SYNTHESIS OF CHELATE PRODUCING SORBENT BASED ON THIOCARBAMIDE, FORMALIN AND SUCCINIC ACID AND ITS APPLICATION

G.J. Mukumova[1], Kh.Kh.Turaev[1], Sh.N. Muminova[1], Sh.A. Kasimov[1], Y.A. Geldiev[1], S.G. Yuldashova[2,3]

[1]Faculty of Chemistry, Termez State University. Termez, 190111 Uzbekistan.
[2]Department of Chemical Engineering, Termez State University of Engineering and Agrotechnologies. Termez, 190100 Uzbekistan.
[3]Department of Medical and Biological Chemistry, Termez branch of Tashkent Medical Academy. Termez, 190111 Uzbekistan.
e-mail: abrornomozov055@gmail.com

Received 09.12.2025
Accepted 20.02.2026

Abstract. *This study reports the synthesis of a chelating sorbent (TKFQ brand) based on thiourea, formaldehyde, and succinic acid at a molar ratio of 2:5:1 and a temperature of 70–80 °C. The structure of the obtained material and the IR spectra of its metal complexes were characterized. The reactivity of the sorbent was evaluated using quantum chemical analysis. Thermal behavior, surface morphology, and elemental composition were also investigated. The sorption performance toward various metal ions was assessed, and spectrophotometric determination of sorption capacity (SAS) yielded values of 0.5–5.7 kV/g for Cu(II), 0.7–5.9 kV/g for Zn(II), and 0.66–6.3 kV/g for Ni(II). Static sorption capacities and adsorption isotherms were analyzed using the Langmuir and Freundlich models. In addition, conditional thermodynamic parameters for Cu(II) sorption on the ligand at 20 °C were determined.*
Keywords: *thiourea, formalin, succinic acid, chelating sorbent, sorption capacity, Langmuir and Freundlich isotherms.*

Introduction

In recent years, significant attention in global industry has been devoted to the removal of various toxic metals and their compounds from natural and wastewater using sorbents and sorption methods capable of forming complexes with intermediate and 3d metal ions in solution. A major challenge in this field is the investigation of the composition, structure, and physicochemical properties of coordination compounds formed during the sorption process [1–7]. Mikhailov O.V. and Chachkov D.V. studied the formation and geometric parameters of M(II) complexes (M = Mn, Fe, Co, Ni, Cu, and Zn) in systems involving 10,11-hexaazatridecatetraene-1,3,8,11-dithiol-1,9 and 2,8,8,10,16-pentamethyl-3,4,6,7,11,12,14,15-octaazaheptadecapentaene–M(II)–hydrazinomethane thiohydrazide–acetone with self-assembled NNSS coordination, using the hybrid B3LYP density functional theory method [4]. The selectivity characteristics of chelating polymers, in particular [2-(2-pyridyl)ethyl]-substituted polyethyleneimines, have also been investigated. These polymers enable group separation of Cu(II), Ni(II), Co(II), and Cd(II) ions within the pH range of 4.5–8.0, while simultaneously allowing their effective discrimination from Mn(II), Zn(II), and Pb(II) ions. Modification of fibrous materials was carried out via liquid-phase oxidation with concentrated nitric acid, followed by impregnation with solutions of various chelating agents, including 1,10-phenanthroline, nitroso-R-salt, 8-hydroxyquinoline (oxine), and diphenylcarbazide. The sorption behavior of chromium and cobalt on the resulting sorbents was examined under static conditions with controlled contact time and pH values. Treatment of the fiber with 8-hydroxyquinoline, 1,10-phenanthroline, and nitroso-R-salt resulted in significant increases in cobalt recovery, by 38%, 34%, and 35%, respectively. At room temperature, the corresponding extraction efficiencies reached 67%, 53%, and 60% [8].

The acid–base behavior of the acyclic tetraaza gold(III) complex $[AuB]^{2+}$, in which B represents N,N′-bis(2-aminoethyl)-2,4-pentanediiminato(1−), was systematically examined under aqueous conditions. Additionally, the synthesis, crystal structure, and molecular geometry of its protonated

form, $AuHB(ClO_4)_4$, were characterized. The unit cell parameters were determined as follows: a = 11.964(2) Å, b = 13.789(3) Å, c = 15.496(3) Å, β = 109.00(3)°, and V = 2417.1(8) Å³ [9]. A tetraaza gold(III) complex containing a β-chlorinated pentanediiminate ligand, N,N′-bis(2-aminoethyl)-3-chloro-2,4-pentanediiminate, was obtained in the form of a bis-perchlorate salt with the molecular composition $Au(C_9H_{18}N_4Cl)_2$ and was successfully synthesized. This compound was thoroughly examined using single-crystal X-ray diffraction, phase analysis, electron spectroscopy, and infrared (IR) spectroscopy. The structural analysis revealed monoclinic crystals with the following parameters: a = 10.1290(5) Å, b = 7.2163(3) Å, c = 12.8010(7) Å, β = 111.865(1)°, V = 868.37(7) Å³, Z = 2, and calculated density ρ(calc.) = 2.347 g/cm³. The complex was found to comprise acentric $[Au(C_9H_{18}N_4Cl)]^{2+}$ cations and perchlorate $[ClO_4]^-$ anions [10].

