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# ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

# DEVELOPMENT OF METHODS FOR DETERMİNİNG THE SORPTİON OF PALLADİUM (II) İONS İN THE PRESENCE OF SYNTHETİC SORBENTS WİTH VARİOUS FUNCTİONAL GROUPS


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## GENERAL CHARACTERISTIC OF THE WORK

**Actuality of the topic and degree of development**. Recently, in the field of analytical chemistry, sorption methods of separation and concentration have been increasingly used for the determination of microquantities of elements in natural and technical objects. These methods are widely used in the practice of analytical chemistry to provide methods of determination and analytical performance, i.e., sensitivity and selectivity. If the sample to be determined contains an extraneous component interfering with the determination, if the component to be determined is unevenly distributed in the object, if the concentration of the sample is below the limit of determination, then the use of separation and thickening methods is considered more appropriate. Taking this into account, the development of methods with high metrological properties for the determination of palladium(II) ions from objects of complex composition is one of the urgent tasks. The presence of palladium in most natural and technical objects is very insignificant, so the development of effective methods for solidification of this metal for the purpose of photometric determination and improvement of existing methods is one of the important issues. Among the methods used for this purpose, the sorption method occupies a special place due to its simplicity, expressivity, high selectivity and environmental safety.

The usage of polymeric sorbents with chelating properties is considered more important for sorption-analytical systems. One of the most urgent questions of analytical chemistry is the investigation of polymeric sorbents containing various functional analytical groups of chelating agents and their analytical application in the determination of microquantities of elements.

The literature provides almost enough information about these polymeric sorbents. It is from the literature that polymeric chelate sorbents are of greater importance because of their simplicity, speed, and high determination efficiency. The main characteristic of polymeric chelate sorbents is the presence of a functional active group in the matrix. These chemically active groups form chelate complexes and ionic associations with metal ions in solution. The process of sorption on complexing sorbents is mainly due to complexation of noble metal ions with functional groups of the sorbent. As is, chelate

sorbents are mainly used in the analysis of a number of objects, such as ores, meteorites, minerals, rocks, etc. it is also widely used in the determination of rare and noble metals.

It is that palladium(II) ion in an acidic medium has the property to form more stable complex compounds with ligands containing donor nitrogen and sulfur atoms than other metals. The chelate sorbents used for the sorption of palladium(II) ions are sorbents containing heterocyclic azo- and amino groups.It can be said that by using hydroxyazo compounds (-OH, -N=N-) it was possible to obtain results with high analytical performance in palladium(II) ion determination methods.Taking this into account, the use of reagents containing the above functional groups - the investigation and analytical application of sorption properties of pyrogallol azo derivatives with palladium(II) ion in the presence of surfactants and hydrophobic amines - is one of the topical problems. problems.

For this purpose to this end, sorption methods for the determination of palladium(II) ions were developed using a new polymeric sorbent containing a chelate group and pyrogallol-based nitro compounds.

**Object and subject of the investigation.** Polymeric sorbents based on maleic anhydride; reagents based on pyrogallol and complexes formed by these reagents with palladium(II) ion. Sorption of palladium(II) ion on synthesized new polymeric sorbents; development of methods for sorption-photometric determination of palladium(II) ion in natural and technical objects (standard alloys, electrodes, ores, nickel dust, rocks).

**The aim and objectives of the investigation.** The main objective of the thesis work is to investigation the sorption of palladium(II) ions by polymeric sorbents obtained as a result of modification of maleic anhydride and styrene copolymer by amines with different functional groups, determination of microquantities of metal ions in natural and industrial objects. (standard alloys, electrodes, ores, clays, rocks) is the development of the initial stage of thickening and separation methods.

In order to obtain the main goal during the research work, the following practical and theoretical issues were taken as a basis:

- Investigation of physicochemical properties and analytical parameters of synthesized polymeric sorbents, sorption and desorption of micro quantities of palladium(II) ion;

- investigation of the influence of different functional groups included in the synthetic sorbents on the sorption process of palladium(II) ion;

- selection of analytically more convenient sorbents for the purpose of separation of palladium(II) ion from objects of complex chemical composition by condensation method, development of new methods based on the results of scientific research.

**Research Methods**. Various physical and physicochemical methods of analysis (IR spectroscopy, UV spectroscopy, scanning electron microscopy (SEM), thermogravimetry, spectrophotometry) and sorption-condensation methods were used in the investigation of the thesis.

**Main points which are defended:**

- Determination of physicochemical parameters and analytical characteristics of synthesized polymeric sorbents by instrumental research methods;

- investigation of the influence of cationic surfactants on analytical parameters of complex compounds formed by palladium(II) ion with organic reagents based on pyrogallol, results of the process of sorption and desorption of palladium(II) ions;

- selection of complex compounds with high analytical performance obtained on the basis of physicochemical and analytical properties of binary and polyligand complexes;

- isolation and crystallization of palladium(II) ions from complex real objects, development of sorption-photometric methods for determination of isolated metal ions.

**Scientific novelty of the investigation:** For the first time, polymer sorbents with complex-processing capacity have been synthesized as a result of modification of maleic anhydride-styrene copolymer with amines with various functional groups for the purpose of sorption thickening of microquantities of palladium (II). ion. Physicochemical characteristics of the synthesized sorbents have been investigated by instrumental methods. Sorption of microquantities of palladium (II)

ions on the obtained chelate polymer sorbents has been systematically investigated and optimal sorption conditions for each metal-sorbent system and analytical parameters of the process have been determined. Sorbents with high sorption capacity have been synthesized and methods have been developed that are more convenient than other methods to determine microquantities of palladium (II) ions in natural and industrial objects.

**Theoretical and practical significance of the investigation.** The relationship between the structure and properties of organic reagents and sorbents used in the researches was established. The feasibility of using synthesized chelate polymer sorbents for separating palladium(II) ions from environmental objects by condensation was determined.

- A number of effective, promising sorption-spectrophotometric methods for determining microquantities of palladium(II) ions in electrodes, rocks, ores, standard alloys, various natural and industrial objects were developed.

- The results of the conducted scientific researches can be almost more useful for scientists working in the field of analytical chemistry.

**Approval and application.** 30 scientific papers have been published on the materials of the thesis. Of them 9 (including three with one author) are articles (5 abroad), 20 thesis are published, 1of them is a patent. The articles were published in periodical scientific editions included in international summarization and indexing systems.

