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Synthesis and Study of a New Polymeric Sorbent, Based on O-Phenylenediamine, for Purifying Water from Heavy Metals

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Abstract

The presented work is devoted to the preparation of a new polymer sorbent, based on o-phenylenediamine, which has a high sorption capacity for heavy metal ions, as well as the study of its physicochemical and sorption properties. A new polymer sorbent (which we called OPDEP) was obtained, based on o-phenylenediamine, epoxy resin and formaldehyde. Ethylenediamine was used as a polymerizing and binding agent during the synthesis. It has been established, that the reaction occurs at a molar ratio of 1:2:2:2 of the starting substances: epoxy resin, ophenylenediamine, formalin and ethylenediamine. Were studied optimal conditions for the processes of sorption and desorption for the synthesized sorbent. As a result of the studies, it was established, that this sorbent has a high absorption (sorption) capacity to Cu^{2+} , Co^{2+} , Cd^{2+} , Zn^{2+} , Ni^{2+} ions. The static exchange capacities (SEC) of the sorbent with respect to the indicated ions were determined. It has been studied the dependence of the sorption process on the pH value of the environment. It has been established, that the static exchange capacity of the polymer sorbent has the highest value in the pH range = 4-6.

The structure of the new sorbent was confirmed by IR spectroscopy. The IR spectra of the polymer and the initial key reagent, o-phenylenediamine, were compared. The composition and structure of the surface layers, as well as the elemental analysis of the polymer, were carried out by Scanning Electron Microscopy (SEM). The thermal stability of the new adsorbent was studied by thermogravimetric analysis (TGA). The ratio of crystalline and amorphous phases in the polymer was determined by X-ray phase analysis. It has been proven that the OPDEP sorbent is reusable, that is, it can be cleaned of heavy metals. It allows you to isolate Cu²⁺, Co²⁺, Cd²⁺, Zn²⁺, Ni²⁺ ions from complex mixtures with high efficiency.

Keywords: Phenylenediamine; Polymeric; Heavy Metals; Synthesis; Ethylenediamine

Introduction

Currently, along with the rapid development of production and industrial enterprises, the amount of man-made waste, entering the environment, is continuously increasing. [1]. According to some calculations, if production continues to grow at the same pace, humanity will have enough iron for 250 years, copper for 29 years, and tin for 35 years, and most of the consumed metals will end up in the aquatic environment in the form of ions [2]. Therefore, currently one of the most pressing problems of modern science is the protection of drinking water from heavy metal ions that enter it [3].

Some of the main sources of water pollution are industrial and agricultural wastewater, oil and petroleum products [4]. But the most toxic are wastewater from industries that use non-ferrous metals and radioactive chemicals [5]. Water pollution with toxic substances such as heavy metals, aromatic compounds and chemical dyes is one of the most important environmental problems of our time. [6]. An increase in the concentration of non-ferrous and heavy metals in industrial and waste waters has an extremely toxic effect on living organisms [7].

Currently, various purification methods are used to purify industrial and natural water from heavy metal ions: ion exchange, evaporation, electrodialysis, sorption and reverse osmosis [8]. However, in recent years, it is sorbents that have found wide application in the extraction of noble metals from complex mixtures during the treatment of industrial wastewater [9]. Since the use of sorbents is highly effective and cheap, this method of water purification has advantages over others [10].

In this regard, in our research work we have obtained a new polymer- adsorbent, based on o-phenylenediamine. The choice is due to the fact, that o- phenylenediamine is one of the important reagents in coordination chemistry and polymer chemistry due to the presence of two adjacent amino groups and active proton-acceptor properties [11]. It has been proven that complexing sorbents obtained on the basis of cross-polymerization of the o-phenylenediamine molecule have high selectivity to silver and mercury ions [12]. Sorbents, based on o- phenylenediamine and graphene oxide, are already used to extract Pb(II) ions from wastewater [13]. Polymer sorbents, based on o-phenylenediamine and starch, are used to extract Ni(II) cations from complex mixtures [14]. The main part of o- phenylenediamine is used as a component in the preparation of herbicides and fungicides and a ligand in coordination chemistry [15].