According to the obtained results, the selectivity of these chelating ligands has a high selectivity for absorbing divalent metal ions in the following order: $Cu^{2+}>Pb^{2+}>Ni^{2+}>Co^{2+}$. As we know, one of the main tasks of sorbents is intermediate cleaning of sorption of various metal ions in different solutions. For this purpose, the prospects of using triethylenediamine $N(CH_2\text{-}CH_2)_3N$ (TEDA) sorbents based on silica gel with slightly high porosity for cleaning ^{137}Cs, ^{90}Sr, ^{90}Y and d-element ions (Cu^{2+}, Ni^{2+}) from aqueous solutions were studied. The obtained results show that 90Sr, 90Y radionuclides, as well as Cu^{2+}, and Ni^{2+} ions, are well sorbed in KSKG containing 0.01-6.72 wt% TEDA. In addition, the equilibrium time of sorption of these sorbents was reached within 3 hours, while the capacity of sorbents for copper Cu^{2+} varied from 63 to 320 mg, and it mainly depends on the conditions of sorbent synthesis, researchers S.A. Kulyukhin and M.P. Gorbacheva noted. Krasavina E.P. and others determined that the sorption capacity of these sorbents for Ni^{2+} does not exceed 130 mg per gram of sorbent [11, 12].

Experimental Part

Material. The chemicals employed in this study included thiourea, formalin (formaldehyde solution), and succinic acid. Additionally, 5 mL of ammonium hydroxide, a 5% sodium hydroxide solution, and aqueous solutions containing Zn(II), Cu(II), and Ni(II) metal ions were utilized.

Methods.

IR analysis. The chemical composition of the sorbent was examined by means of Fourier-transform infrared (FT-IR) spectroscopy using a Shimadzu IR Tracer-100 instrument (Shimadzu, Japan).

SEM-EDX analysis. Surface morphology images of the chelating sorbent were obtained at various magnifications using a JEOL JSM-IT200 scanning electron microscope (Japan) equipped with energy-dispersive X-ray spectroscopy (SEM-EDX) for elemental analysis.

TGA and DTA analysis. Thermogravimetric analysis (TGA) was performed to evaluate the thermal stability and changes in the physicochemical properties of the synthesized sorbents using a DTG-60 instrument from Shimadzu (Japan). The measurements were conducted under an argon atmosphere with a flow rate of 80 mL/min, and the heating rate was set at 10 °C/min.

Quantum chemical analysis. The reactivity of the TKFQ sorbent was evaluated using quantum-chemical computational methods. Molecular modeling and geometry optimization were carried out using Avogadro, HyperChem 8.01, and Accelrys Materials Studio 3.0.1 software packages. The calculations employed unrestricted Hartree–Fock (UHF) semi-empirical methods, including AM1, MNDO, PM3, RM1, and MINDO/3, within the self-consistent field molecular orbital (SCF-MO) framework. All computations were executed on a computer equipped with an Intel Pentium processor operating at 1.40 GHz.

Synthesis of sorbent. For the synthesis of the nitrogen- and oxygen-containing chelating ion-exchange resin, 1.52 g (0.02 mol) of thiourea was dissolved in 4 mL (0.05 mol) of formalin solution in a three-necked round-bottom flask fitted with a reflux condenser and a mechanical stirrer. The pH of the mixture was adjusted to 8–9 by gradual addition of ammonium hydroxide solution. The reaction mixture was heated at 70–80 °C with stirring until viscous. A solution of 2.36 g (0.02 mol) succinic acid in 10 mL ammonium hydroxide was added dropwise, and the temperature was raised to 110–

140 °C to form a solid or gummy resin. The product was dried at 100 °C for 20 h, ground, and purified by washing with 5 % NaOH followed by distilled water until neutral. The final product was a white, porous granular material with a yield of 91%.

Properties of sorbent. The moisture content of the synthesized sorbent according to GOST 10898.1–84, the mass density according to GOST 10898.2–84, the density of the sorbent in the hydrated state according to GOST 10898.3–84, the specific volume of the swollen sorbent according to GOST 10898.4-84, and the static exchange capacity according to GOST are determined by 20255.1-89. A chelating ion-exchange resin based on a polycondensation reaction was synthesized to develop sorbents with optimized properties and enhanced performance. The influence of reaction temperature on the polycondensation of thiourea, formalin, and succinic acid was systematically investigated. The process was conducted at four different temperatures: 110, 120, 130, and 140 °C. Throughout the experiments, the reaction time, swollen specific volume of the sorbent in water, and static exchange capacity (SEC) toward 0.1 M NaOH solution were evaluated. The obtained results are summarized in Table 1.

