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*Original Article*

# Research on the Synthesis of Nitrogen-Containing Sorbents

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***Abstract*** *- In this paper, research was conducted on synthesizing nitrogen-containing complex-forming sorbent based on urea, formaldehyde and 1-naphthylamine. In this synthesis, the mole ratio of the starting materials was found to be 2:5:0,2 (carbamide, formaldehyde and 1-naphthylamine). It was also determined that the temperature of the synthesis process was 90 °C and the time duration was 1.5-2 hours. According to the obtained results, the static exchange capacity of the synthesized sorbent was 3.8 mg-eq/g for 0.1 N NaOH solution. The surface morphology of the synthesized complex-forming sorbent was studied using a scanning electron microscope (SEM) and IR spectroscopic analysis of the structure of the sorbent. The sorption of Ni (II), Zn (II), Cu (II), Co (II) and Cd (II) ions in solutions was studied using a complex-forming sorbent.*

***Keywords*** *- Urea, Formaldehyde, 1-naphthylamine, Sorbent, Scanning electron microscope, IR.*

## 1. Introduction

Nowadays, the world is focused on creating low-waste or no-waste technologies from secondary raw materials and finding economical, ecologically clean ways to use natural resources rationally. Such sorbents are widely used in the metallurgical industry: for the separation of non-ferrous metal ions in technology and wastewater, as well as for the separation of mixtures of metal ions.

At the current time, a large range of ionizable, complex polymers and polymer matrices has been developed [1,2]. These ion exchange resins are widely used for the sorption of heavy metals and harmful components in hydrometallurgy [3,4]. Today, promising chemical industry directions and methods of synthesizing new ion-exchange resins by modifying cheap, local raw materials are widely used [5].

In the process of sorption, it was observed that the amount of non-ferrous metal ions in aqueous solutions is effective even at the lowest concentration [6,7]. Currently, effective and economical methods of extracting precious and non-ferrous metals using sorbents containing nitrogen and sulfur are being introduced. The sorption capacity of such sorbents depends on the composition of functional groups in them, the nature of the sorbed ion, and the sorption conditions [8].

NN. Under the leadership of Basargin, together with the scientists of the Institute of Environmental Safety (Kursk), acid-base and complex formation properties of chelated polymer sorbents [9]; The synthesis and physicochemical properties of chelating sorbents with functional groups of N-aryl-3-aminopropionic acids were unsuccessfully studied by L. K. Neduchina, scientists of the Ural Federal University [10].

In the following study, the influence of various factors on the rate of modification of polysilicic acid [8], absorption of distilled water and saline solutions of different concentrations into hydrogels based on starch copolymer was studied [11]. The synthesis of a chelating sorbent based on urea, formaldehyde, and 2,4dinitrophenylhydrazine has been studied[12]. The effect of the temperature and molar ratio of the starting materials on the properties of the resulting chelate sorbent was observed, and a new polymer sorbent structure with a spatial structure was studied [13].

This study recorded and compared the IR spectra of polyampholyte complexes with copper, nickel, zinc, cobalt, and cadmium ions with the spectra of the polyampholyte itself and ligands. In the course of studies, it was observed that the synthesized sorbent contains intra- and intermolecular hydrogen bonds of hydroxyl groups, methylene -$CH_2$-, carbonyl groups -C=O, and vibrations characteristic [14].

A new chelating fiber (PET-TSX) sorbent consisting of polyethylene and terephthalate was synthesized for the sorption of $Hg^{2+}$, $Cu^{2+}$, and $Co^{2+}$ ions from solutions [15]. Based on nitrogen-containing reactive compounds, new complex-forming sorbents were obtained [16]. In the article, a new type of sorbent based on thiourea and sodium metasilicate is synthesized, which has selective sorption properties due to the presence of the thio group in its

molecule and the presence of silicon. During the sorbent synthesis, 60 g of thiourea and 100 g of sodium metasilicate were taken in solution and heated in a water bath at a temperature of 90 °C. Based on this, the results of the thermal analysis of the sorbent and SEM analysis of the sorbent were presented [17,18].

Sorbents impregnated with various dithiophosphoric acids (diethyl-, diisopropyl-, diisobutyl-) and coordination formed by sorption of Ag (I), Cu (II), Ni (II), Fe (III) ions from 0.05 M solutions using them IR spectra of compounds were studied [19].