Due to the presence of various heterofunctional groups in polymer sorbents, the number of bonds forming complexes with metal ions increases [16]. In addition, the high selectivity of the sorbent with respect to metal ions depends on its morphology, ability to form complexes with metal ions, stability during sorption and desorption processes and other physicochemical properties [17].

In addition to everything, you need to understand that in the sorption process the selective action of the sorbent is important [18]. To do this, the sorbent composition should not contain active centers that slow down the sorption process [19]. In addition, the pH value of the medium affects the equilibrium and is one of the important parameters controlling the sorption process [20]. The regenerative ability of sorbents is also closely related to the pH value of the environment [10].

According to the International organization Global Footprint Network, mankind today has exhausted natural resources, namely water, air, soil, which the planet can restore in a year(resource use is 1.75 times higher than restoration) [2]

Conservation and restoration of water resources are one of the most pressing issues of our time with a lack of clean water or its unsatisfactory quality in developing countries.

Wastewater (washing and spent concentrated solutions) of galvanic plants to a large extent contains heavy metal ions [21], which are not only highly toxic, but also valuable components. Indeed, in our time, for all countries, the problem of the loss of valuable metals and their removal from wastewater from galvanic plants is becoming urgent. One of the main tasks is the development of new methods of treatment, disinfection, neutralization and disposal of contaminated wastewater from industrial enterprises. So, the purification of industrial effluents from heavy metal ions is very relevant for Ukraine and other countries of the world. This problem can be solved by introducing lowwaste technologies for the extraction of valuable metals from industrial wastewater.

Thus, one of the important tasks, facing the chemical industry today, is the production of cheap chemically and thermally stable sorbents with high selectivity towards metal ions and their use on an industrial scale [22]. That is why the purpose of our research is to synthesize a new polymer sorbent, based on o-phenylenediamine, and study its adsorption abilities towards heavy metal ions. To achieve this goal, the following tasks were:

1. Synthesis of a new polymeric sorbent based on ophenylenediamine, capable of absorbing heavy metal ions;

2. Study of the chemical composition and physico-chemical properties for the resulting polymer;

3. Selection of optimal conditions for sorption processes of toxic metal ions (Ni^{2+} , Cu^{2+} , Co^{2+}) from aqueous solutions.

Obviously, the main specific goal of our research is making contribution to the ability of modern science to purify water resources from toxic metal ions. The new polymer sorbent, that we have obtained, must have high performance properties (thermal stability). Its synthesis should be fast and easily reproducible, and the components for its preparation should be commercially available. And most importantly, the new polymer must have the ability to regenerate in order to be able to use it not in one time, but in many cycles for purifying industrial wastewater from metals.

Experiment

IR spectra of complex compounds were recorded in the range 400-4000 cm⁻¹ on an IR Fourier spectrometer IRTracer 100 (Shimadzu, Japan). Thermoanalytical studies were carried out on a Netzsch STA 409 PG analyzer (Germany) in aluminum crucibles in an inert nitrogen atmosphere at a nitrogen flow rate of 50 ml/min. The temperature sensor was a K-type thermocouple (Low RG silver). The temperature measurement range was 25-370 °C, heating rate was 5 degrees/min, sample volume per measurement: 5-10 mg.

Microscopic studies of the surface of the synthesized sorbent were carried out using a scanning electron microscope (SEM) - MIRA 2 LMU, equipped with an energy-dispersive microanalysis system INCA Energy 350. SEM analysis was carried out in high vacuum. The elemental composition of the sorbent was analyzed in an electromagnetic field with an accelerating voltage of 20 keV and a current of 1 nA.