Table 1. Influence of polycondensation temperature on the reaction time, swollen specific volume, and static exchange capacity of the synthesized ion-exchange resin.

№	Reaction temperature T, °C	Reaction time τ, hours	Specific volume of sorbent dissolved in water form H, ml/g	SAS, in mg-eq/g of 0.1 N NaOH solution
1.	110	5-6,5	1,7	3,3
2.	120	4,5-5	1,4	3,7
3.	130	2,5-3	1,2	5,1
4.	140	1,5-2	1,0	4,1

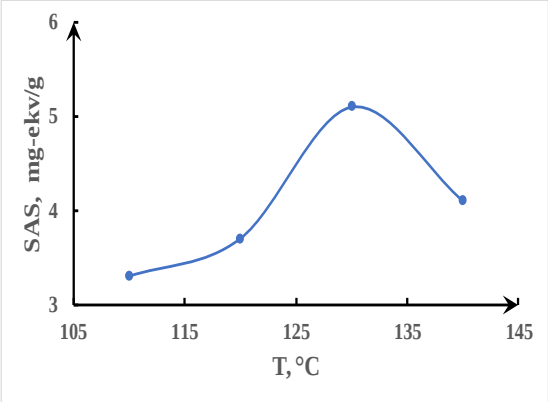


Fig. 1. Graph of the temperature dependence of the synthetic exchange capacity of the synthesized TKFQ sorbents

As shown in Table 1 and Fig. 1, the polycondensation reaction at 110 °C required 5–6.5 hours and yielded a sorbent with a static exchange capacity (SEC) of 3.3 mg-eq/g toward KFV, owing to the lower reactivity of the monomers at this temperature. Increasing the temperature to 140 °C accelerated the reaction, reducing the duration to 1.5–2 hours, but simultaneously decreased both the SEC and the swelling degree of the resin. This behavior is attributed to the formation of a denser crosslinked structure, which restricts the mobility of ionogenic groups. Based on these findings, an optimal reaction temperature of 130 °C was selected, providing a reaction time of 2.5–3 hours and a maximum SEC of 5.1 mg-eq/g for 0.1 M NaOH [13]. To optimize the structure and performance of the ion exchanger, the effect of the initial monomer ratio was also investigated [14]. Several resin samples were prepared by varying the molar ratio of thiourea:formalin:succinic acid from 2:5:0.5 to 2:5:1.5 [15]. The influence of succinic acid content on the properties of the TKFQ ion exchanger is summarized in Table 2.

Table 2. Effect of reactant molar ratio on the sorption and ion-exchange properties of the TKFQ resin

Molar ratio (thiourea: formalin: succinic acid)	Reaction product, %	Static exchange capacity, mg-eq/g, 0.1 N solutions		
		Cu^{2+}	Zn^{2+}	Ni^{2+}
2:5:0.5	89	4.03	5.30	5.10
2:5:1.0	91	5.70	5.90	6.30
2:5:1.5	88	4.55	4.60	4.54

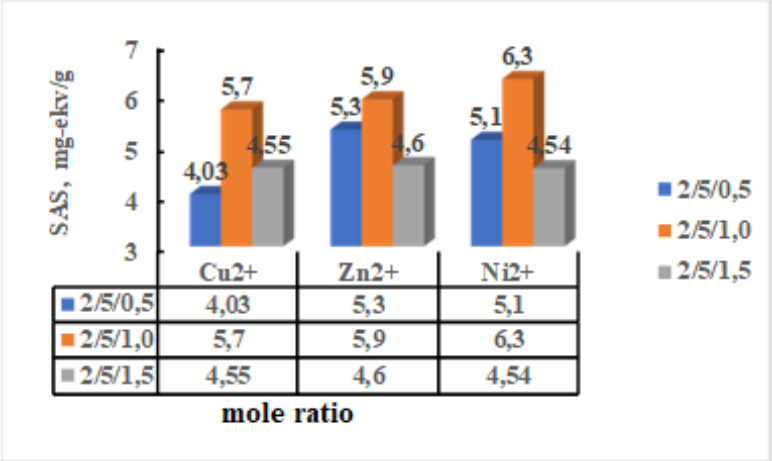
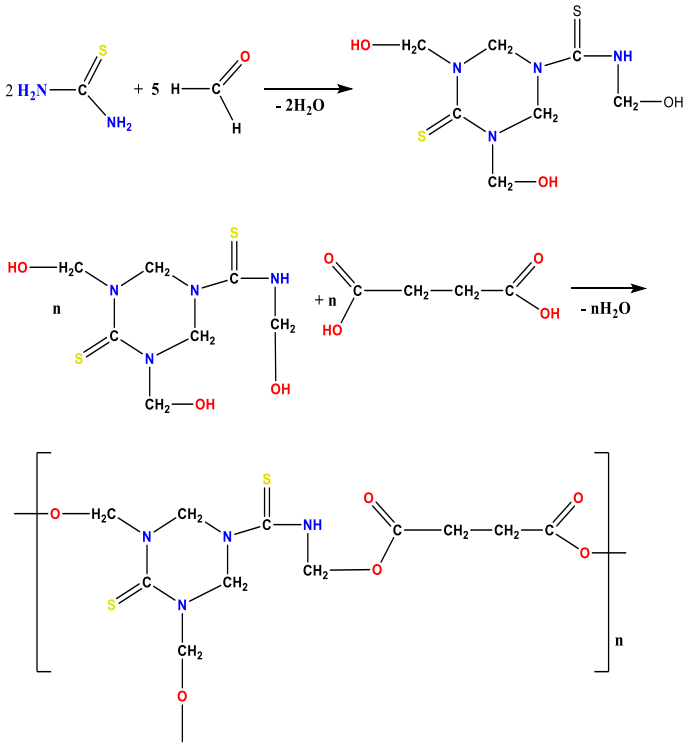