The results of the dissertation work were presented at the following scientific conferences (including international scientific conferences) held in Azerbaijan and abroad. The 11th Republican Conference of doctoral students, undergraduates and young researchers "Actual Problems of Chemistry" dedicated to the 94th anniversary of national leader Heydar Aliyev (Baku, 2017), international scientific conference dedicated to the 85th anniversary of academician Rafig Alirza Giza Aliyeva "Chemistry of coordination compounds: Actual problems of analytical chemistry" (Baku 2017), XXI All-Russian Conference of young chemists (with international participation) (Nizhny Novgorod 2018), graduate students, masters and young researchers dedicated to the 95th anniversary of the birth of national leader Heydar Aliyev"

12th Republican Conference (Baku 2018), materials of the 7th Republican Scientific Conference "Actual problems of ecology and soil science in the XXI century" dedicated to the 95th anniversary of the birth of national leader Heydar Aliyev (Baku 2018), dedicated to the 110th anniversary of Academician M.Nagiyev. Materials of scientific conference "Nagiyev Readings" (Baku 2018), Baikal School-Conference on Chemistry, Collection of articles of II All-Russian School Conference dedicated to the 100th anniversary of Irkutsk State University and 85th anniversary of the Faculty of Chemistry of ISU (Irkutsk 2018), Wastes, causes of their formation and prospects of the basics, collection of scientific papers on the materials of the International Scientific Ecological Conference (Krasnodar 2019), XXIX Russian Youth Scientific Conference with international participation, dedicated to the 150th anniversary of the Periodic Table of Chemical elements 5th International Turkish Congress on Molecular Spectroscopy (2022), 6th International Congress on Commodity, Agricultural and Veterinary Sciences (Ganja, 2023).

**Name of the institution where the dissertation work was completed.** The dissertation work was completed in accordance with the scientific research conducted by the Department of Chemistry and Technology of Inorganic Substances of the Faculty of Chemical Technology of the Azerbaijan State University of Oil and Industry (State Registry No. 0111Az2003).

**The total volume of the dissertation with a sign indicating the volume of structural sections of the dissertation separately.** The dissertation is presented on 164 pages of printed text, consists of an introduction, 4 chapters, conclusion, 43 figures, 29 tables, a list of used literature in 173 titles and a list of abbreviations. The main part of the work is the number of characters 206185.

*The first chapter* (item number 81847) analyzes the literature related to the dissertation research, as well as literary data for the past 10 years, as well as important analytical characteristics of the developed methods for photometric and sorption-photometric determination of palladium (II) ions.

*The second chapter (*item number 47234*)* synthesizes organic

reagents and sorbents according to a technique, uses devices and reagents, identifies the synthesized new polymer sorbents, and discusses the results obtained, Freundlich and Langmuir isothermal models. sorption equilibrium for palladium(II) ions.

*The third chapter* (item number 19445) deals with the photometric determination of colored complex compounds formed by organic reagents with the palladium (II) ion, and the photometric investigation of binary and multi-ligand complex compounds formed by them during the spectrophotometric determination of palladium(II) ion with organic reagents.

*In the fourth chapter* (item number 45679), optimal conditions for the sorption and desorption of palladium(II) ions by chelating sorbents are determined.

**Personal contribution of the applicant to the conducted research.** The plaintiff's role in conducting the research work and experiments is direct. The plaintiff synthesized organic reagents based on pyrogallol and chelate polymer sorbents, collected literature data over the past 10 years on their use of palladium(II) ion in analytical chemistry. Based on the results of the research, he put forward his proposals when compiling published scientific papers.

## CASE SUMMARY

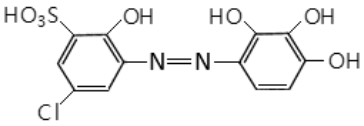
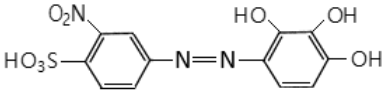
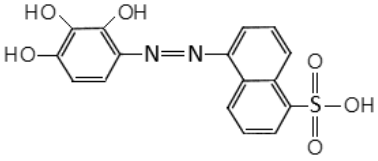
**Reagents and their identification.** Pyrogallol-based reagents are to be widely used in the photometric determination of a number of metal ions. In the presented thesis work, pyrogallol-based azo compounds were used for the photometric determination of palladium (II) ion. These reagents are well in the literature, but in our research they were used for the first time in the photometric determination of palladium(II) ion. The complexes formed by palladium(II) ion with reagents are obtained by the formation of π-dative bonding due to the electronic structure of the donor atoms (N, S) and the empty d-orbital of the metal.

Since the valence orbitals of the platinum series elements are further away from the nucleus than those of other elements, the donor-acceptor interaction between the complexing agent and the ligands becomes easier. Since the reagents are not available, they were synthesized by us ac-

cording to the technique in the research work. The results of identification of reagents by NMR and IR spectroscopy were compared with literature data. The reagents synthesized on the basis of pyrogallol, their formulas and names are given in Table 1.

**Table 1**

**The reagents used in this work are pyrogallol-based**

| Notional sign | Reagent formula | Name |
|---|---|---|
| *R1* |  | 2,2′,3,4-tetrahydroxy 3′-sulfo 5′-chlorazobenzene |
| *R2* |  | 2,3,4-trihydroxy 3′-nitro 4′-sulfoazobenzene |
| *R3* |  | 2,3,4-trihydroxy-phenylazo-5'-sulfonaphthalene |

**Surfactants.** The three reagents synthesized for the investigation were used for the photometric determination of palladium(II) ions. A third component was added to the system to increase the analytical efficiency of these binary complexes. The surfactants used in the complexation reactions were cetylpyridine bromide (SPBr), cetylpyridine chloride (SPCl), Triton X114 and sodium dodecyl sulfate (SDS) as the third component.

The spectrophotometric indices of the complex compounds were calculated. Optimum conditions of complexation were determined from the light spectra. The influence of reagent concentration and its third component, as well as temperature and time on complexation was investigated. The obtained results are presented in Table 2.

As can be seen from the table, the optimal conditions for the formation of complexes PdR1-SPBr, PdR2-SPCl, PdR2-DDS, PdR3-SPCl, PdR3-SPBr in the presence of the third component are shifted towards a more acidic environment compared to binary complexes.

Bathochromic wavelength shift is observed at this time.

**Table 2**

**Spectrophotometric characteristics of the investigated binary and multiligand complex compounds of the palladium(II) ion**

| Com-plex | pH $_{opt.}$ | Wave length, $\lambda_{max}$ nm | Molar absorption Coefficient, $ce_{max.}\cdot 10^{-4}$ | The ratio of compo-nents, M:R:X | The stabilitycon-stant, $lg\beta_1$ | The interval obeying Ber's law, mkg/ml |
|---|---|---|---|---|---|---|
| $PdR_1$ | 4 | 425 | 1,36±0,02 | 1:2 | 4,45±0,05 | 0,426-3,405 |
| $PdR_1$ - SPBr | 3 | 459 | 1,59±0,01 | 1:2:2 | 8,62±0,08 | 0,21-4,26 |
| $PdR_1$ - Triton-X114 | 1 | 463 | 3,2±0,01 | 1:2:2 | 8,1±0,03 | 0,34-4,25 |
| $PdR_2$ | 4 | 440 | 1,26±0,02 | 1:2 | 4,42±0,04 | 0,426-4.256 |
| $PdR_2$ - SPCl | 3 | 457 | 2,42±0,02 | 1:2:2 | 8,10±0,04 | 0,213-0,3405 |
| $PdR_2$ - DDS | 2 | 463 | 2,56±0,01 | 1:2:2 | 9,15±0,05 | 0,213-0,426 |
| $PdR_3$ | 4 | 426 | 1,74±0,01 | 1:2 | 4,80±0,01 | 0.426-2,536 |
| $PdR_3$ - SPCl | 3 | 449 | 2,12±0,02 | 1:2:2 | 8,96±0,02 | 0,213-4,256 |
| $PdR_3$ - SPBr | 3 | 458 | 2,32±0,01 | 1:2:2 | 9,11±0,03 | 0,3405-5,107 |

In this connection, the value of molar absorption coefficient and stability constants of complexes with different ligands are higher. The value of the molar absorption coefficient is higher in PdR1-TritonX-114, PdR2-DDS, and PdR2-SPCl complexes.