The X-ray phase characteristics of the resulting polymer sorbent were determined on a Panalitik Empyrean diffractometer with a voltage of 40 kV and a current of 30 mA, and the diffraction patterns were analyzed, using computer programs OriginLab and Profex.The preparation of a polymer, based on epoxy resin (ED-20), formaldehyde, o-phenylenediamine and ethylenediamine at a ratio of 1:2:2:2 was carried out as follows:

12.9 g (30 mmol) epoxy resin (grade ED-20, which means "epoxy-dian resin with at least 20% epoxy groups in the composition") and 6.48 g (60 mmol) o- phenylenediamine were placed in a flat-bottomed flask, equipped with a magnetic stirrer and reflux condenser. Mixing was carried out first at 30-35 °C during 20 minutes, then for 1 hour at 85-90 °C. The resulting reaction mixture was cooled to a temperature of 60 °C, and there was added 4.8 ml of 40% water solution, containing 1.8 g (60 mmol) of formaldehyde. Then new mixture was stirred for 1 hour on a magnetic stirrer at the same temperature. Then, to carry out polycondensation, it was added 2 ml of ethylenediamine (60 mmol), and mixture was stirred at a temperature 80 °C during 1 hour. As a result, a yellow resinous mass was formed. The resulting mass was poured into a porcelain bowl and dried in an oven at 80-85 °C during 24 hours. The dried polymer was washed several times with distilled water and dried again in a thermostat at 45-50 °C diring 5 hours. As a result, a yellow solid porous compound was obtained - a polymer sorbent with the yield of 85%.

Study of the Polymer Sorption Properties.

It has been studied sorption of ions Cu²⁺, Co²⁺, Cd²⁺, Zn²⁺, Ni²⁺ ion a synthesized complexing sorbent. For this purpose, were prepared solutions of these metals chlorides in water, with a concentration of 0.1 eq/l and a volume of 10 ml. Before sorption, 30 mg of sorbent was immersed in 10 ml of distilled water for 1 hour. Then the swollen ion exchanger was placed in a solution, in which the adsorbed ion was present, and stirred in a thermostated water bath for 4 hours so that the metal was sorbed. In this case, was observed a change in the color of the solution and the sorbent itself, because of metal ions transition to the adsorbed state. Next, the sorbent-metal complex was separated from the solution. As a result, a multi-colored swollen solid mass was obtained, which was then dried in an oven at room temperature.

To determine the adsorption capacity (Static exchange capacity) for ions, the concentration of metals in the solution was measured before adsorption (0.1 N in each case) and after adsorption.The concentrations of absorbed ions in post-adsorption solutions were determined from the difference in optical density, using the spectrophotometric method (for colored ions Cu^{2+} , Co^{2+} , Ni^{2+}) and using the complexometric method (for colorless ions $Cd^{2+}\pi$ Zn^{2+}). The spectrophotometric method of analysis was carried out using the device SP-IU-V7.The length of the cuvette in each dimension was 5 cm. In this case, the ion Cu^{2+} forms ammonia complexes with dilute ammonia solution (dark blue solution): wavelength for measuring optical densities is 610 nm. Ion Ni²⁺ forms complexes with a dilute ammonia solution, (blue-violet color of the solution), wavelength for measuring optical densities and columnation optical density 565 nm. When determining an ion Co^{2+} from a pink solution, a saturated yellow solution was formed, using murexide (measurement wavelength is 460 nm).

The concentration of Cd²⁺ and Zn²⁺ions was determined, using the complexometry method, as in the works [23] and [24]. In this case, were used solutions of dithizone with complexone III. The static exchange capacity (SEC) of the sorbent is determined by Russian State Standard (GOST) 20255.1-89 « Ionites. Methods for determining static exchange capacity», similar to work [3].

SEC calculated by the formula:

where C_0 -ion concentration before adsorption (in our case 0.1 N), C – ion concentration after adsorption, m – bulk mass of adsorbent.

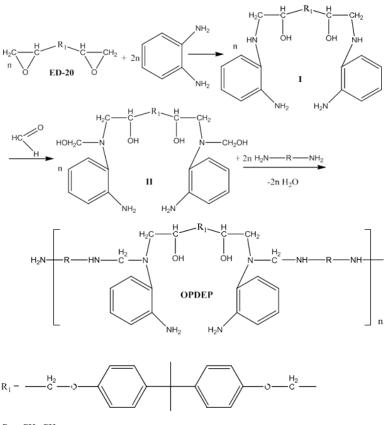
In accordance with GOST 20255.1-89, is taken the arithmetic mean value of the results of two parallel determinations, as the result of measuring the Static Exchange Capacity of the ion exchanger, if the acceptance condition is met:

 $|SEC_1 - SEC_2| < 0,025 \cdot (SEC_1 + SEC_2)/2,$

where SEC₁ and SEC₂ – results of parallel determinations of the static exchange capacity of the ion exchanger; 0,025 (2,5%) — repeatability limit value, %, with confidence probability P = 0.95.