Fig. 2. SAS dependence diagram of thiourea. formalin and succinic acid in mole ratio

The data in Table 2 and Fig. 2 indicate that the optimal molar ratio for obtaining the ion exchanger with the highest performance is thiourea:formalin:succinic acid = 2:5:1.0. At this composition, the static exchange capacity (SEC) of the TKFQ resin toward metal ions in 0.1 M solution reached 5.7 mg-eq/g for Cu(II). 5.9 mg-eq/g for Zn(II), and 6.3 mg-eq/g for Ni(II) [16].

Based on the experimental findings, the synthesis of the TKFQ sorbent can be represented by the following polycondensation reaction Scheme:



In the CHem3D 21.0.0 program. different views of the TKFQ sorbent in X. Y and Z coordinates were obtained.

Results and Discussion

IR-spectroscopic analysis.

TKFQ sorbent and its metallocomplexes with Cu(II), Zn(II) and Ni(II) ions. The Fourier-transform infrared (FT-IR) spectrum of the TKFQ sorbent, recorded on a Bruker instrument, revealed several characteristic absorption bands. FTIR analysis revealed characteristic vibrations of the polymer's functional groups: the primary amine (–NH_2) at 3294.78 and 1140.44 cm^{-1}; C–N at 1020.79 cm^{-1}; methylene (–CH_2–) stretching at 2908.42–2810.38 cm^{-1} and bending at 781.49 cm^{-1}; and carboxylate (–COO^-) and carbonyl (C=O) at 1660.24, 1324.47, and 866.92 cm^{-1}. Stretching vibrations associated with the ester (–COOC–) and thiocarbonyl (C=S) groups were identified at 1264.47 cm^{-1} and 1202.54 cm^{-1}, respectively. The full FT-IR spectrum is shown in Fig. 3.

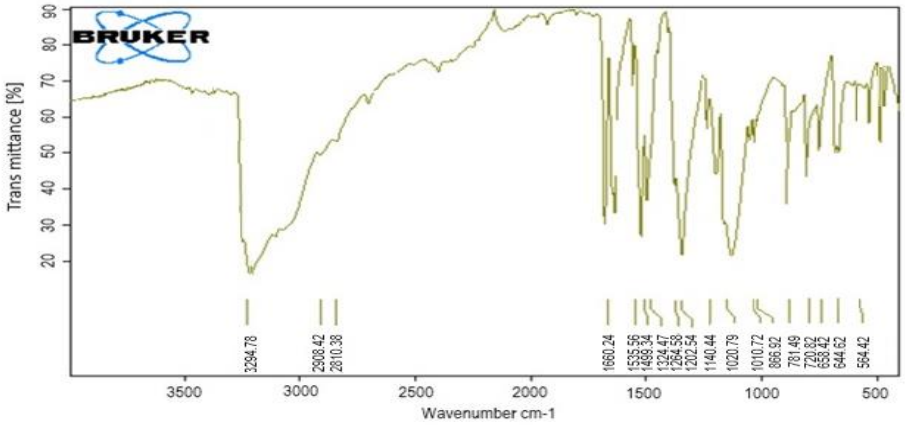


Fig. 3. IR- spectrum of TKFQ sorbent.