The composition ratio of binary and multiligand complexes was investigated by isomolar series, Starik-Barbanel and equilibrium shift

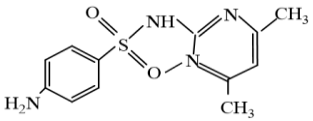
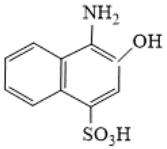
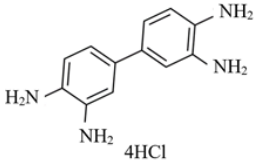
methods. As can be seen from Table 2, the composition of PdR1, PdR2, and PdR3 complexes corresponds to a 1:2 ratio.

The influence of extraneous ions and shielding agents on the formation of binary complex compounds of palladium(II) ion with the presented reagents was investigated. It was found that many ions do not interfere with the complexation reaction. When a third component is introduced into the system, the selectivity in the presence of complexes with different ligands is higher, since complexation shifts to an acidic environment.

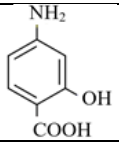
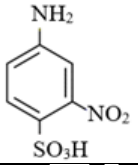
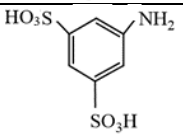
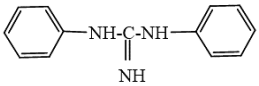
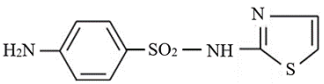
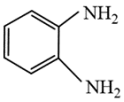
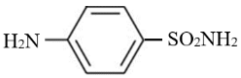
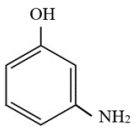
**Sorbents and their synthesis.** In the dissertation work, chelate polymer sorbents containing various amino fragments were synthesized based on a copolymer of maleic anhydride and styrene using a method. The monomers added to the copolymer by chemical modification and their formulas are given in Table 3.

**Table 3**

**Monomers incorporated in copolymers and their structures**

| Monomer designation and name | Monomer |
|---|---|
| MS1 Sulfodimesine |  |
| MS2 - 1-amino-2-hydroxy-4-sulfonic acid |  |
| MS3 - Diaminobenzidine tetrahydroxychloride |  |

**Continuation table 3**

| | |
|---|---|
| MS4 - p-aminosalicylic acid |  |
| MS 5 - 3-Nitro 4-sulfo amino benzene |  |
| MS6 - 2-amino phenol 4,6-disulfoacid |  |
| MS7 - N,N/diphenylguanidine |  |
| MS8 - Norsulf asol |  |
| MS9 - o-phenylendiamin |  |
| MS10 - Streptocide (sulphanilamide) |  |
| MS11 - m-aminophenol |  |

**Investigation of synthesized polymer sorbents and their complexes by infrared spectroscopy method.** It is known that on the basis of the observed spectra of organic and inorganic substances in the infrared range, relevant valuable information is obtained. Sorbents obtained from

the modification of maleic anhydride-styrene sopolymer with amines of various functional groups and polychelate complexes formed by these sorbents with palladium (II) ion were investigated by IR-spectroscopy method. In our studies, IR spectra were drawn in the range of 400-4000 $sm^{-1}$. In the Spectra, a number of absorption bands are observed, characteristic of groups included in the composition of reagents (naturally, as well as complex compounds obtained on their basis). The attribution of these strips to the oscillation of the corresponding chemical links was carried out on the basis of reliable literature materials. Naturally, the main attention in the interpretation of Spectra is paid to the content of functional groups included in the polymer (-$NH_2$,-$NO_2$,-$HSO_3$, etc.) focuses on the characteristic absorption bands of its various oscillations (valence and deformation oscillations).

*The polymer and its complex synthesized by the construction of the monomer MS5 (3-Nitro 4-sulfo amino benzene) to the anhydride styrene copolymer of alein.*In the spectrum of this polymer, absorption bands are observed, which belong to the oscillations of various groups in its composition. It should be noted that the unambiguous interpretation of the observed absorption bands in the range ~1100-1400 $sm^{-1}$ is associated with a number of difficulties. These difficulties are primarily due to the existence of mutual veiling of the bands in that spectral area. It is known that in this range mainly proto-storing groups containing the lightest Atoms (for example OH, NH, etc.).) deformation oscillations, as well as chemical bonds formed by relatively heavy atoms (C-N, C-S, S-O, S=O, etc. the) absorption bands of valence oscillations are observed.

The spectral range of the main interest in our studies is the area of 3000-3800 $sm^{-1}$. It is in this area that the absorption bands of Valence oscillations of the-$NH_2$ and-OH groups, which are part of the polymer, are observed. In the given area, non-sharp, wide absorption bands 3440 $sm^{-1}$ and 3275 $sm^{-1}$ are observed. 3440 $sm^{-1}$ absorption band-belongs to the hydroxyl group, which is included in the $SO_2$-OH group. And the absorption band 3275 $sm^{-1}$ in most cases is attributed to the valence oscillation of the $NH_2$ group. In the low frequency area, absorption bands are observed, which belong to the deformation oscillations of these groups.

The absorption band observed at a frequency of 580 $sm^{-1}$ characterizes the oscillation of the C-S connection. The absorption band 1168 $sm^{-1}$ can be attributed to the valence oscillation of the S=O connection, which is part of the group -$SO_2$-OH, which is directly involved in the adsorption interaction. The oscillation of the S-o connection is characterized by an absorption band of 845 $sm^{-1}$.

The IR-spectrum of chelate complexes formed by this sorbent with palladium (II) ion differs from the spectrum of the sorbent itself mainly in terms of the spectral picture observed in the range of 3000-3800 $sm^{-1}$. Comparison of the corresponding Spectra shows that the wide absorption band observed in the spectrum of the complex at a frequency of 3440 $sm^{-1}$ in the spectrum of the sorbent and attributed to the oscillation of the hydroxyl group is no longer observed with a maximum. In the spectrum of the complex, a very weak integral intensity absorption band is observed in the area 3000-3600 $sm^{-1}$.

The fact is that as a result of sorbent-metal interaction, the Proton of the hydroxyl group is replaced by a metal ion, although not completely. The observed weakly wide absorption band can be considered as a band of aggregations of the valence oscillations of the residual OH - groups and the-$NH_2$ group. The absorption band of the oscillation of the Pd-O connection is observed in the area below 400 $sm^{-1}$ (~320 $sm^{-1}$).The IR-spectrophotometer used in our studies does not have the ability to capture the spectrum in this area.

