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Studying synthesis of a new polymer sorbent based on *o*-phenylenediamine and epichlorohydrin and its sorption properties

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This article is devoted to the synthesis and sorption properties of a new polymer sorbent (FD-EXG) based on *o*-phenylenediamine and epichlorohydrin, which separates metal ions from solutions with high precision. In the process of synthesis, the starting materials: *o*-phenylenediamine and epichlorohydrin are taken in a 1:2 mol ratio. Ethylenediamine is used as a polymerizing and binding agent. The composition, physicochemical and sorption-desorption properties of the obtained polymer sorbent have been studied. In the IR spectrum analysis of the sorbent, we can see the presence of n(OH) functional groups in the 3371 cm^{-1} and 3313 cm^{-1} and n(NH) ion exchange functional groups in the 1558 cm^{-1} region. Based on X-ray phase analysis methods, the degree of crystallization of the obtained sorbent and its compound with metal cation (Ni(II)) has been determined. Also, the morphology and thermal properties of the obtained sorbent have been studied using SEM analysis and TGA and DTA analysis methods. The surface area and the pore size of the polymer sorbent have been determined, and its morphology has been determined to be macroporous. It has been proven that the synthesized polymer sorbent can be reused many times during the sorption process. It is proved that the obtained polymer sorbent can efficiently remove Ni(II), Co(II), Cu(II), Zn(II) and Cd(II) ions from aqueous solutions. The static exchange capacity (SEC) of the obtained polymer sorbent has been determined to be SECNi=9.67, SECCo=8.33, SECCu=7.01, SECZn=6.67 and SECCd=5.01. It has been proven that the static exchange capacity is high in the range of *p*H 4-6. The time dependence of the sorption process and desorption processes have also been studied.

Keywords: *o*-Phenylenediamine, Epichlorohydrin, Polymer sorbent, X-ray phase analysis

Currently, along with the rapid development of production and industrial enterprises, man-made waste to nature is increasing year by year[1]. Nowadays, one of the most urgent problems in the world is the preservation and restoration of clean drinking water resources. One of the main sources of water pollution is industrial, communal and agricultural wastewater, oil and oil products. However, the most toxic wastewaters are waste waters from industries that use non-ferrous metals and valuable chemicals[2]. Water pollution with toxic substances such as heavy metals, aromatic compounds and chemical dyes is one of the important environmental problems[3]. An increase in the concentration of non-ferrous and heavy metals in industrial waters and wastewater causes toxicity to living organisms.

Today, there are various purification methods for removing heavy metal ions and chemical dyes from industrial and natural water, such as ion exchange, evaporation, electrodialysis, sorption, and reverse osmosis methods[4,5]. In recent years, polymer sorbents have been widely used in the extraction of precious metals from complex mixtures in the hydrometallurgical process and the extraction of metal ions from industrial wastewater[6]. The use of sorbents is widely used compared to other methods due to their high efficiency, low cost and ease of use[7]. In this research work, we used *o*-phenylenediamine and epichlorohydrin as the main reagents for obtaining sorbents. *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 protonation. Polymer sorbents based on *o*-phenylenediamine and starch have been proven to extract Ni(II) cations from aqueous solutions[8]. In addition, it was determined that polymer sorbents based on *o*-phenylenediamine have high selectivity for silver and mercury[9]. Epichlorohydrin has an epoxide group and a mobile chlorine atom in its composition, which increases its