Chelating sorbents were synthesized based on the covalent attachment of dithizone [20] to the matrix of urea-formaldehyde resin. Article [21] studied the ligand covalently fixed in place with O, O-di-(2-aminoethyl)-dithiophosphate potassium in a polyester matrix complexed with d-metal cations. A quantitative description of $Cu^{2+}$ ion sorption laws with fiber complexity containing amino hydroxyl was given [22], and the main kinetic and concentration characteristics of the processes, as well as the coordination properties of the complexities formed in the sorbent phase, were determined.

In this paper [23], 2,21-dihydroxy biphenyl was synthesized by condensation of urea and formaldehyde in the presence of 2 M HCl catalyst, terpolymer resins of p-nitrophenol, triethylenetetramine, and formaldehyde in the presence of 2 M NaOH catalyst. The sorption properties of this polymer are $Fe^{3+}$, $Cu^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Zn^{2+}$, $Cd^{2+}$, and $Pb^{2+}$ studied for ions. The research was conducted in environments with different pH. The ion exchange polymer showed higher selectivity for $Fe^{3+}$, $Cu^{2+}$, and $Ni^{2+}$ ions than for $Co^{2+}$, $Zn^{2+}$, $Cd^{2+}$ and $Pb^{2+}$ ions [22,23].

In the course of the research, a new adsorbent (A-PGMA) was synthesized by modifying poly(glycidyl methacrylate) microspheres with 2-aminothiazole. Good selectivity for ions such as Zn (II), Mg (II), Cu (II), Ge (IV), and B (III) was observed during the sorption of Au (I) ions from solutions. The results showed that the maximum adsorption capacity of Au (I) ions is 440.54 mg/g, and the equilibrium adsorption time is 3 hours when the pH of the soluble medium is 4 [24].

In the article, metal complexes were obtained from the interaction of Cu (II) chloride with random copolymers of 2-acrylamido-2-methylpropane sulfonic acid, AMPS, and the sodium salt of dodecyl methacrylate, i-DMA [24]. The sorbent obtained based on melamine-urea-formaldehyde has the property of sorption of metal ions Cu (II), Co (II), Cd (II), Zn (II), Cr (III), Hg (II), and Cr (VI) in the aqueous phase. [25,26], the sorption kinetics of Ni (II) and Zn (II) ions by polysiloxane with 2-aminoethyl pyridine functional groups were studied in different pH media [27-29]. During the literature analysis, it was found that the structure of coordination compounds formed by complex-forming polymer ionites with various metal ions has not been sufficiently studied.

Therefore, the synthesis of a new sorbent based on urea, formaldehyde, and 1-naphthylamine, the sorption process of the sorbent with Ni (II), Zn (II), Cu (II), and Co (II) ions was determined by IR-spectral method.

## 2. Materials and Methods

### *2.1. Chemicals*

The research work aims to study the sorbent based on urea, formaldehyde, and 1-naphthylamine (KFNA) and its formation of complexes with non-ferrous metals. "Pure" and "chemically clean" reagents were used in the work. Urea, formaldehyde, 1-naphthylamine solution in alcohol, buffer solution ($NH_4Cl+NH_4OH$), 0.02 n solutions of $NiCl_2$, $ZnCl_2$, $CuSO_4$ *$5H_2O$ and $CoSO_4$. Reagent solutions were prepared by dissolving a specific sample in a known volume of solution. The specific volume of the swollen sorbent was determined according to GOST 10898.4-84, and the static exchange capacity (SAS) according to GOST 20255.1-89.

### *2.2. Methods*

*2.2.1. Scanning Electron Microscope (SEM)*

Studies were conducted to study the surface morphological structure of the obtained sorbent and its sorption capacity with some d-metals. They were identified by SEM EVOMA 10 scanning electron microscope (Carl Zeiss). This allows for determining the elemental composition of ion exchange phases with an accuracy of 0.5%. Images were taken at different sizes using SmartSEM software. The image taken using an electron microscope was taken at the Advanced Technologies Center under the Ministry of Innovative Development of the Republic of Uzbekistan.

*2.2.2. IR Spectroscopy*

IRTracer-100 SHIMADZU infrared Fourier IR spectrometer (Japan) (range 400–4000 $cm^{-1}$, resolution 4 $cm^{-1}$) was determined using the powder method; the images were taken at the Tashkent Scientific Research Institute of Chemical Technology.