**Investigation of polymeric sorbents by thermogravimetric analysis.** It is from the literature that differential thermal analysis (DTA) is a method based on the measurement of the temperature difference between the substance under investigation (i.e. sorbent) and an inert (standard) substance. The thermal conductivity and heat capacity of the substance under investigation should be almost the same as that of the reference substance. Also, during heating in the temperature range under investigation, phase transitions and other processes accompanied by absorption or release of heat should not occur in this substance.

If another process occurs during heating of the sorbent under test due to thermal influence, the temperature of the reference substance

differs from the temperature of the substance under test. This difference is recorded as peaks on an instrument called a derivatograph. These peaks are called endo- or exo-peaks. The curve above the above peaks is called DTA curve. By the shape of the DTA curves, the area of the peaks, the values of the temperature corresponding to the maximum on the curves, you can get information about the structure of the sorbent under investigation and other processes occurring. in this sorbent with increasing temperature. The exo- and endo-effects correspond to a number of processes. Thus, the exo-effect corresponds to the processes of crystallization and oxidation, and the endo-effect corresponds to the processes associated with melting and decomposition of the structure. If different substances are separated from the investigated substance (sorbent) during heating, this process is observed mainly due to the endoeffect. During this process, the mass of the sorbent changes. It is that the method of thermogravimetric analysis (TG) researches the change in mass of the substance under investigation as a function of temperature.

*Thermogravimetric analysis of the sorbent synthesized by addition of MS8 monomer (norsulfazole) to the copolymer of maleic anhydride and styrene.* The thermogram of the sorbent containing the amine moiety of norsulfazole demonstrate that the peak is observed at 209,83 $^0$C. At this time, the enthalpy value is 130,3469 J/g and at temperature 391,680 $^0$C the enthalpy value is 594,9851 J/g. As can be seen from the DTA curve, the initial mass change occurs at a temperature of 70,05 $^0$C and the enthalpy of the process is 57,45 J/g. The last mass change occurs at a temperature of 724,14 $^0$C and the enthalpy is 62,84 J/g.

*The chelate complex formed by palladium (II) ion sorbent synthesized by the addition of MS8 monomer (norsulfazole) to maleic anhydride-styrene copolymer was investigated by thermogravimetric analysis.* According to the polychelate thermogram, peaks were observed at 351,0 $^0$C, 626,1 $^0$C, 560,0 $^0$C and 779,6 $^0$C. According to the TQ curve, 50.26% of the residue remained at 798,1 $^0$C and the mass loss was 49,74%. By comparing both thermogravimetric curves, it was seen that the polychelate was more resistant to temperature.

From the thermogravimetric and differential thermogravimetric

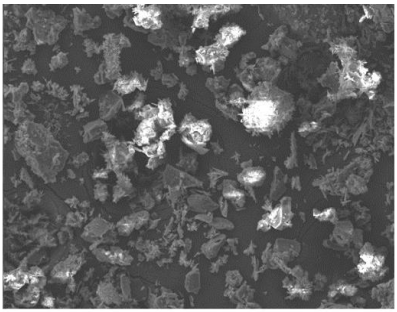
curves, it can be concluded that chelate complex compounds containing palladium(II) ions are more stable to temperature than polymeric sorbent. This law is also true for other synthesized sorbents.

**İnvstigation of polymeric sorbents and metal sorbent complexes by ultraviolet analysis.** It is known from the literature that UV spectroscopy is a method that involves obtaining, researching and using absorption, reflection spectra in the ultraviolet field. This method is distinguished by high sensitivity, accuracy and high speed of analysis, as well as simplicity of experimental methods and devices, sufficient content of a small amount of substance required for research. All organic matter is absorbed in the ultraviolet field. s the sorbents used in the research work are organic matter, UV spectra of the MS1 sorbent and its complex formed by palladium (II) ion were also studied using UV spectroscopy method. Our studies were carried out in the field of wavelengths of 200-700 nm. The ultraviolet spectra of polyxelate differ from each other in their shape and maximum values of wavelengths in comparison with the spectra of the sorbent.

**Scanning electron microscopy analysis of synthesized sorbents and chelates formed by these sorbents with palladium (II) ions.** In this work, a sorbent (MS1) synthesized on the basis of maleic anhydride-styrene copolymer and sulfodimazine monomer and the complexes formed by this sorbent with palladium(II) ion were investigated on scanning electron microscope JSM-6610 of Japanese company JOEL. With this microscope, the appearance of the substance can be magnified 300,000 times. The polymeric sorbent synthesized as a result of the research was magnified 6000 times. At this time, a SEM picture was obtained and it was apparent from initial observations that the polymeric sorbent was quite porous. The distribution map of the elements in the polychelate complex formed by the sorbent of palladium (II) MS1 is given (fig.1. (b)). According to this map, it was determined that the complex contains S, O, C and Pd. It should be noted that O, S and Pd were distributed homogeneously on this map. Due to the fact that the sorbent is an organic compound, the electronic image is almost completely repeated during the distribution of the carbon atom.

The number of elements and the distribution map can also be

determined using this method. In addition to the scanning electron microscope, there is a special part of the instrument called the X-MAX electron spectrometer (20 $mm^2$), which is an analyzer that determines the chemical composition of the substance. In this case, rays fall on the substance from the analyzer, and the rays of metals, namely palladium(II), are registered in the analyzer.



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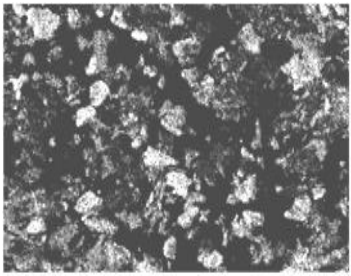

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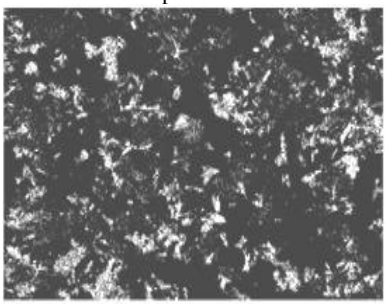