possibilities of use. Epichlorohydrin is mainly obtained as a result of the reaction of glycerol and allyl chloride. The main part of epichlorohydrin is widely used in the preparation of epoxy resins, highly selective sorbents, moisture-resistant papers, surfactants and polymeric sorbents[10]. Sorbents formed by epichlorohydrin with aromatic amines are used to separate Cu(II) and Ni(II) ions from wastewater[11]. Epichlorohydrin and carboxymethylcellulose based sorbents have been proven to separate Cu(II) ions from complex mixtures[12]. Polymer sorbents formed by epichlorohydrin with diethyl-dithiophosphate[13], urea[14], amino acids[15], melamine[16] and other active donor atom trapping substances are widely used in industrial wastewater treatment and metal separation in the hydrometallurgical process[17]. Due to the presence of heterofunctional groups in some polymer sorbents, the number of bonds formed with metal ions increases. In addition, the high selectivity of the sorbent against metal ions depends on the porosity of ionite, the ability to form a complex with metal ions, resistance to sorption and desorption processes, and other physical and chemical properties[18,19]. In addition, sorption processes depend on the ionic radius, hydration process and valency of metal ions[20]. It is known that the hydration process decreases as the ionic radius increases. The less hydrated a substance is, the better it is absorbed[21]. In the process of sorption, the selective effect of the sorbent is important. For this, there should be no active centres that slow down the sorption process from the sorbent composition[22]. In sorption processes, the equilibrium between solution and ionite is quickly established[23]. The *p*H value of the environment affects the balance and through it, the optimal environment for the sorption process is selected[24]. When most sorbents are repeatedly used in the sorption-desorption process, their activity level decreases[25]. One of the important tasks facing the chemical industry today is synthesis of polymer sorbents with low cost, selectivity and high selectivity for metal ions[26].

Research object

o-Phenylenediamine, epichlorohydrin, ethylenedia mine, 0.1 N solutions of salts of Ni(II), Cu(II), Co(II), Zn(II) and Cd(II), weak solutions of acids and alkalis.

The purpose and task of the research

Synthesis of new polymer sorbents with high selectivity based on *o*-phenylenediamine and epichlorohydrin and high-precision extraction of non-ferrous metals from industrial wastewater using them. To achieve this goal, the following tasks were set:

(i). Synthesis of new polyfunctional ionites based on *o*-phenylenediamine and epichlorohydrin with high selectivity and study of their physicochemical properties.

(ii). Selection of optimal conditions for the sorption and desorption processes of the resulting polymer sorbent and high efficiency extraction of Ni(II), Co(II), Cu, Zn(II) and Cd(II) ions from industrial wastewater.

Experimental Section

Weak solutions of *o*-phenylenediamine, epichlorohydrin, 3d and 4d metal salts, ethylenediamine, ammonium chloride, alkali and acids were used in this research.

IR-analysis

The structure of this obtained pigment was determined by IR-spectroscopy (Fure spectrometer manufactured in Japan. IR spectroscopic studies were carried out in the powder method on a Shimadzu Fourier transform infrared spectrometer (range 4000-600 cm^{-1}, dimensions 4 cm^{-1}).

SEM analysis

The newly synthesized polymer sorbent was studied with a (MIRA 2 LMU) scanning electron microscope (SEM) equipped with an INCA Energy 350 energy dispersive microanalysis system.

UV-Vis Spectrophotometric analysis

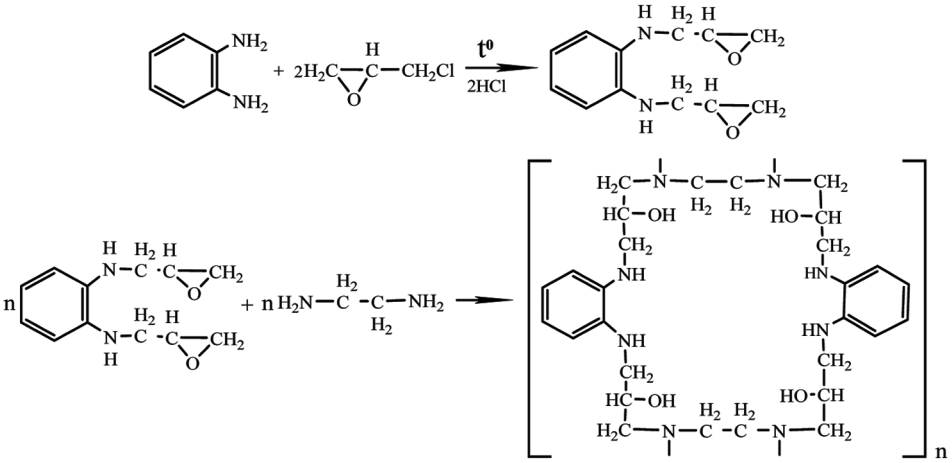
SP-IUV7 UV-Vis spectrophotometer was used to calculate the results of sorption and desorption processes.

TGA and DTA analysis

Shimadzu (simultaneous thermal analysis) is an easy-to-use, reliable, and high-performance thermal analysis platform for TGA simultaneous TGA-DTA, and TGA-DSC analysis methods. It is used to identify parts, measure the optical properties of samples, and also for various research purposes.