IR-spectroscopic analysis of the Cu(II) ion with TKFQ sorbent. The FT-IR spectrum of the TKFQ sorbent after Cu(II) sorption, recorded on a Bruker instrument, is shown in Fig. 4. Comparison with the spectrum of the unloaded sorbent revealed several notable shifts and changes in band intensities. FTIR analysis showed –NH_2 N–H stretching/bending at 3211.48 and 1098.21 cm^{-1}, C–N at 1011.34 cm^{-1}, –CH_2– C–H stretching/bending at 2952.92–2871.98 and 792.62 cm^{-1}, and C=O/–COO^- asymmetric stretching and deformation at 1702.27, 1321.35, and 832.57 cm^{-1}. Additionally, the stretching vibrations attributed to the ester (–COOC–) and thiocarbonyl (C=S) groups were identified at 1258.28 cm^{-1} and 1206.39 cm^{-1}, respectively [17].

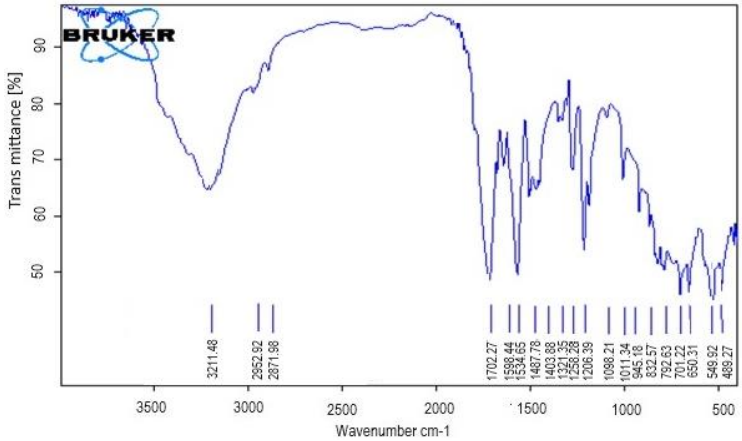


Fig.4. IR-(BRUKER) spectrum of Su(II) complex formed with TKFQ sorbent

TGA and DTA analysis of the sorbent.

The thermal behavior of the TKFQ sorbent was systematically investigated using differential thermal analysis (DTA) and thermogravimetric (TG) analysis. The DTA curve (Fig. 5) exhibits a series of well-defined thermal events, characterized by endothermic peaks at 135.01, 193.43, and 294.39 °C, alongside exothermic peaks at 172.27, 242.34, and 350.03 °C, indicating the occurrence of multiple overlapping physicochemical transformations.

The TG profile reveals a three-stage mass-loss process within the studied temperature range. The first stage occurs between 99.75 and 220.88 °C and is associated with a mass loss of 1.696 mg (11.27 wt%), over a duration of 12.22 min, which can be attributed to the removal of physically adsorbed moisture and low-molecular-weight volatile species. The second stage, observed in the temperature interval of 215.43–234.49 °C, proceeds rapidly within 1.17 min and corresponds to a substantial mass loss of 10.216 mg (67.81 wt%), indicating the primary decomposition of the sorbent matrix. The third stage, occurring between 234.49 and 320.03 °C, involves an additional mass loss of 1.906 mg (12.66 wt%), likely associated with the further degradation and carbonization of residual fragments.