## 3. Experimental Part

### *3.1. Obtaining a Sorbent based on Urea Formaldehyde and 1-Naphthylamine.*

For the synthesis of a complex-forming sorbent, 12 g (0.2 mol) of urea was placed in a three-necked flask equipped with a reflux condenser and an automatic stirrer, and 39.5 ml (0.5 mol) of formaldehyde was added and heated at 40° It was carried out at a temperature of C. In order to carry out the polycondensation process in an alkaline medium, 5 ml of buffer solution ($NH_4Cl+NH_4OH$) was added to the solution, and the medium was brought to pH = 9-10. Then, 2.86 g (0.02 mol) of a solution of 1-naphthylamine in alcohol was added dropwise with vigorous stirring. Then, the temperature was heated to 85-95 °C and intensively mixed. The mixture formed a tarry mass in 1-1.5 hours. The synthesized sorbent was dried and reduced to a small mass. Then it was washed with 5% NaOH solution and distilled water until it became neutral. As a result, a red-yellow solid porous sorbent was obtained. The yield of the reaction is 85%. The molecular formula of 1-naphthylamine is $C_{10}H_7NH_2$.

In order to study the effect of changing the mole ratio of the initial substances in the synthesis of the sorbent on the sorption properties of some d-metals: In the above experimental method, urea and formaldehyde were taken in a 2:5 mole ratio, and 0.1, 0.2 and 0.3 of naphthylamine complex-forming sorbents were synthesized based on mole ratios.

### *3.2. Preparation of $Ni^{2+}$, $Zn^{2+}$, $Cu^{2+}$ and $Co^{2+}$ Solutions Forming Complex*

The sorption process of the complex-forming sorbent was studied in solutions of some d-elements of the III periods ($Ni^{2+}$, $Zn^{2+}$, $Cu^{2+}$, and $Co^{2+}$). For this, 0.02 N solutions of these metal salts were prepared. Then urea, formaldehyde 2:5 mol ratio, 1-naphthylamine 0.1:0.2:0.3 were added to $Ni^{2+}$, $Zn^{2+}$, $Cu^{2+}$, and $Co^{2+}$ solutions obtained from 10 ml. 0.015 g of the granules of sorbents obtained in the ratio of mol were placed for absorption for 24 hours. During the study, a change in the color of the sorbent in the solution was observed, indicating the metal ions' adsorption. The dried sorbent was separated from the solution and dried in a drying oven at room temperature.

Changes in the initial and post-sorption concentrations of metal ions during the sorption process were measured using a spectrophotometer, and the sorption capacity was calculated using the following equation.

$$CAC = \frac{(C1 - C2) * V}{m}$$

where

$C_1$ - the initial concentration of the solution in mol/l;

$S_2$ is the concentration of the solution after sorption, mol/l;

V – the volume of solution taken for sorption, ml;

m - mass of dry sorbent, g.

## 4. Results and Discussion

### *4.1. Effect on Polycondensation Temperature*

According to the results of the conducted studies, the effect of temperature on the polycondensation process of KFNA was observed, and the polycondensation process was studied at 343, 353, 363, and 373 K. The duration of the reaction, the specific volume of the sorbent in water, and the static exchange capacity of 0.1 n NaOH solution were determined. The obtained results are presented in Table 1.

From the data in Table 1, it can be seen that the duration of the polycondensation reaction at a temperature of 343 K was 3-4 hours, and the exchange capacity of the sorbent was 2.5 mg-eq/g. As the temperature increases, the polycondensation process accelerates, the structure of the obtained sorbent becomes denser, and the mobility of ionogenic groups becomes difficult.

363 K was chosen as the optimal temperature for the polycondensation reaction, with a duration of 1.5-2 hours. The course of the polycondensation reaction was somewhat balanced, and the static exchange capacity of the obtained ionite in 0.1 N NaOH solution reached 3.8 mg-eq/g.