Synthesis of polymer sorbent

First, 15.7 mL (20 mmol) of epichlorohydrin and 10.8 g (10 mmol) of *o*-phenylenediamine was added to a flat-bottomed flask equipped with a magnetic stirrer and a reflux condenser and stirred at 30-35°C



Scheme 1 — Synthesis reaction of FD-EXG polymer sorbent

for 6 h, then at 80-85ºC for 6 hours. At the end of the process, the reaction mixture consists of two parts, the upper part is a clear liquid and the lower part is a pale yellow gel substance. These two liquids form the same phase when heated. The reaction mixture was stirred on a magnetic stirrer, reheated at 80º-90ºC for 2 hours, and made alkaline. Then, 2 mL of ethylene diamine was added to polymerize the reaction mixture, and the temperature was stirred at 80ºC for 1 hour. At the end of the reaction, after the temperature was cooled, a yellow granular polymer sorbent was obtained. The obtained sorbent was first dried and cleaned of impurities. Then it was neutralized using a 3% solution of NaOH washed several times in distilled water and dried in a thermostat at 45-50 ºC for 5 hours. At the end of the process, yellow polymer sorbent (FD-EXG) was obtained with 85% yield (Scheme 1).

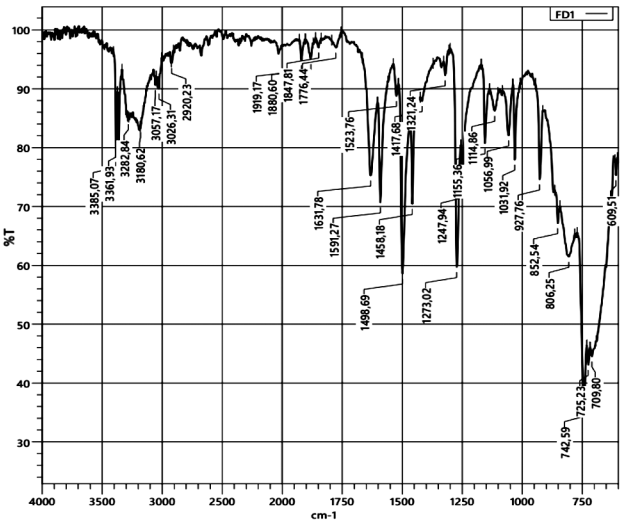


Fig. 1 — IR spectrum of *o*-phenylenediamine

IR-Analysis

Asymmetric and symmetric valence vibrations of NH_2-groups in *o*-phenylenediamine are in the doublet state at 3385 cm^{-1}, 3361 cm^{-1}, 3282 cm^{-1} and 3180 cm^{-1}, and the deformation vibrations of this group are at 1631 cm^{-1} and 1591 cm^{-1} observed. The frequencies of valence vibration of the C-N bond are observed in the region of 1273 cm^{-1}. In the 3063-3057 cm^{-1} region of the spectrum, there are bands caused by the valence vibrations of the C-H bonds of the aromatic ring. We can observe the valence vibration of the aromatic ring along the plane of the ring in the range of 1776-1919 cm^{-1} (Fig. 1).

According to the analysis of the results described in Fig. 2 below, the valence vibrations of OH groups can be seen in the broad areas of 3371 cm^{-1} and 3313 cm^{-1},