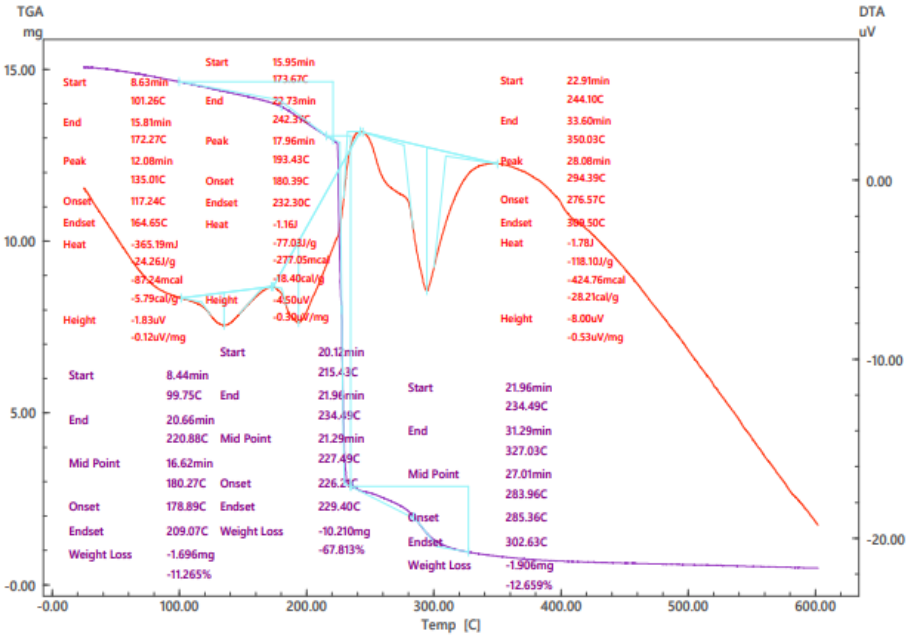


Fig. 5. Graph of thermal analysis for TKFQ sorbent

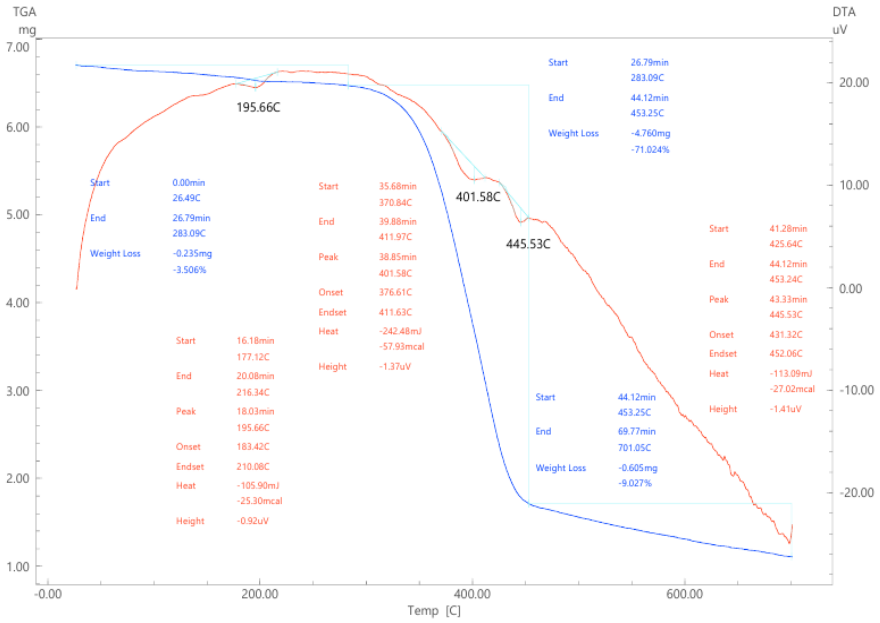


Fig. 6. Graph of thermal analysis for the metallocomplex formed by TKFQ sorbent with Cu(II)

Complete thermal decomposition of the TKFQ sorbent is achieved after 31.29 min [18, 19]. Based on the TG data (Fig. 6), the decomposition process is accompanied by the evolution of gaseous products, including H_2O, NH_3, CO, SO_2, and CO_2, which are plausibly generated through the cleavage of functional groups within the sorbent structure.

SEM-EDX- analysis. According to the results of the conducted research, the remains of unreacted initial substances are not visible in the 1:100 and 1:1000 scale images of the sample of the TKFQ sorbent with a new composition. During the research, it was found that the reaction took place to the end, and it allowed us to get information about the composition of the elements of the substances formed in the parallel reaction [20]. The obtained results are presented in Fig. 7 and 8.