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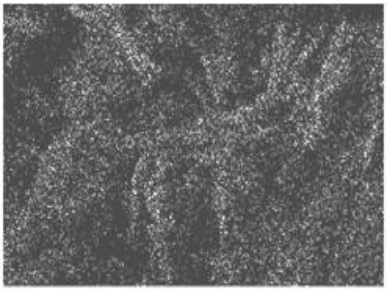
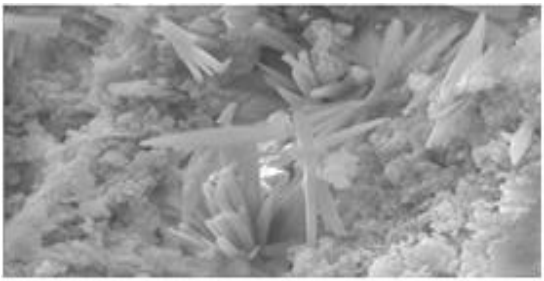
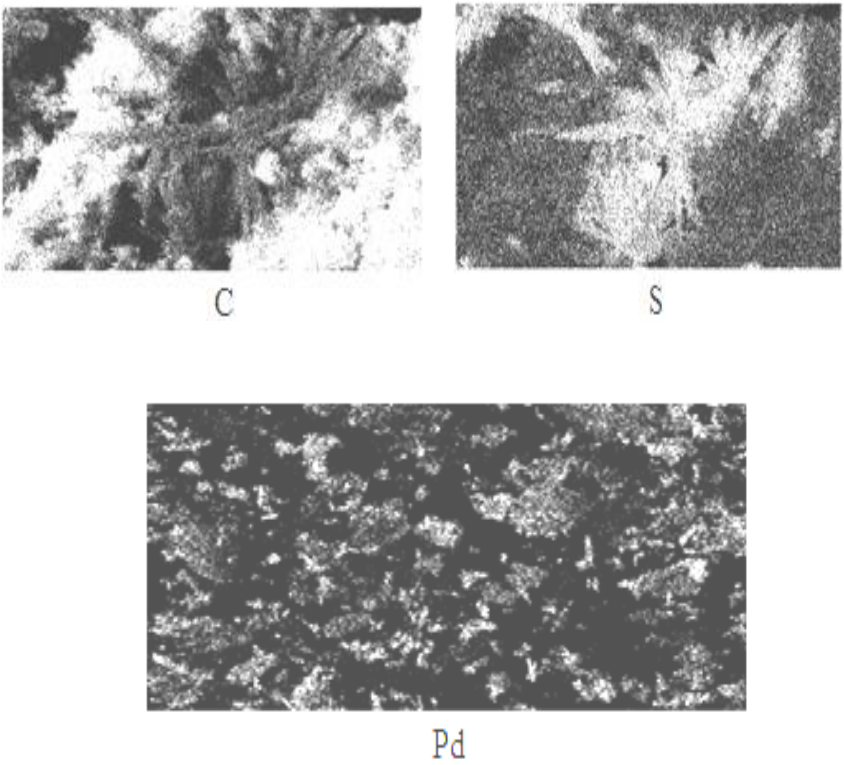


N



O

***a)***




***b)***

**Figure 1. SEM image of the polymeric sorbent (a) obtained on the basis of maleic anhydride-styrene copolymer and sulfodimazine-fragmented amine (a) and its complex with palladium(II) ion (b)**

As can be seen from Figure 1, the palladium(II) ion was not uniformly distributed on the sorbent surface and formed a mold with different structure. The energy spectra of the fragmented sulfodimesis sorbent (Figure 2) and the chelate complex with palladium ion formed by it (Figure 3) were constructed. The energy spectra demonstrate that palladium (II) ions penetrated into the macromolecules of the sorbent and formed a stable chelate complex.

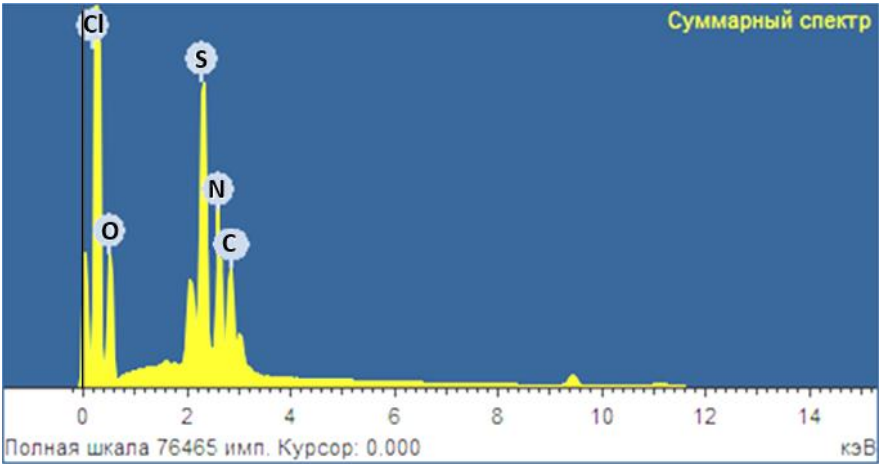



**Figure 2. Energy spectra of elements in sulfodimesine-fragmented sorbent**

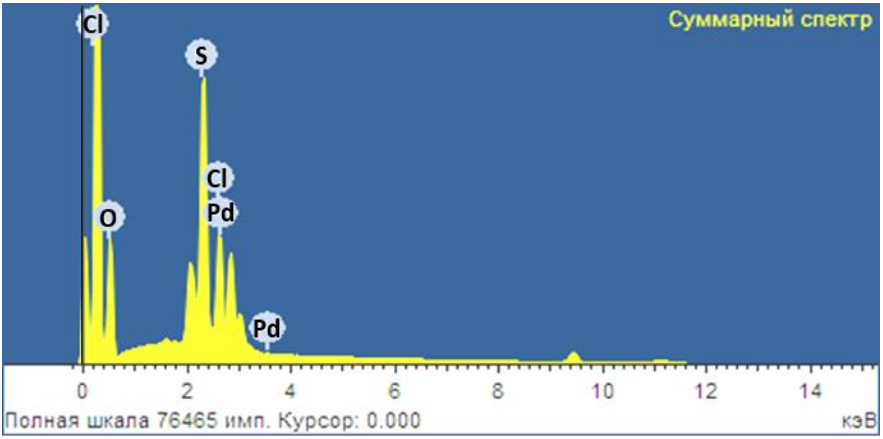



**Figure 3. Energy spectra of the complex of sulfodimesis fragmented sorbent with palladium (II).**

The mass fractions of elements contained in the sorbent are summarized in Table 4, and the mass fractions of elements in the chelate complex are summarized in Table 5.

**Table 4**

**Results of MS1 sorbent investigation under electron microscope**

| Element | Mass % | Atom% | Composition % | The formula |
|---|---|---|---|---|
| C | 22,45 | 29,88 | 84,27 | $CO_2$ |
| N | 1,47 | 1,62 | 4,33 | $N_2O_5$ |
| S | 1,68 | 2,55 | 3,6 | $SO_3$ |
| Cl | 8,2 | 0,54 | 0,00 | - |
| O | 66,2 | 65,41 | 7,8 | - |
| Result | 100,00 | | | |

**Table 5**

**Results of electron microscopic investigation of the complex of sorbent MS1 with palladium (II) ion**

| Element | Mass % | Atom% | Composition % | The formula |
|---|---|---|---|---|
| C | 23,03 | 29,92 | 84,42 | $CO_2$ |
| N | 1,36 | 1,51 | 6,15 | $N_2O_5$ |
| S | 1,55 | 0,75 | 4,38 | $SO_3$ |
| Cl | 1,46 | 0,64 | 0,01 | - |
| Pd | 4,38 | 0,65 | 5,04 | PdO |
| O | 68,22 | 66,53 | - | - |
| Result | 100,00 | | | |

According to the distribution map of elements in the chelate complex of palladium with polymer sorbent, S, C, O, and Pd were observed. According to the distribution map of the chelate complex formed by the polymeric sorbent with palladium (II) ion, Pd, O, S were distributed homogeneously. And the distribution of C almost completely repeats the electronic image. This distribution further proves that the sorbent is an organic compound.

