) ions

The results of the SEM analysis of the samples are shown in Figure 9.



a b c

Figure 9. SEM images of PhCP with metal ions: a – with Zn(II) ions, b – with Fe (III) ions, c – with Cr (III) ions

A comparison of these images with the result shown in Figure 7 shows that they differ significantly from the surface of PhCP.

Sorption of microorganisms

Studies of yeast sorption have shown that relatively good results are achieved at pH values of 7–9. An increase in temperature leads to a decrease in the adsorption rate. It should also be noted that phosphonate and phosphate groups, as well as C-Cl fragments of the functional polymer, have an irritating effect on the cells of microorganisms, destroying their structure [5, 7].

CONCLUSIONS

1. For the first time, PhCP was synthesized as a result of the oxidative chlorophosphorylation reaction of DSSK-2560/M27 styrene-butadiene rubber followed by hydrolysis of the resulting intermediate modifier. The polymer was studied using XRD, SEM, TGA, UV-visible, and IR spectroscopy. The results of these studies made it possible to determine the presence of chlorine atoms and phosphorus-containing acid groups in the composition of the new polymer [1].

2. The sorption capacity of the PhCP for Zn^{2+}, Fe^{3+}, and Cr^{3+} ions was studied, as well as the influence of factors such as the initial concentration of ion solutions, the amount of polymer, the pH of the medium, the temperature, and the sorption time on the process. The results showed that, depending on the values of various factors, the degree of sorption can reach 100%. The maximum sorption capacity is observed at a pH of 6. As the temperature increases, the sorption time decreases, while the sorption capacity and degree of sorption increase [3, 4, 6, 9, 10, 11].

3. The kinetics of sorption were studied and it was found that the process is well described by a mixed diffusion and pseudo-second-order model. A sorption isotherm was constructed using two-parameter isothermal models. It was established that the sorption of metal ions corresponds to monomolecular sorption and is chemical in nature. As a result of thermodynamic studies, it was established that the sorption of metal ions is endothermic and proceeds spontaneously [8, 12, 13, 14].

4. In a comparative study of the sorption of Zn^{2+} ions by PhCP and industrial cation exchanger KU-1, the synthesized polymer proved to be more effective [2].

5. It has been found that when Zn^{2+}, Fe^{3+}, and Cr^{3+} ions are present together, Zn^{2+} ions are predominantly sorbed, which is related to the sorption pattern: the ability of ions to be sorbed depends on the ionic

radius—the smaller the radius, the lower the sorption capacity [3, 9, 10].

6. Metal-containing samples were studied using XRD, SEM, TGA, UV-visible, and IR spectroscopy. It was found that metal ions are evenly distributed throughout the polymer volume [1, 3].

7. The synthesized PhCP can also be successfully used for the sorption of microorganisms. The degree of yeast adsorption increases with increasing sorbent mass, sorption time, and stirring rate. An increase in temperature and the number of yeast cells leads to a decrease in the degree of adsorption, and the highest adsorption values are observed at pH 6-9. It is easily regenerated and suitable for reuse [5, 7].

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