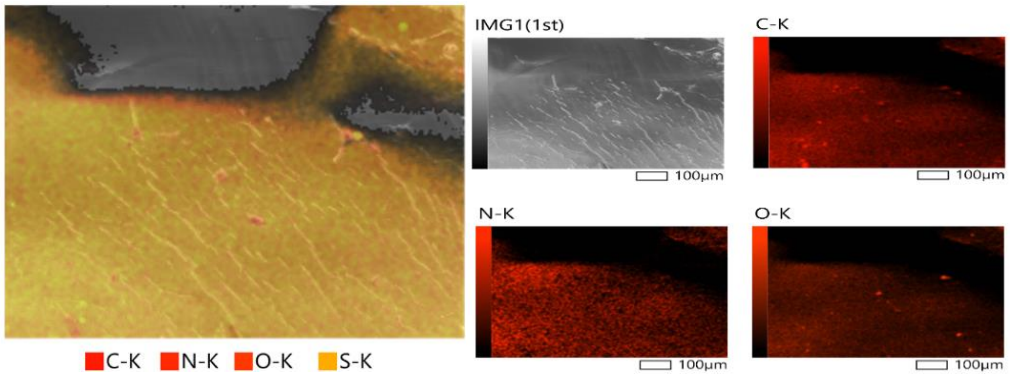


Fig. 7. SEM image of TKFQ complex forming sorbent

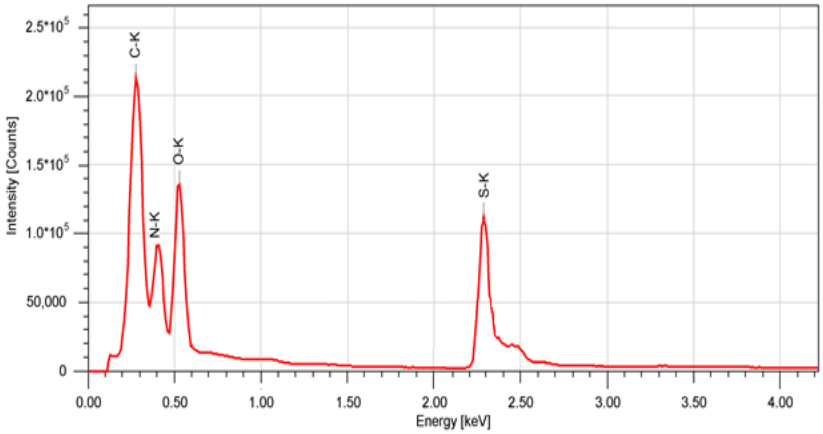


Fig. 8. Elemental analysis of TKFQ complex forming sorbent

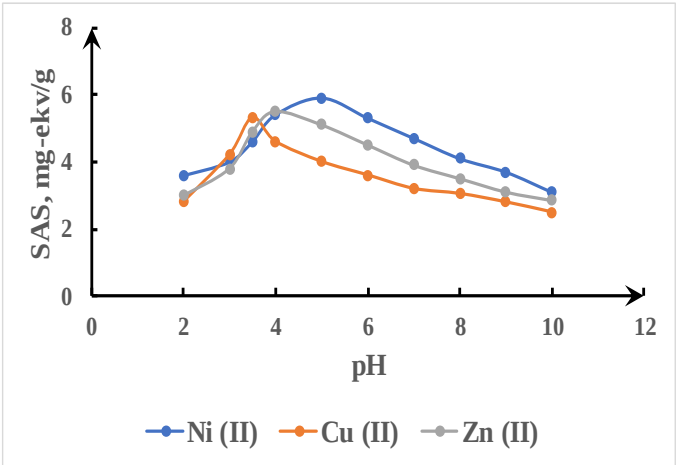


Fig. 9. Dependence of sorption of Cu(II), Zn(II) and Ni(II) ions on TKFQ sorbent on environmental pH value (C_{Me}=0.1 n; m_{sorb}=0.1 g; τ =2 s; V=10 ml)

Effect of pH on metal sorption. The metal sorption study of the TKFQ sorbent showed its static exchange capacity (mg-equiv/g) at the corresponding pH values as follows: Cu (II) – 5.3 (pH =3.5); Ni (II) – 5.9 (pH =5.0); Zn (II) – 5.5 (pH =4.0) As can be seen from the graph in Fig. 9, the sorption level of metal ions exceeds the maximum in the range of the pH value of the solution medium in this sorbent from pH=3 to pH=6.

The degree of sorption of studied metal ions on TKFQ sorbent increases in the following order:

Cu(II) < Zn (II) < Ni (II)

Calculating the sorption capacity of metal ions. Finding the sorption capacity of metal ions was carried out for all sorbents under static conditions: pH Cu(II)=4.2, pH Zn(II)=6.0-6.5, and pH Ni(II)=5.3. Metal salts (sulfates, chlorides, and nitrates) were used. The solution medium was set with a universal buffer solution. For sorption. 40.00 mg of sorbent was taken and left in 10.00 ml of distilled water for 1 hour. Then the dried sorbent was added to the studied solution (10.00 ml) and stirred in a thermostated water bath for 4.5 hours. Then the sorbent was separated from the solution, and the pH value of the filtrate was measured. In the EMC-31PC-UV spectrophotometer. Cu(II) was measured in a 25% ammonia solution at λ_{max} Cu(II)=610 nm, Zn(II) at λ_{max} Zn(II)=538 nm, and Ni(II) at λ_{max} Ni(II) using dimethylglyoxime. At 445 nm, the concentration of components was determined spectrophotometrically based on the difference in optical density of the initial and post-sorption solutions when ℓ=1.0 cm (Tables 3-5, Fig.s 10-12).

Table 3. Spectrophotometric determination of SAS of Cu(II) ions in TKFQ

№	Sor bent	T°C	pH	Cu^{2+}. mkg-ekv/ml	λ	A before sorption	After sorption A	ΔA	SAS mg-kv/g
1	TKFQ	50	4.2	50	610 nm	0.074	0.024	0.050	0.5
2				100		0.154	0.026	0.128	1.27
3				200		0.308	0.030	0.278	2.76
4				300		0.454	0.025	0.429	4.25
5				400		0.606	0.029	0.577	5.7
6				500		0.645	0.068	0.577	5.7

Table 4. Spectrophotometric determination of SAS of Zn(II) ions in KFQ

№	Sor bent	T°C	pH	Zn(II) mkg-ekv/ml	λ	Before sorption. A	After sorption. A	ΔA	SAS mg-ekv/g
1	TKFQ	50	5.3	50	445 nm	0.052	0.016	0.036	0.7
2				100		0.094	0.020	0.074	1.41
3				200		0.174	0.023	0.151	2.88
4				300		0.252	0.019	0.233	4.44
5				400		0.332	0.021	0.311	5.9
6				500		0.372	0.061	0.311	5.9