Results and Discussion

The synthesis scheme for a polymer based on o-phenylenediamine, epoxy resin, formaldehyde and ethylenediamine is as follows:



 $R = -CH_2-CH_2-CH_2$

Figure 1: Scheme for the synthesis of a polymer sorbent, based on o-phenylenediamine

Based on the structure, presented in Figure 1, we decided to call the new polymer OPDEP, i.e. O-PhenylenDiamine – Epoxide – Polyamine.

In the first stage of the process, o-phenylenediamine reacts with the epoxy resin, opening both epoxy rings to form an intermediate product I.

The molecule of epoxy resin ED-20 (before combining with the hardener - diamine, i.e. liquid resin) is shown in a simplified form in Figure 1. This simple molecule is made up of one Bisphenol A fragment and two epoxy groups. The molecular weight of one epoxy group is 43 g/mol, two, respectively, is 86. The total mass of the molecule is 346 g/mol. Let's calculate the mass fraction of epoxy groups as a percentage: (43*2)/346* 100% = 25.4%. If the epoxy resin consists entirely of just such molecules, the proportion of epoxy groups in it is 25%. According to the Russian State Standard GOST 10587-84 «Epoxy-diane resins, uncured», it would be under the brand name ED-25, it would be very liquid (the smaller the molecule, the more mobile the liquid) and light. A resin molecule, composed of two Bisphenol A molecules and two epoxy groups, gives a mass fraction of the latter of already 14%. There is a grade ED-14 in GOST, it is a high-viscosity resin, at temperatures below +10 °C it will be a solid body, not a liquid. However, any epoxy resin is a mixture of

molecules of different lengths. For example, if we have 70% of molecules of the first type, with one Bisphenol A, and 30% of molecules of the second type, with a chain of two Bisphenols, then the mass fraction of epoxy groups will be 21.7%, and such resin will fall within the range, allowed by GOST (20.0-22.5 % for ED-20). However, to simplify understanding of the structure of the resulting polymer, we propose to write the formula of the epoxy resin exactly as in Figure 1, with only Bisphenol A in the composition.

At the second stage of the reaction, intermediate I interacts with formaldehyde. In this case, formaldehyde interacts with secondary amino groups - NH-, because they are more spatially accessible compared to primary amino groups, which can be directed into the intermediate product I. Besides that, -N-H- groups have higher nucleophilic properties due to the positive inductive effect of the neighboring CH₂ group. The presence of free amino groups was proven by us by analyzing the IR spectrum.

Intermediate II, obtained at the second stage, then enters into a polycondensation reaction with ethylenediamine, forming the final polymer.

To confirm the polymer structure we indicated, we carried out its IR spectroscopic analysis (Figure 2):

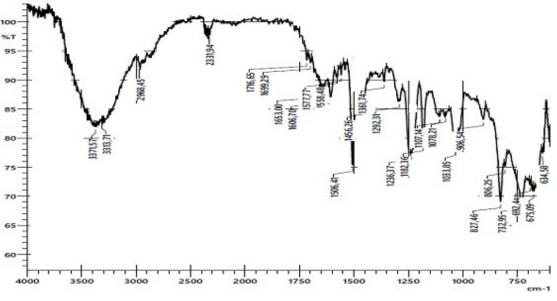


Figure 2: IR spectrum of the new polymer-sorbent OPDEP

The IR spectrum of the polymer shows that:

-stretching vibrations of OH groups are observed in a wide range with peaks 3371 cm⁻¹ and 3313 cm⁻¹;

-stretching vibrations of methylene (CH₂) group we can see at 2968 cm⁻¹;

-bending vibration of the secondary amino group is observed

(-NH-) Bat 1558 cm⁻¹ and bending vibration of the primary amino group (-NH₂) at 1456 cm⁻¹;

-bond stretching vibrations of C-N bond we can see at 1107 cm^{-1} and 1182 cm^{-1} , so these are different C-N bonds.