### *4.2. Sorption of Sorbent with Heavy Metals*

The dependence of the main sorption and physicochemical properties of the synthesized complex-forming sorbent on its concentration was studied in the experiment. The polycondensation reaction was carried out in the molar ratio of reactants: urea, formaldehyde, and 1-naphthylamine, from 2:5:0.1 to 2:5:0.3, respectively. Changes in the amount of 1-naphthylamine and the degree of sorption by $Cu^{2+}$, $Zn^{2+}$, $Co^{2+}$, and $Ni^{2+}$ ions were determined (Table 2).

**Table 1. Effect of polycondensation temperature on the properties of sorbents**

| № | Reaction temperature, K | Duration of the reaction, t, hours | Specific volume of H-shaped sorbent dissolved in water, ml/g | SAS by 0.1 N NaOH solution, mg-eq/g |
|---|---|---|---|---|
| 1. | 343 | 3-4 | 1,78 | 2,5 |
| 2. | 353 | 2,5-3 | 1,56 | 3 |
| 3. | 363 | 1,5-2 | 1,37 | 3,8 |
| 4. | 373 | 1-1,5 | 1,31 | 3,4 |

**Table 2. Dependence of the sorption properties of KFNA on the ratio of reactants**

| Naming of indicators | | In the ratio of moles: urea, formaldehyde, 1-naphthylamine | | |
|---|---|---|---|---|
| | | 2:5:0,1 | 2:5:0,2 | 2:5:0,3 |
| Powder weight, g/ml | | 0,72 | 0,58 | 0,5 |
| Static exchange capacity for 0.02 N solutions, mg-eq/g: | $Cu^{2+}$ | 2,1 | 3,5 | 2,7 |
| | $Zn^{2+}$ | 2,7 | 3,3 | 2,3 |
| | $Co^{2+}$ | 1,33 | 2,6 | 2 |
| | $Ni^{2+}$ | 1,9 | 2,6 | 2,2 |

From the data in the table, it was observed that the ion exchange capacity of the sorbent gradually decreases with the increase in the amount of 1-naphthylamine. When KFNA is in the amount of 2:5:0.3 mol, the functional groups in the sorbent are brought closer to each other, which leads to the reduction of the pore radius. As a result, the sorption of sorbent with metal ions becomes difficult. According to the research results, the complex sorbent with the best performance is the 2: 5: 0.2 mol ratio of urea, formaldehyde, and 1-naphthylamine, respectively.

### *4.3. Environmental Influence on the Sorption Process*

As a continuation of the experiment, the dependence of the sorption properties of the complex-forming sorbent on Cu (II) ion on the solution environment was studied. The obtained results were depicted graphically (Fig. 1).

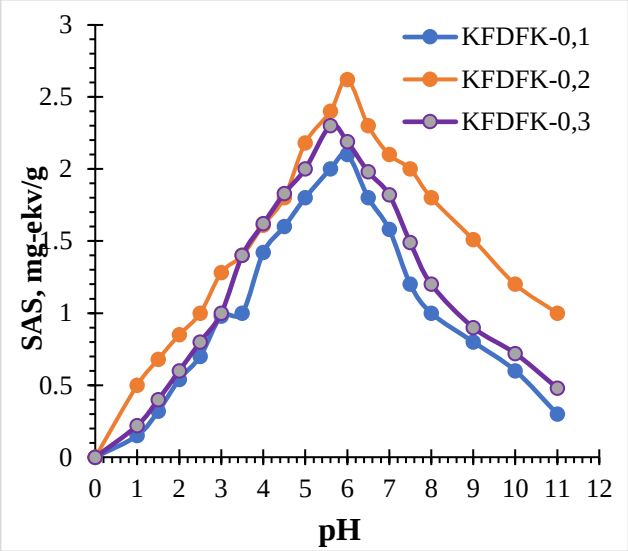



**Fig. 1 Dependence of the sorption of Cu (II) on UFNA with different molar ratios on the pH of the environment**

In the graph in the figure, it was observed that the sorption of Cu (II) ions on complex-forming sorbents is high in a weakly acidic environment. It was found that the level of sorption decreases when the environment changes from neutral to alkaline. We can conclude from this that protonated active functional groups of the sorbent can form complex compounds with the Cu (II) ion in a weakly acidic environment.

### *4.4. Scanning Electron Microscopic Analysis of Complexing KFNA Sorbent*

The microscopic structure of the complex-forming sorbent based on KFNA (2:5:0.2) was studied using a scanning electron microscope (SEM). In the 10 μm photo, it was observed that the complex-forming ionite has a microporous structure. This shows that the sorbent has a very high sorption capacity. The following figure shows the results of scanning electron microscopy of KFNA sorbent (Figure 2).