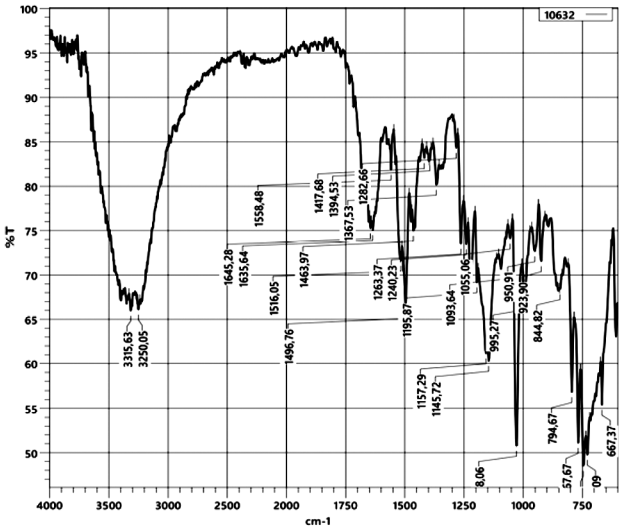


Fig. 2 — IR spectrum of the obtained FD-EXG polymer sorbent

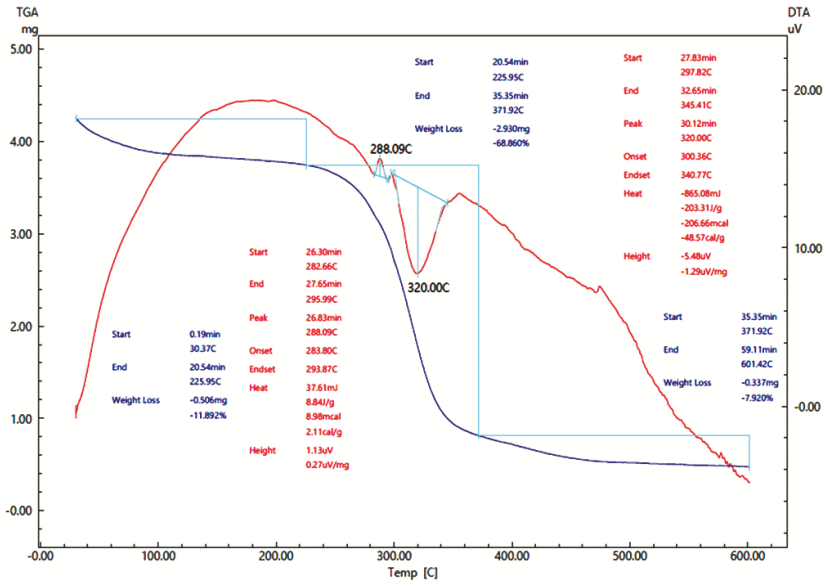


Fig. 3 — Derivatogram of obtained FD-EXG polymer sorbent

Table 1 — TGA analysis of the resulting FD-EXGionite

The steps of the reaction	Duration of the reaction process (s)	Temperature range (°C)	Loss of mass (%)
1-bosqich	880	26.33-163.7	14.53
2-bosqich	2007	163.7-348.83	68.14
3-bosqich	3550	348.83-601.5	17.34

and the valence vibrations of the methylene (CH_2) group in the area of 2968 cm^{-1}. Also, the deformation vibration of the secondary amino group (NH) was observed in the 1558 cm^{-1} area and the deformation of the primary amino group in the 1456 cm^{-1} area. The vibration of the aromatic ring along the ring plane was observed in the absorption bands of 1653 cm^{-1}, and the valence vibration of the C-N bond was observed in the 1107 cm^{-1} and 1182 cm^{-1} areas (Fig. 2).

TGA and DTA analysis of the polimer sorbent

In the DTA analysis of the obtained FD-EXG polymer sorbent, a high exothermic peak was observed at a temperature of 288.09°C. This is mainly due to the breakdown of weak hydroxyl (OH) and primary and secondary amino groups (NH, NH_2). The energy capacity for the exothermic process is 8.98 mcal (37.61 mJ). The peak of the endothermic process was observed at 320.00°C. In an endothermic process, saturation of covalent bonds is observed at the expense of atoms broken up in exothermic processes. The value of absorbed energy for this process is –206.66 mcal (–865.08 mJ) (Fig. 3).

Thermogravimetric analysis of the obtained polymer sorbent is carried out in 3 stages. The first stage takes place due to the decomposition of amino groups (NH_2) and hydroxyl groups (OH) in the polymer sorbent. In this case, the temperature is up to 163.7°C and the mass reduction is 14.53%. In the second stage, strong covalent bonds and *o*-phenylenediamine core in the sorbent are broken. At this stage, the temperature is 348.83°C and the mass reduction is 68.14%. In the third stage, the temperature increases to 601.5°C. At this stage, there are mainly organic residues, which make up 17.34% of the total mass (Table 1).