-vibrations of the C-C bonds of the aromatic ring are observed in the region of 1653 cm^{-1} ,

It will be interesting to compare the IR spectra of the polymer and its main component o-phenylenediamine (Figure 3):

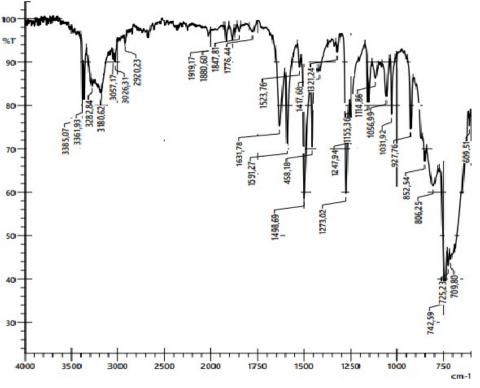


Figure 3: IR spectrum of o-phenylenediamine

In the IR spectrum of o-phenylenediamine we can see:

-asymmetric and symmetric stretching vibrations of $-NH_2$ group observed in a doublet state at 3385 cm⁻¹, 3361 cm⁻¹, 3282 cm⁻¹ and 3180 cm⁻¹. In Figure 2 they were not visible, because overlapped by the broad vibration frequency of the OH group;

-the absorption frequency, characteristic of the OH group itself, is not shown in Figure 3;

-stretching vibrations of methylene (CH₂) group now we can see at 2920 cm⁻¹;

-stretching vibration frequencies of the C-N bond are observed in the region 1273 cm^{-1} ;

-at the area of 3063-3057 cm⁻¹ in spectrum there are bands of C-H bonds stretching vibrations from aromatic ring, which were also not visible in Figure 2;

-stretching vibration of the C-C bonds of the aromatic ring can be observed in the range 1776-1919 cm⁻¹.

Here is a complete comparative table of the IR spectra of the original o-phenylenediamine and the polymer:

Subsnance	ν(OH)	ν(NH ₂)	$\delta(NH_2)$	δ(NH)	v(C-N)	ν(CH ₂)	Ar(C-C)
o-phenylenediamine	-	3180-3385	-	-	1273	-	1776-1919
polymer OPDEP	3313-3371	-	1456	1558	1107-1182	2968	1653

Table 1: IR spectrum analysis of o-phenylenediamine and polymer sorbent OPDEP

Next, we needed to study the thermal stability of the resulting polymer sorbent. This is a standard procedure when studying the properties of such compounds. [25]. For this purpose, the method of differential scanning calorimetry is used, which allows one to analyze the various exothermic and endothermic effects observed when the mass changes as a result of the destruction of the structure of the compound when heated (Figure 4):

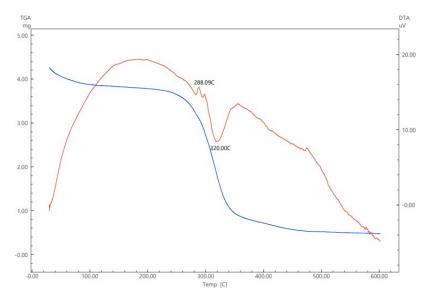


Figure 4: Thermogravimetric analysis curve of the OPDEP polymer (red line - energy absorption, blue line-change in mass upon heating)

At 226 °C the polymer begins to melt. This corresponds to the maximum energy absorption during heating. A small exothermic peak is observed at a temperature 288 °C. Its presence can be explained by reactions, occurring inside the polymer between hydroxyl, primary and secondary amino groups. The peak of the endothermic process, associated with the greatest energy absorption and maximum mass loss, was observed at 320 °C. In between 226 and 372 °C there is a loss of 80% of the polymer mass, that is, its gradual destruction. The results of thermogravimetric analysis are reflected in more detail in Table 2:

Process	Time (sec)	Temperature diapason	Loss of weight (mg)	Loss of weight (%)
1	12 c	30 °C - 226 °C	0,506	11,89 %
2	1232 c	226 °C - 372°C	2.934	80,19 %
3	2121 c	372 °C – 601 °C	0.337	7,92 %

Table 2: Results of thermogravimetric analysis of OPDEP polymer

Next, we studied the sorption properties of the resulting polymer. As a result of experiments, it was established, that the new sorbent has a high absorption capacity with respect to ions Ni(II), Co(II), Cu(II), Zn(II) и Cd(II).