### *4.5. IR-Spectroscopic Analysis of KFNA Sorbent*

IR spectra were used to study the composition, structure and functional groups of the synthesized sorbent.

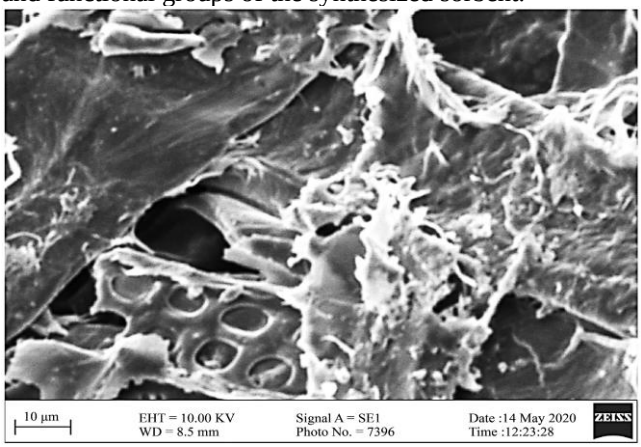



**Fig. 2 Scanning electron microscope image of KFNA sorbent surface**

The IR spectra of the synthesized KFNA sorbent are as follows: vibrations corresponding to the bound OH group were observed in the region of 3354 cm-1. The bands in the region of 2954 cm-1 correspond to methylene-$CH_2$, and the vibrations corresponding to the primary amide group in the region of 1625 $cm^{-1}$ were observed. Vibrations associated with the R-O-H group appeared in the region of 1274 $cm^{-1}$, and in the regions of 898-659 cm-1, an out-of-plane vibration of the n (NH) group appeared (Fig. 3).

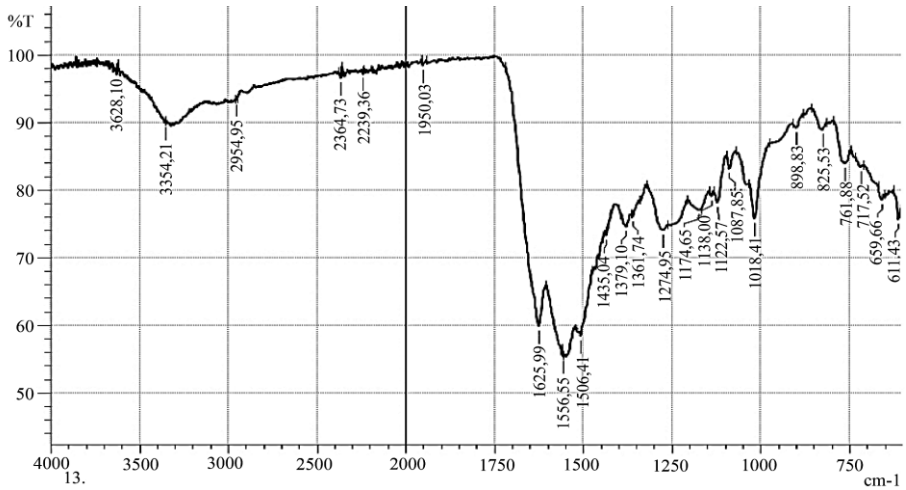



**Fig. 3 IR spectrum of the UFNA sorbent**

**Table 3. KFNA sorbent and the shift in the IR spectrum of the compound formed by the sorption of Ni (II), Zn (II), and Cu (II) ions in KFNA**

| Compound | $\delta(CH_2)+\delta(CN)$ $sm^{-1}$ | Δν, $sm^{-1}$ | ν(C-O), $sm^{-1}$ | Δν, $sm^{-1}$ | ν(C-N) $sm^{-1}$ | Δν, $sm^{-1}$ |
|---|---|---|---|---|---|---|
| KFNA | 1615 | - | 1138 | - | 1435 | - |
| KFNA + Ni (II) | 1626 | -10 | 1198 | -60 | 1417 | 18 |