Results and Discussion

Sorption processes

As a result of the experiments, it was found that the sorbent has a high selectivity for Ni(II), Co(II) and Cu(II) ions. Solutions of metal salts (Ni(II), Co(II), Cu(II), Zn(II) and Cd(II)) with different concentrations of 0.05 N, 0.075 N and 0.1 N were used (Table 2). 0.1 N HCl and 0.1 N NaOH solutions were prepared to control the *p*H value of the medium. 10 mL of Ni(II), Co(II), Cu(II), Zn(II) and Cd(II) salt solutions were poured into 5 vials, and 30 mg of sorbent (EXG-FD) was placed on top of them, and the container was filled and closed. The sorption process

Table 2 — Static exchange capacities of the obtained polymer sorbent against Ni(II), Co(II), Cu(II), Zn(II) and Cd(II) salts (*p*H=4-6)

Ionite obtained for sorption	Mass of ionite obtained for sorption (mg)	Salts obtained for sorption	Solution volume (mL)	Initial concentration of the solution (C_1)	Concentration of solution after sorption (C_2)	Sorption time (h)	SAS
EPX-FD	30	Ni(II)	10	0.1	0.071	5	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	6	6.67
	30	Cd(II)	10	0.1	0.085	6	5

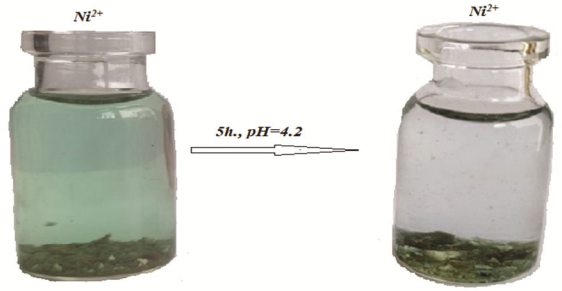


Fig. 4 — Sorption process of FD-EXG polymer sorbent in Ni(II) salt solution

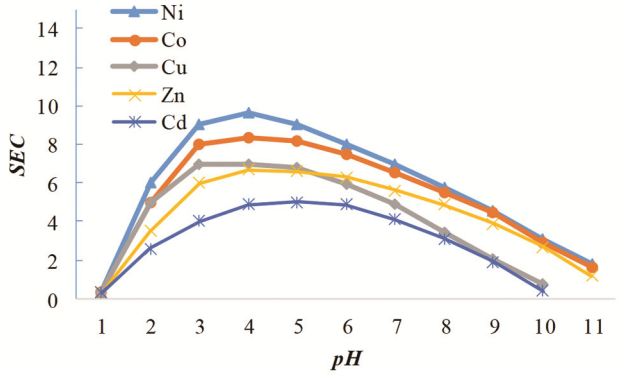


Fig. 5 — Dependence of the sorption process of EXG-FD ionite on the *p*H value of the environment

was monitored for an average time interval of 6 hours, and the optimal conditions of the *p*H environment were selected. The sorption process of FD-EXG polymer sorbent in Ni(II) salt solution is shown in Fig. 4. The change in concentration during the sorption process was determined by SP-IUV7-spectrophotometer, and the sorption capacity was calculated by the following equation (1):

$$SAS = \frac{V(C_1 - C_2)}{m} \quad \ldots (1)$$

Here, V is the volume of the solution obtained for sorption, C_1 is the initial concentration of the solution, C_2 is the concentration of the solution after sorption, and m is the mass of the obtained sorbent.

Effect of environmental *p*H on the sorption process

The influence of *p*H in an aqueous solution is one of the main parameters. In this study, the effect of FD-EXG sorbent on metal ions was carried out at different *p*H values, and there was no phenomenon of deposition of metal ions in any range of *p*H value. In the sorption process, the static exchange capacity (SEC) achieves high efficiency in the range of *p*H 4-6. In an acidic environment, a strong interaction occurs between protons and metal ions in the solution. This reduces the possibility of formation of complex bonds of metals with the sorbent. As the acidic medium decreases, *i.e.*, as the *p*H value increases, the rate of exchange of protons and metal ions increases (*p*H 7). We can observe a significant decrease in the sorption process in alkaline conditions. Because, based on the results of potentiometric titration, it can be explained by the decrease of ionic bonds and the increase of polar bonds in the composition of the solution. As the bonds weaken and the transition of metal ions into the solution increases, the sorption capacity decreases, and it was found that the sorption capacity decreases sharply when the *p*H changes to an alkaline environment (Fig. 5).