The investigation of polymeric sorbents and their chelate complexes with palladium (II) ions by scanning electron microscopy once again established that the palladium (II) ion forms a stable complex

association with the functional analytical groups contained in the sorbent.

**Determination of palladium(II) ion by crystallization method on polymeric chelating sorbents.** The sorption ability of palladium(II) ions of polymeric sorbents obtained by modification of maleic anhydride-styrene copolymer with various amines was investigated. The effect of pH, metal ion concentration and ionic strength of the solution on the concentration of metal ions by chelating sorbents was investigated. As a result of experiments, it was found that the value of solution ionic strength increases to 0.6-1.0 mol/L in all sorption systems investigated by us. This value of ionic strength does not significantly affect the sorption process. Further increase in the value of ionic strength causes an almost gradual decrease in the degree of sorption. As a result of researches it was found that the process of sorption of palladium(II) ions by polymers MS2, MS3, MS6, MS7, MS9 has an ionic strength of 0.6; 0.8 ionic strength to the process of sorption by polymers MS1, MS4, MS8; The process of sorption by polymers MS1, MS10, MS11 is not affected by increasing the ionic strength to 1.0.

The time dependence of the sorption process was investigated and the moment of sorption equilibrium was determined. It was found that the complete sorption of palladium(II) ion by polymeric sorbents occurs within 1-2 hours. Thus, the sorption equilibrium of palladium (II) ion with polymers MS1, MS2, MS3, MS5 is established in 1 hour, with polymers MS2, MS8, MS11 - in 1.5 hours, and with polymers MS4, MS6, MS10, MS11 - in 2 hours. The effect of environmental acidity on the sorption process was investigated. The maximum sorption of palladium(II) ions by all investigated sorbents occurred in the pH range of 3-6.

The maximum sorption capacities of the synthesized polymeric sorbents for the determined palladium(II) ions were calculated. It was found that the sorption capacity of the sorbent increases with increasing concentration of palladium (II) ions in solution. It mainly depends on the number of donor atoms, i.e. N, O, S, in the functional groups contained in the sorbent. After a certain time, the sorption capacity

does not change. This is due to the fact that the functional groups involved in the reaction in the macromolecules contained in the sorbent are completely filled with metal ions. Depending on the initial concentration of palladium(II) ion, the indices of the sorption process change. As can be seen from Table 6, the highest sorption capacity was observed in the sorbent sulfodimesine-amino fragment (MS1).

**Table 6**

**Main parameters of palladium(II) ion sorption on polymeric sorbents**

| Sorbent | Sorption capacity ST, mg/g | Degree of sobrtion R,% | $pH_{opt.}$ | Ionic force μ*, mol/l | Time, hour |
|---|---|---|---|---|---|
| MS1 | 523 | 98 | 5 | 0,8 | 1,0 |
| MS2 | 521 | 98 | 4 | 0,6 | 1,0 |
| MS3 | 513 | 97 | 3 | 0,6 | 1,0 |
| MS4 | 512 | 99 | 4 | 0,8 | 2,0 |
| MS5 | 510 | 96 | 4 | 1,0 | 1,0 |
| MS6 | 490 | 90 | 5 | 0,6 | 2,0 |
| MS7 | 487 | 97 | 4 | 0,6 | 1,5 |
| MS8 | 451 | 98 | 6 | 0,8 | 1,5 |
| MS9 | 407 | 98 | 5 | 0,6 | 1,5 |
| MS10 | 354 | 96 | 6 | 1,0 | 2,0 |
| MS11 | 276 | 98 | 4 | 1,0 | 2,0 |

$\mu^*$- ionic strength value causing a significant decrease in the sorption rate

**Desorption of palladium(II) ion from polymeric chelating sorbents.** The desorption of sorbed palladium from the sorbent was investigated. Initially, the effect of different mineral acids ($HClO_4$, $H_2SO_4$, $HNO_3$, HCl) of equal hardness on the desorption process was investigated. Then experiments were conducted with different concentrations of optimum acid and the optimum acid concentration for the desorption process was determined. From the experiment, it was found that HCl and $HclO_4$ have higher acid desorption ability than other acids. The results of desorption of palladium(II) ions from polymer sorbents are presented in Table 7.

**Table 7**

**Sorption-desorption indicators of palladium (II) ion on polymeric sorbents**

| Acid | Sorbent | Contentration, mol/l | Volume ml | Degree of desorption, % | Acid | Sorbent | Concentration, mol/l | Volume, ml | Desorption degree, % |
|---|---|---|---|---|---|---|---|---|---|
| HCl | MS1 | 0,5 | 10 | 93 | $HClO_4$ | MS7 | 0,5 | 10 | 95 |
| | | 1,0 | 10 | 95 | | | 1,0 | 10 | 96 |
| | | 2,0 | 5 | 98 | | | 2,0 | 5 | 97 |
| $HClO_4$ | MS2 | 0,5 | 10 | 95 | $HClO_4$ | MS8 | 0,5 | 10 | 94 |
| | | 1,0 | 10 | 97 | | | 1,0 | 10 | 96 |
| | | 2,0 | 5 | 98 | | | 2,0 | 5 | 98 |
| HCl | MS3 | 0,5 | 10 | 94 | $HClO_4$ | MS9 | 0,5 | 10 | 95 |
| | | 1,0 | 10 | 95 | | | 1,0 | 10 | 96 |
| | | 2,0 | 5 | 97 | | | 2,0 | 5 | 98 |
| $HClO_4$ | MS4 | 0,5 | 10 | 96 | $HClO_4$ | MS10 | 0,5 | 10 | 93 |
| | | 1,0 | 10 | 98 | | | 1,0 | 10 | 95 |
| | | 2,0 | 5 | 99 | | | 2,0 | 5 | 97 |
| $HClO_4$ | MS5 | 0,5 | 10 | 93 | $HClO_4$ | MS11 | 0,5 | 10 | 95 |
| | | 1,0 | 10 | 95 | | | 1,0 | 10 | 97 |
| | | 2,0 | 5 | 97 | | | 2,0 | 5 | 98 |
| $HClO_4$ | MS6 | 0,5 | 10 | 87 | | | | | |
| | | 1,0 | 10 | 89 | | | | | |
| | | 2,0 | 5 | 90 | | | | | |

**Investigation of the sorption equilibrium of the sorbent with respect to the palladium(II) ion using the Freundlich and Langimur isotherm models.** The sorption process can be interpreted in more detail using experimental sorption isotherms. It is more convenient to ensure temperature stability by these isotherms than by other parameters. It is that sorption isotherms characterize the dependence of sorption capacity and sorption capacity on the solution concentration of sorbed components at constant temperature. For a more detailed investigation of the interfacial interaction in the sorption of palladium (II) ions, the Freundlich and Langmuir monomolecular adsorption isotherm models characterizing multilayer sorption were used.

Freundlich adsorption isotherm. It is an adsorption isotherm model that characterizes multilayer sorption. The equation of this model is used to express the adsorption isotherm over a range of average pressure and density values. The Freundlich adsorption isotherm is applied to phases with energetically inhomogeneous surface in sorption systems and is presented in Fig. 3.