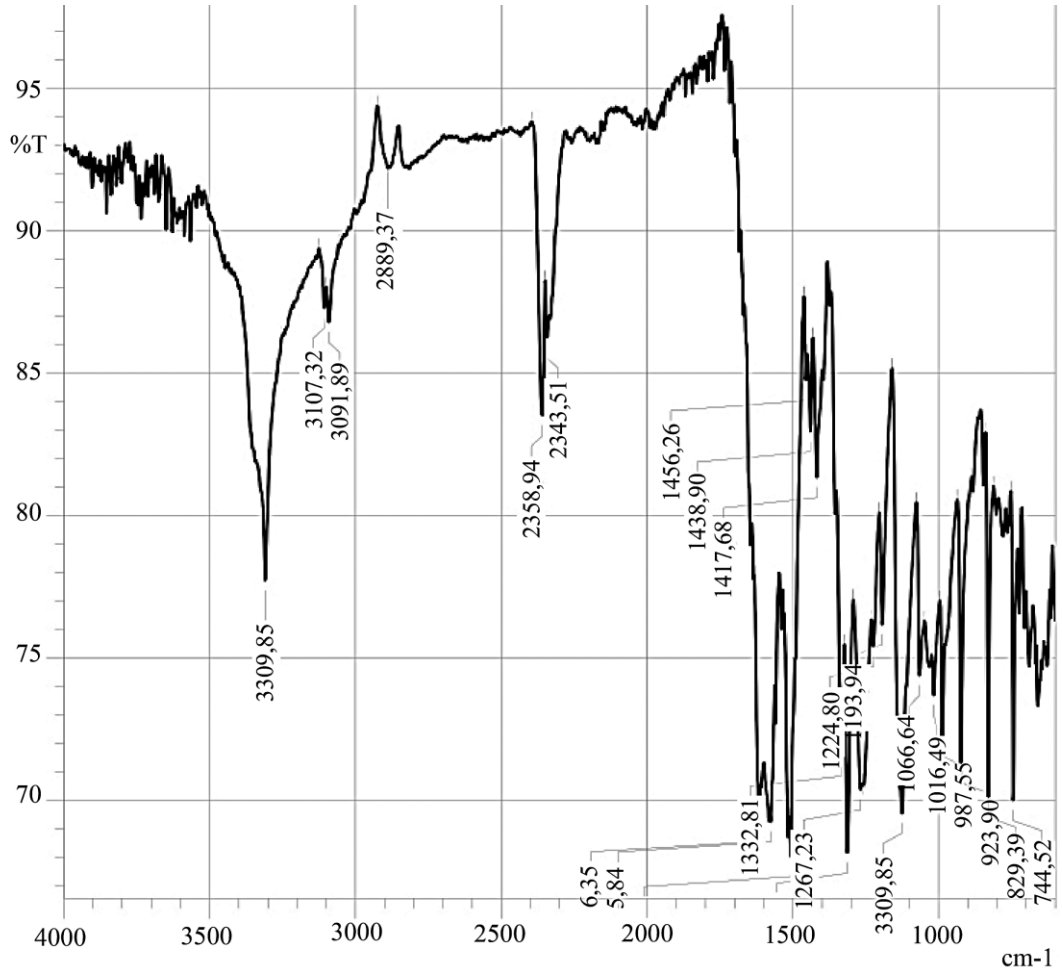
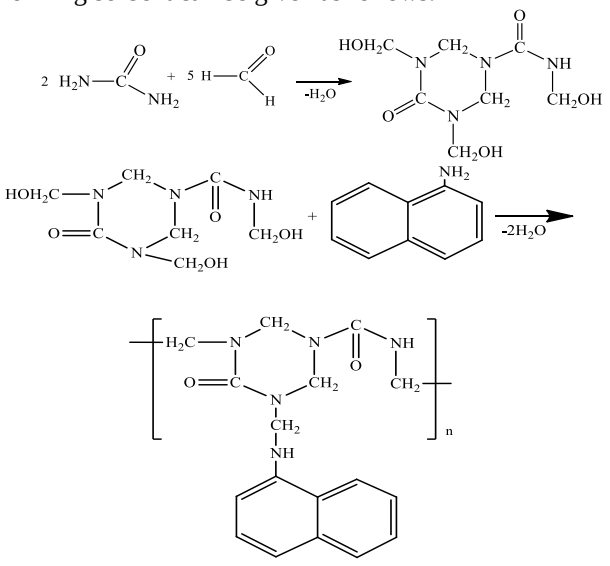



**Fig. 4 IR spectrum of the Ni (II) ion coordination compound with UFNA**

Based on the conducted research and obtained results, the approximate structural formula of KFNA complex-forming sorbent can be given as follows.