Time dependence of the sorption process

The sorption process of EXG-FD ionite lasted 5 hours for Cu(II) and Ni(II) ions, and 6 hours for Co(II) ions. At low concentrations of the solution, the rate of sorption increases and slows down as the concentration increases. At the end of the process, even if the concentration increases, the sorption capacity remains unchanged. Fig. 6 shows the processes of solutions of metal salts at a concentration of 0.1 N.

Desorption process

The desorption process is the process of metal ions absorbed into the ionite during the sorption process and returning to the solution. In this process, metal ions are replaced by hydrogen ions. We used 0.1

Table 3 — Desorption capabilities of the resulting polymer sorbent

Polymer sorbent	Desorption reagent	Desorption reagent concentration (M)	Desorption process (%)				
			Ni(II)	Cu(II)	Co(II)	Zn(II)	Cd(II)
EXG-FD	HCl	0.1	86.7	69.3	81.3	73.2	61.5
	HNO_3	0.1	85.2	70.3	82.6	65.3	55.9
	NH_4Cl	0.1	23.6	18.3	22.5	27.8	31.6

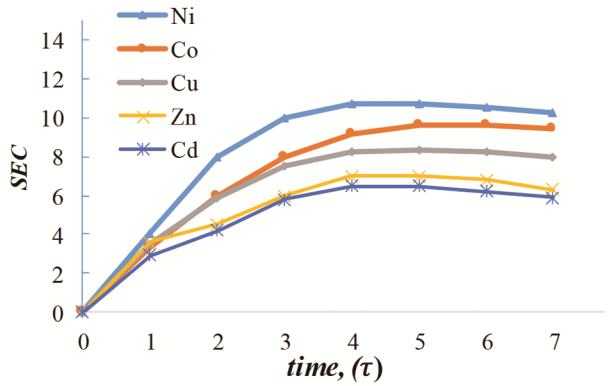


Fig. 6 — Time dependence of the sorption capacity of EXG-FD sorbent for Ni(II), Co(II), Cu(II), Zn(II) and Cd(II) ions

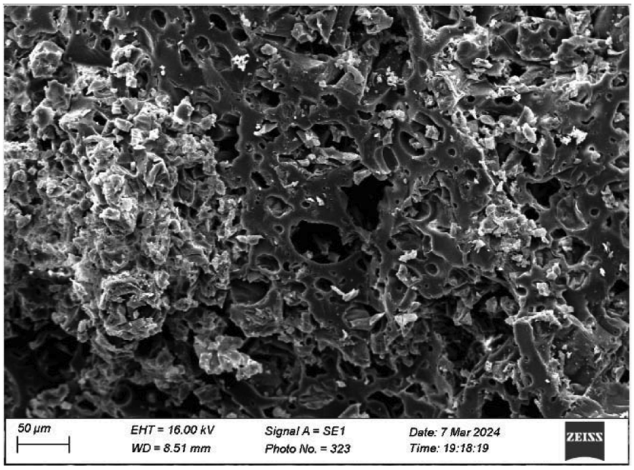


Fig. 7 — Scanning electron microscope image of FD-EXG polymer sorbent

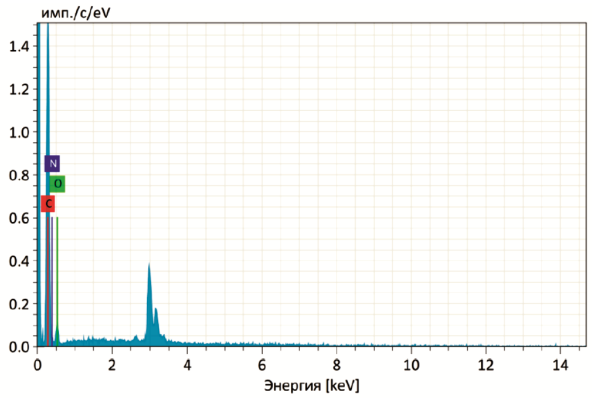


Fig. 8 — Elemental analysis of FD-EXG polymer sorbent

Table 4 — Elemental analysis of FD-EXG polymer sorbent

Element	Amount (%)	Sigma percentage (%)
C	67.2	0.70
O	12.8	0.57
H	8.8	0.09
N	11.2	0.19

molar solutions of NH_4Cl, HNO_3 and HCl acids in the desorption process and monitored the change in solution concentrations using the SP-IUV7 spectrophotometer (Table 3).