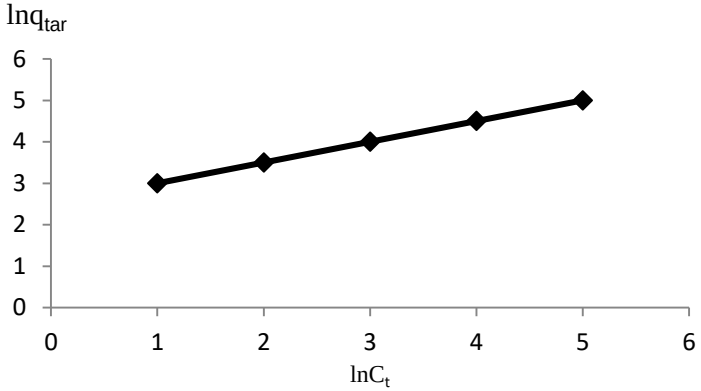



**Fig. 3. Freundlich sorption ion palladium (II) synthetic sorbent, fragmented NN/-diphenylguanidine.**

On the basis of the Freundlich isotherm, the parameters of the isotherm were calculated and the possibility of describing the sorption isotherm by this model was established. At very small and enormous values of hardness, the experimental results do not coincide with the values obtained from the equation. Therefore, it is more convenient to express the sorption results using the Langimura model.

**The model of Langimur l.** It is from the literature that the Langimour model characterizes monomolecular sorption. The adsorption phenomenon is localized (molecules cannot move on the surface), and the forces causing it are close to the chemical forces of nature. According to the Langimura model, the sorption process does not occur on the entire surface of the sorbent, but on active centers always present on the surface. The dependence of the sorption results $\frac{C_e}{q_e}$ and f ($C_e$) is represented in Figure 4.

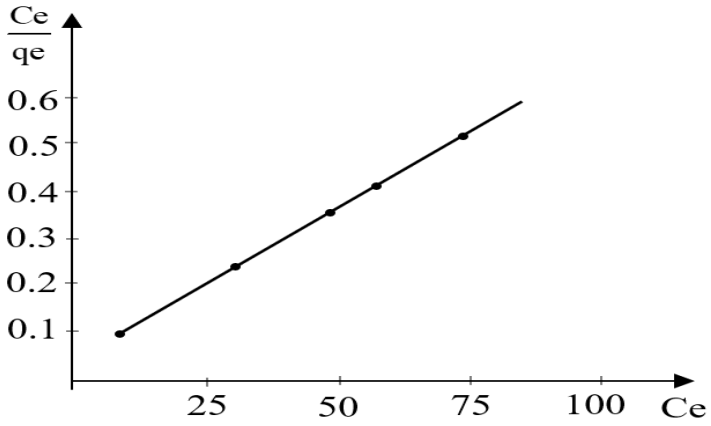



**Fig. 4. Langimur isotherm of sorption of palladium (II) ion by NN'-diphenylguanidine fragmented synthetic sorbent in linear coordinates.**

As can be seen from the graph, the Langmur curve is more linear. Therefore, the sorption of Pd (II) ion by the synthesized polymer sorbent occurs due to chemical forces. Considering what has been said, and based on the calculated isothermal parameters of the Langimur isotherm, it can be said that it is more appropriate to express the sorption of palladium (II) ion on the polymer sorbent with the Langimur model.

Unlike the Freienfeld equation, the Langmuir equation describes adsorption in a wide range of pressures and concentrations quite well.

Methods of sorption-spectrophotometric determination of palladium (II) ion in environmental facilities. In the presented thesis, methods of sorption-spectrophotometric determination of palladium (II) ion were developed. The sorbents obtained are identified by various physico-chemical methods and the optimal conditions for their adsorption on palladium(II) ions are determined. Eleven methodologies were developed and tested at various sites.

*Determination of Pd(II) ions in nickel powder by a sorbent-based sulfodiesidine (MC1) condensation.* With the purpose of preparing the collected sample for analysis, 2,0 g of samples (0,028% Pt; Pd 0,071%; Rh 0,0018-0.0021%; Ir 10-4%; Ru 10-5%; Ni-base) were preheated in a glass. , and then dissolved in 20 ml of gold. After the sample is dissolved, the sample is steamed. The evaporation continues

until a saturated solution is left. The remaining liquid is dissolved by adding 20 ml of distilled water. The evaporation continues until a dry residue is obtained. It is then dissolved by adding *Determination of palladium ion in standard magmatic rock (M03) by sorbent solidification on the basis of 1-aminophenol-2-hydroxy-4-sulpho oxides (M02).* Based on a synthetic sorbent based on 1-aminophenol-2-hydroxy-4-sulphuric acid, the method for determining the concentration of palladium (II) ion in standard mountain magmatic rock (MO-3) has been developed. Chemical compounds in the rock are given as percentages: CaO-15,75%,$Al_2O_3$-13,67%, $Fe_2O_3$-8,48%, FeO-9.05%, MgO-8,66%, $Na_2O$-0,72%, $K_2O$-0,204%, $TiO_2$ -1.46%, $P_2O_5$-2,15%, MnO - 0,222%, S-0,124%, F-0,072, 0,05 g/t Pd, 0,008 g/t Pt. Experiments were conducted according to the above mentioned method. When 0,01 g of the sample is dissolved in a suspension at 50°C, 1 ml HCl, 2-3 drops of nitric acid and 10 ml of distilled water are added. The resulting mixture is dissolved. Optimal sorption conditions for the reaction are created by using $HNO_3$, the analysis is performed by concentrating with MS2 sorbent.

10 ml of $HNO_3$ or HCl at a concentration of 0,01M. The optimum pH value of sorption was obtained with 0,01 M solution of acid $HNO_3$. At the end it is condensed and analyzed with sorbent MS1. The experiment found that palladium (II) in nickel powder is 1,39 mg/t.

Results of the experiments at both sites are displayed in table 8.

**Table 8**

**Pd(II) ion concentration (%) in nickel powder and standard MO-3 (sample volume 1000 ml; volume of eluent 5 ml; $m_{sorb.}$=100,000 mq; P=0,95; n=5)**

| Objekt | Sorbent | Pd(II) passport indicator mq/t (q/t) | Pd(II) was determined, mq/t $\bar{x} \pm \frac{t_P s}{\sqrt{n}}$ |
|---|---|---|---|
| Nickel powder | MS1 | 1,42 | 1,39±0,003 |
| Magmatic Rock | MS2 | 0,05 | 0,049±0,001 |

The researches conducted have shown that the proposed 11 sorbents are suitable for the photometric determination of palladium(II) ions. Polymer sorbents used in the determination of palladium(II) ion concentration have higher sorption indices (ionic force, sorption capacity, sorption equilibrium, analysis time, etc.) than from the literature sorbents. The polymer sorbents used in the research can be reused for almost 7-8 cycles.