### *4.5. IR-Spectroscopic Analysis of the Complex Formation of KFNA Sorbent with Ni (II) ion*

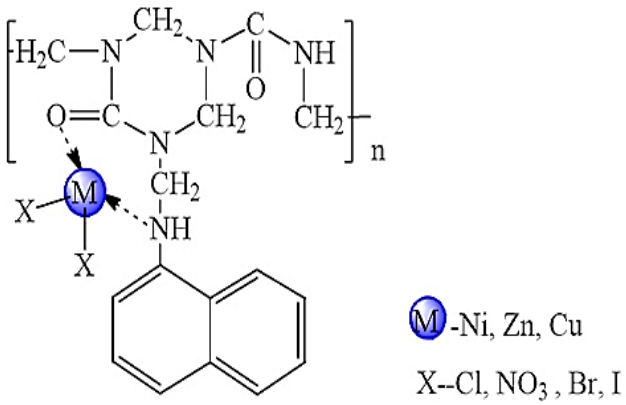
The IR spectra of the complex formed by KFNA sorbent with Ni (II), Zn (II) and Cu (II) ions were analyzed.

The IR spectra of the complex formed by the synthesized KFNA sorbent with $Ni^{2+}$ ions can be described as follows. Vibrations corresponding to the bound OH group were observed in the region of 3309 $cm^{-1}$. The bands in the region of 2889 $cm^{-1}$ correspond to methylene -$CH_2$, and the vibrations corresponding to the primary amide group in the region of 1610 $cm^{-1}$ were observed. Vibrations associated with the R-O-H group appeared in the region of 1267 $cm^{-1}$, and in the regions of 829-744 $cm^{-1}$, an out-of-plane vibration of the n (NH) group appeared (Fig. 4).

With KFNA, Ni (II), ions υ ($\delta(CH_2)+\delta(CN)$) 1626- $sm^{-1}$ have a vibrational frequency characteristic of the 1626-$cm^{-1}$ spheres, while the vibrational frequency of the KFNA sorbent υ (C-O) is in the 1138 cm-1 sphere, it was observed that after the sorption of metal ions of the sorbent, it shifted

according to the fields of KFNA + Ni (II) (1198 $sm^{-1}$). The obtained IR-spectroscopic analysis data indicate that the synthesized sorbent forms a complex with metal ions. When coordination is through nitrogen and oxygen atoms, the valence vibration frequencies of C-NH, C = O, bonds increase as a rule, and these shifts lead to the formation of C= O →$M^{+2}$ – N-$H_2$C coordination bond. indicates that

Based on the results of the IR-spectral analysis, the structure of the coordination compound formed by metal ions with KFNA can be presented as follows:




## 5. Conclusion

A complex-forming sorbent based on the polycondensation reaction of urea, formaldehyde, and 1-naphthiamine was synthesized. The effect of temperature on the process of polycondensation of the synthesized sorbent is presented. The complex-forming sorbent ion exchange sorption properties with some d-metals, depending on the ratio of reactants and static exchange capacity, have been studied. The sorption properties of urea, formaldehyde, and 1-naphthiamine in mole ratios from 2:5:0.1 to 2:5:0.3 with Cu (II) ions were observed in different environments: in a weakly acidic environment, 2 of urea, formaldehyde, and 1-naphthiamine: 5:0.2 mol ratio found the sorption index to be the highest. The surface of the synthesized sorbent was imaged using scanning electron microscopy. The presence of various holes and voids was observed on the surface of the complex-forming sorbent. This indicates that the sorbent has a very good sorption capacity for various metal ions. The results of the IR spectra of the synthesized sorbent are presented, and the IR spectra of the complex compound formed from some d-metals with Ni (II) ions are studied. The changes in the IR spectrum lines of the sorbent and the IR spectrum lines of the sorbent sorbing metal atoms are compared, and the difference in the shifts in vibration frequencies was brought. Approximate formulas of the complex formation of KFNA with Ni (II), Zn (II), Cu (II), and Co (II) ions are presented.

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