SEM analysis

The synthesized FD-EXG polymer sorbent was analyzed by MIRA 2 LMU brand scanning electron microscope (Fig. 5). SEM analysis was performed under a high vacuum. Microanalysis of the elements of the sorbent was carried out in this device, and it was studied in fields with an accelerating voltage of 20 keV and a current of 1 nA. According to the results of the analysis, we can see that the composition of ionite is uniformly distributed. The composition of the sorbent is shown at a magnification of 5000 times, and the degree of reaction and porosity layers are described. Based on the results of SEM analysis, the compositional proportions of substances were calculated (Table 4). The results of the analysis show the absence of additives in the substance and the high level of porosity (Fig. 7). Elemental analysis of FD-EXG polymer sorbent is shown in Fig. 8.

Pore volume and surface area were analysed in an Autosorb iQ Station 2 device under a nitrogen atmosphere. In this case, the absorption temperature is 77.35 K and the exit temperature is 120°C. Based on it, the surface area of the obtained polymer sorbent is determined by the volume of nitrogen absorbed into the pores of the sorbent. The mass of the obtained sample was 0.1768 g and the diameter of the macropores was 300 nm (Ref. 27). The analysis shows that this polymer sorbent is macroporous (Fig. 9).

X-ray phase analysis

The X-ray phase properties of the resulting polymer sorbent were performed on a Panalitik