## RESULTS

1. In the presented work had been synthesized 11 new chelated polymer sorbents based on modification of the copolymer of maleina-styrene anhydride-styrene by various amines for selective fixation of palladium(II) ion and carried out their identification by IR spectroscopy, UV spectroscopy, SAM, The DTA methods have determined their physico-chemical properties. Based on the DTA method, it has been established that the investigated polymer sorbents are unstable at temperatures above 100-120$^0$C. Based on SAM-analysis it was noted that the polymer sorbents are quite porous and in the adsorbent macromolecules there are traces of palladium (II) ions.
2. The usage pyrogallol-based nitrogen compounds, binary and multilinanguenic ion complexes of palladium(II) have been investigated. Their main spectrophotometric characteristics are determined and it has been researched that external ions and shielding agents do not prevent complex formation. The sensitivity and selectivity of complex compounds to palladium(II) ion was researched, and it was found that complexes with different ligands have higher analytical properties than binary complexes.
3. The optimal conditions of sorption of palladium(II) ions on synthesized sorbents were researched. The influence of various factors on the sorption process was researched: pH, ionic force, sorption time and metal density. The sorption capacities of the sorbents were calculated and it was found that with increasing number of donor atoms, namely N, S and O in the sorbent, sorption capacity of the sorbent on palladium (II) ions increases. The results of the calculation showed that sorbents MS1, MS2, MS3, MS4 have the highest sorption capacity, and sorbents M10 and M11 have the lowest results.

4. The influence of different mineral acids on palladium(II) ion desorption has been researched and it was found that $HclO_4$ acid better desorbs palladium from sorbents.
5. Using the Freyindlja adsorption istherm and the monolecular Langimora, which characterize multilayer sorption, the interphase interaction of palladium(II) ion was investigated. It has been established that the sorption of palladium(II) ions by synthetic polymer sorbents is due to chemical forces, and the calculated single-molecular sorption capacity of the Langimura model is higher.
6. Methods for the sorption-photometric determination of palladium (II) ion micro quantities in standard magmatic rocks (M03) with sorbent MS2 and nickel dust with sorbent MS1 have been developed. The new method is simple compared to other methods and is characterized by a good reproducibility. The methods presented provide accuracy of results in real objects analysis (accuracy confirmed by addition and passport indicators).

**The main results of the thesis are reflected in the following publications**


1. Abilova, U.M., Hashimova, E.N., Chiragov, F.M. Study of sorption of $Pd^{2+}$ ion by polymer sorbent containing sulfodimezine fragment // XI republican conference of doctoral students, masters and young researchers dedicated to the 94$^{th}$ anniversary of the national leader Heydar Aliyev's "Actual problems of chemistry", BSU, – Baku: – 2017. – p. 119.
2. Maharramov, A.M., Abilova, U.M., Hashimova, E.N., Chiraqov, F.M. Study of sorption of palladium (II) ion by polymer sorbent containing NN/ diphenyl guanidine fragment // International scientific conference dedicated to the 85th anniversary of Academician Rafiga Alirza Aliyeva: "Chemistry of coordination compounds: actual problems of analytical chemistry", BSU, – Baku: – 2017. – p. 108.
3. Abilova, U.M., Hashimova, E.N., Chiraqov, F.M. İnvestigation of determination of palladium (II) ion by concentration with NN´-diphenyl guanidine fragmented chelate forming sorbent // – Austria: Austrian Journal of Technical and Natural Sciences, – 2018. №1-2, – p. 88-91.

4. Abilova, U.M., Hashimova, E.N., Chiragov, F.M. Separation of Pd(II) ion by condensation with a newly synthesized sorbent // "Ecology and soil sciences in the 21st century" dedicated to the 95th anniversary of the national leader Heydar Aliyev's birth current problems" materials of the VII Republican Scientific Conference, – Baku: – 2018. – p. 72-73.
5. Abilova, U.M., Hashimova, E.N., Chiragov, F.M. Study of the sorption of palladium (II) ion with a synthetic sorbent containing 1 amino 2 hydroxy 4 amine with sulfo acid fragments // "Actual problems of chemistry" XII of doctoral students, masters and young researchers dedicated to the 95th anniversary of the birth of the national leader Heydar Aliyev republican conference, BSU, – Baku: – 2018. – p. 169-170.
6. Hashimova, E.N., Abilova, U.M., Chiragov, F.M. Study of sorption of palladium ion of polymer sorbents containing a fragment of sulfodimezine and NN/diphenylguanidine //– Nijni Novgorod: XXI All-Russian Conference of Young Scientists and Chemists (With international participation) National Research Nijni Novgorod State University. N.I.Lobachevsky, – Nizhny Novgorod: – 2018. – p. 279.
7. Hashimova, E.N., Abilova, U.M., Chiragov, F.M. Study of the sorption of palladium(II) ion with a synthetic sorbent containing p-amino salicylic acid amine // Proceedings of the "Naghiyev readings" scientific conference dedicated to the 110th anniversary of Academician M. Nagiyev, ANAS Institute of Catalysis and Inorganic Chemistry, – Baku: – 2018. - p. 230.
8. Abilova, U.M. Hadjieva, S.R., Hashimova, E.N., Chiraqov, F.M. Sorption concentration of palladium with a polymer sorbent containing a fragment of diaminobenzidine // Baikal School-Conference in Chemistry Collection of abstracts of the II All-Russian School-Conference dedicated to the 100th anniversary of Irkutsk State University and the 85th anniversary of the Faculty of Chemistry of ISU, Ircutsc, – 2018. – p. 71
9. Abilova, U.M., Hashimova, E.N., Chiragov, F.M. Sorption-photometric determination of palladium (II) ions with a polymer sorbent // – Warsaw: East European Science Journal, – 2018. – part 2, – p. 46-51.
10. Abilova, U.M., Hashimova, E.N., Chiragov, F.M. Determination

of palladium (II) in igneous rock after concentration with a chelating sorbent // Waste, reasons for its formation and prospects for use Collection of scientific papers based on the materials of the International Scientific Ecological Conference, “Kuban State Agrarian University named after I.V. T. Trubilin”, – Krasnodar: – 2019. – p. 167-169.

11. Abilova, U.M., Hadjieva, S.R., Hashimova, E.N., Chiragov, F.M. Sorption of palladium(II) ions by chelating sorbents // XXIX Russian youth scientific conference with international participation, dedicated to the 150th anniversary of the Periodic Table of Chemical Elements Ural Federal University, Yekaterinburg: – 2019, – p. 68.
12. Hashimova, E.N., Abilova, U.M. İnvestigation of the Pd (II) ion's sorption by synthetic sorbent containing 2 amino phenol 4-6 disulfo acid fraqment // XXIX Российская молодежной научной конференции с международным участием, посвященной 150-летию Периодической таблицы химических элементов Уральский Федеральный Университет, – Екатеринбург: – 2019. – s. 163
13. Abilova, U.M., Hashimova, E.N., Hajiyeva, S.R., Chiragov, F.M. Spectrophotometric sorption of palladium (II) ions with maleic anhydride styrene copolymer-norsulfazolum system // Advanced in synthesis and complexing, Book of abstracts The fifth international scientific conference, RUDN University, – Moscow: – 2019. – vol. 1, – p. 85.
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