The research results are presented in Table 3:

Sorbent	Mass of sorbent (<i>mg</i>)	Ion	Vlume of solution(<i>ml</i>)	Initial solution concentration(C _o)	Solution concentration after sorption(<i>C</i>)	Time of sorbtion (<i>h</i>)	SEC
OPDEP	30	Ni(II)	10	0.1	0.071	6	9.67
	30	Co(II)	10	0.1	0.075	6	8.33

30	Cu(II)	10	0.1	0.079	5	7.01
30	Zn(II)	10	0.1	0.080	5.5	6.67
30	Cd(II)	10	0.1	0.085	5	5.00

If we compare the data in Table 2 with the adsorption capacity of a polymer based on carbamide, formaldehyde and bromocresol purple [10] or polyampholyte based on carbamide, formaldehyde and aminoacetic acid [3], then we can conclude, that the sorption capacity of the new sorbent is 2-2.5 times higher than for already known ones.

The general scheme for the production of complex polymers based on the OPDEP sorbent is presented in Figure 5:

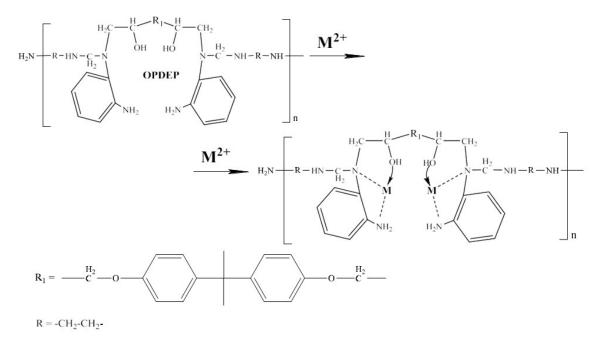


Figure 5: Formation of complexes between OPDEP polymer sorbent and M2+ cations ($M^{2+} = Ni^{2+}, Cu^{2+}, Co^{2+}, Zn^{2+} \varkappa Cd^{2+}$).

Next, we determined the dependence of the static sorption ca-

pacity on the pH of the solution in which the absorbed ion is located (Figure 6):

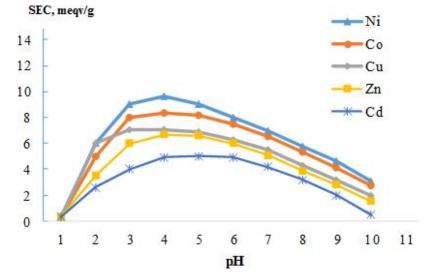


Figure 6: Dependence of the sorption process of the OPDEP sorbent on the pH value of the medium

As can be seen from the Figure, the sorption capacity of adsorbents has a maximum value in the pH range = 3-5. In a strongly acidic environment, the interaction of protons and the amino groups of the sorbent occurs. This reduces the possibility of the complex bonds formation for metals with the ion exchanger. As the pH value increases, increases the rate of exchange of protons, attached to the polymer and metal ions. A significant reduction in the sorption process can be observed even in an alkaline environment. This can be explained by a decrease in the number of ionic bonds in the polymer. The rate of exchange of ions in the solution and the transition of metal ions into the solution slows down, therefore the sorption capacity decreases.

To fully understand the efficiency of the sorbent, the dependence of the sorption process on time was studied. The sorption process lasted 5 hours for ions Ni (II) and Co(II), 6 hours for ions Cu(II) and Cd(II), 5,5 hours for ions Zn(II). First, the rate of ion sorption gradually increases, and then gradually slows down. At the end of the process, the sorption capacity remains unchanged and reaches a maximum value. Figure 7 shows the dependence of the sorption of metal ions on time at a concentration 0.1 N:

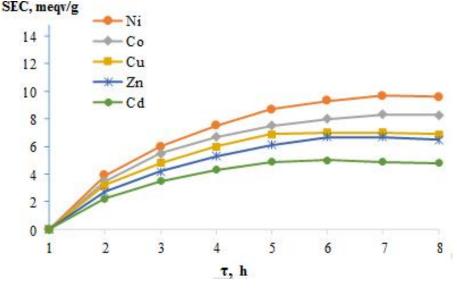


Figure 7: Dependence of the sorption capacity of the OPDEP sorbent on time in relation to Ni(II), Co(II) and Cu(II) ions

We also studied the process of desorption of ions under the influence of desorbing reagents. In this process, metal ions are replaced by hydrogen ions. and HCl (0.1 molar solutions), and also salt NH₄Cl. Changes in the concentration of solutions were monitored using a spectrophotometer SP-IUV7. The results of the study are presented in Table 4:

Sources of protons were used as desorbing reagents as HNO₃

Sorbent	Desorbing reagent	Concentration ofdesorbing reagent (M)	Percent of desorbtion (%))		
			Ni(II)	Co(II)	Cu(II)	Zn(II)	Cd(II)
OPDEP	HCl	0.1	86.7	69.3	81.3	73.5	69.3
	HNO	0.1	85.2	70.3	82.6	75.6	65.6
	NH ₄ Cl	0.1	23.6	18.3	22.5	17.6	11.7

Table 4: Desorption capabilities of the resulting polymer sorbent

From Table 4 it can be seen, that the desorption process is effective only in the presence of strong acids as effective sources

of protons. Thus, thanks to strong acids in small concentrations, regeneration of the sorbent is possible, as well as the release of previously absorbed ions from it. For example, an adsorbed ion Ni2+ released from the sorbent to 85-87%, and ion Cu^{2+} - to 81-83%.

Next, the synthesized OPDEP sorbent was analyzed using a scanning electron microscope..

Figure 8 shows an image of the sorbent surface at a magnification of 5000 times:

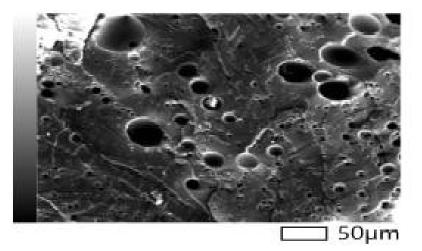


Figure 8: Scanning electron microscope image of OPDEP sorbent

The analysis results show that the substance has no additives before ion sorption and has a high degree of porosity. The elemental composition of the samples was analyzed during microscopic studies (Table 5):

Elements	Quantity, (%)	Confidence interval, (%)
С	60.43	0.70
0	11.51	0.67
Н	7.91	0.19
N	20.14	0.09

Table 5: Elemental analysis of OPDEP sorbent

The resulting polymer was also studied by X-ray phase analysis..

In the analysis of mathematical methods OriginLab software releases offered amorphous and crystalline кой структур

сорбента. In the process of filling Ni(II) ions with OPDEP sorbent, it is necessary to use in crystallization naturally. The results of the analysis, presented in Table 6, include the most common form of the work is amorphous:

Table 6: X-ray phase analysis of the OPDEP sorbent and its polymer complex formed by Ni(II) ions (OPDEP+Ni)

Nº	Samples	Total area of surface, nm	Crystalline phase, nm ²	Amorphous phase, nm ²	Degree ofcrystallization (%)
1	OPDEP	47635,5	5683,4	41952,1	11.93
2	OPDEP +Ni	45212,2	9856,8	35355,4	21,8

Conclusions

1.For the first time was synthesized a polymer sorbent, based on o- phenylenediamine, epoxy resin, formalin and ethylenediamine. Its physicochemical and sorption-desorption properties were studied.

2.Based on experiments, it was established that the new polymer sorbent has a high sorption capacity with respect to ions Cu^{2+} , Co^{2+} , Cd^{2+} , Zn^{2+} , Ni^{2+} .

3.It has been proven that the resulting polymer sorbent allows ions to be separated with high accuracy Cu^{2+} , Co^{2+} , Cd^{2+} , Zn^{2+} , Ni^{2+} from solutions.

4. The dependence of the sorption process on time and pH value of the medium has been studied.

5. The composition and properties of the polymer sorbent were studied using physicochemical methods, such as IR spectrum, SEM analysis, thermogravimetric analysis, X-ray phase analysis.

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