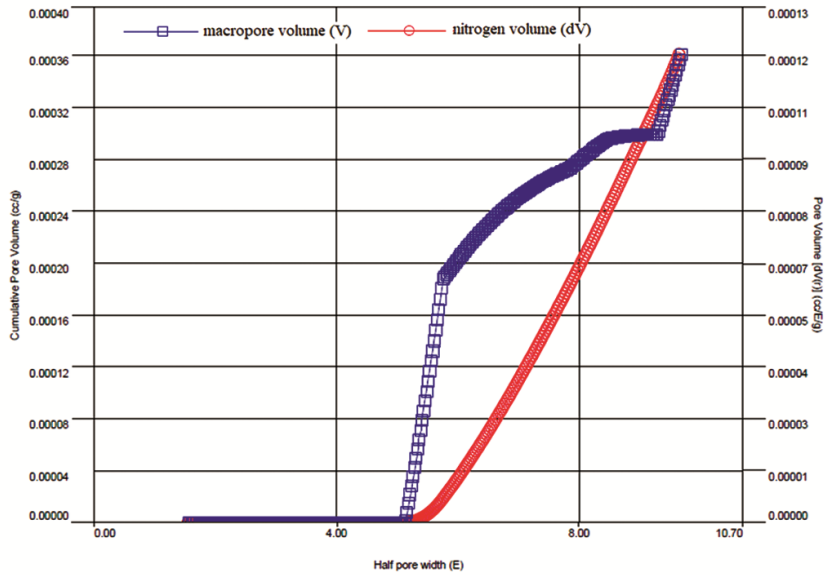


Fig. 9 — Surface area and pore volume of EXG-FD polymer sorbent

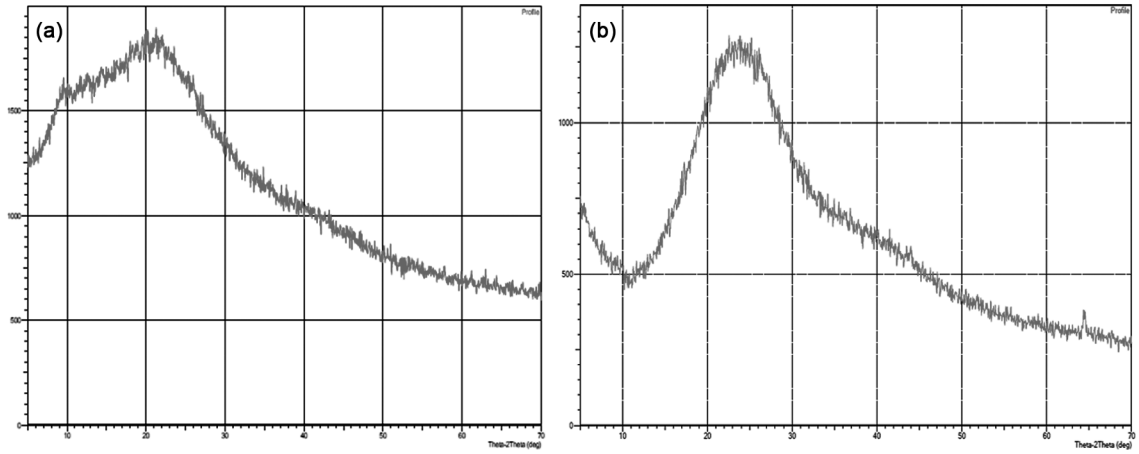


Fig. 10 — X-ray diffractograms of FD-EXG polymer sorbent (a) and its polymer complex with Ni(II) ions (b) (FD-EXG+Ni)

Table 5 — X-ray phase analysis results of the polymer complex formed by EXG-FD polymer sorbent with Ni(II) ions (FD-EXG+Ni)

S. No.	Sample received	General area	Crystal phase	Amorphous phase	Degree of crystallization (%)
1	EXG-FD	47635.5	5683.4	41952.1	11.93
2	EXG-FD+Ni	45212.2	9856.8	35355.4	21.8

Empyrean diffractometer, and the diffractograms were analyzed using OriginLab and Profex computer programs (Fig. 10).

Based on the obtained data, the amounts of amorphous and crystal structures of ionite were found using mathematical analysis using OriginLab software. The process of identification of components, the presence of peaks related to sulphogroup, organic ring compounds, and hydroxyl substances, along with various derivatives of epichlorohydrin, was determined. This proves the presence of organic modification in the sample. In the absorption process of Ni(II) ions of EXG-FD ionite, we can observe that the level of crystallization is relatively high. Based on the analysis results, we can observe that the amorphous phase is the main part of the samples (Table 5).

Based on the results of physicochemical analysis, coordination of FD-EXG polymer sorbent with metal ions (Ni(II), Cu(II), Co(II), Zn(II), Cd(II)) was proposed as shown in Fig. 11.

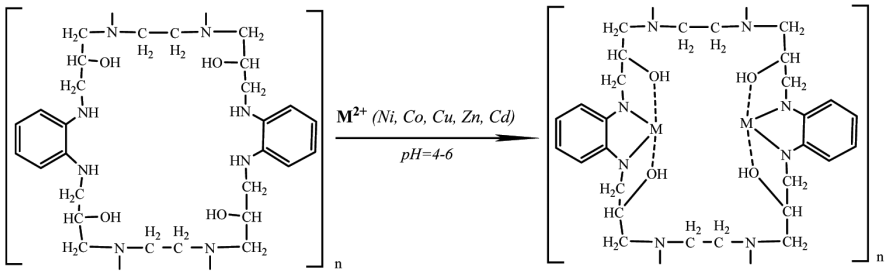


Fig. 11 — Coordination of FD-EXG polymer sorbent with metal ions

Conclusion

(i) In this research work, a new polymeric sorbent based on *o*-phenylenediamine and epichlorohydrin was synthesized. Synthesis conditions, morphology and physico-chemical properties of the obtained polymer sorbent were studied.

(ii) Pore volume and surface area analysis was performed on an Autosorb iQ Series 2 device. Based on the analysis results, it was proved that the morphology of EXG-FD polymer sorbent has a macroporous structure.

(iii) The optimal medium for the sorption process of the synthesized polymer sorbent was selected. EXG-FD sorbent has been proven to have high selectivity for Ni(II), Co(II), Cu(II), Zn(II) and Cd(II) ions and to remove these ions in aqueous solutions with high efficiency. It was found that EXG-FD sorbent has a high regeneration ability in sorption-desorption processes.

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Authors' Declaration

Conflicts of Interest: None.

We hereby confirm that all the Figures and Tables in the manuscript are ours.

Ethical Clearance: The project was approved by the local ethical committee in Termez State University.

Authors' Contribution Statement

Ahatov A. A: Writing – Original Draft. Ahatov A.A: Reviewing and editing paper. Turaev Kh. Kh: Reviewing and editing paper. Tillaev X.R. Software, Validation. Kasimov Sh.A and Nomozov A.K: Writing – Original Draft, Conceptualization, Investigation, Visualisation.

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