Table 5. Determination of static exchange capacity of Ni (II) with TKFQ sorbent

№	Sor bent	T°C	pH	Cu^{2+}. mkg-ekv/ml	λ	A before sorption	After sorption A	ΔA	SAS mg-kv/g
1	TKFQ	50	5	50	495	0.070	0.020	0.050	0.66
2				100		0.134	0.028	0.106	1.4
3				200		0.260	0.032	0.228	3.0
4				300		0.384	0.025	0.362	4.74
5				400		0.512	0.028	0.484	6.3
6				500		0.540	0.056	0.484	6.3

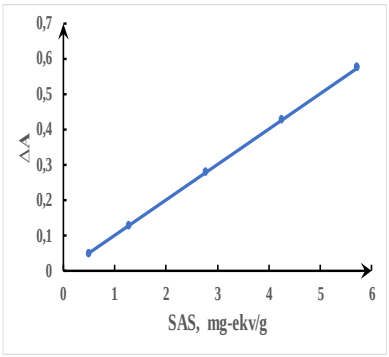


Fig. 10. Graph of determination of static exchange capacity of Cu(II) ion in TKFQ

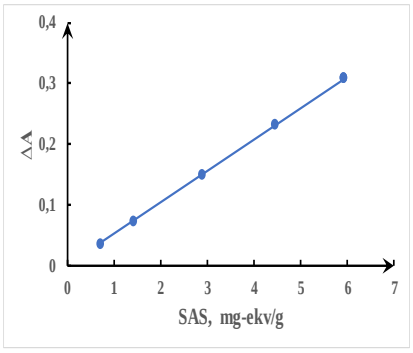


Fig. 11. Graph of determination of static exchange capacity of Zn(II) ion in TKFQ.

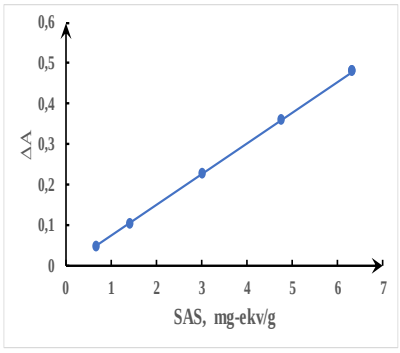


Fig. 12. Graph of determination of static exchange capacity of Ni(II) ion in TKFQ.

Sorption isotherm of sorbents. Synthesized sorbents were studied according to the sorption isotherm of Cu (II) ion, and the obtained results were presented graphically in Fig. 13. The results obtained in the experiments were processed according to the Langmuir and Freundlich models (Tables 6 and 7).

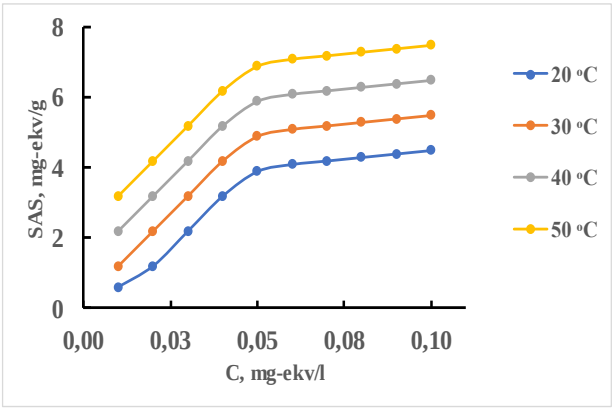


Fig. 13. Sorption isotherms of Cu (II) ions in TKFQ at different temperatures (t_{sorb}=25 mg, t=4 h, pH=6, V=25 ml)

Table 6. Values of F_{exp} and coefficients of determination (R^2) for the sorption model of Cu (II) ions on studied ligands (F_{mezon}=2.74; P=0.95; f_1=6; F_2=16)

Sorbent	**Model**		**Temperature °C**			
			25	35	45	55
TKFQ	Langmuir model	R^2	0.989	0.987	0.987	0.993
		F_{exp}	0.58	0.62	0.88	0.79
	Freundlich model	R^2	0.951	0.965	0.992	0.995
		F_{exp}	7.63	5.29	2.17	1.93

Table 7. Constants of sorption isotherms of Cu (II) ions according to the Langmuir model

Sorbent	**Constants**	**Temperature °C**			
		25	35	45	55
TKFQ	$G_\infty \cdot 10^5$. mol/g	13.71	14.39	15.73	18.12
	K_L. l/mol	$29.08 \cdot 10^5$	$22.1 \cdot 10^5$	$18.3 \cdot 10^5$	$12.7 \cdot 10^5$

Table 8. Conditional thermodynamic parameters of Cu (II) ion sorption on ligands at a temperature of 20 °C.

Sorbent	ΔH. kJ/mol	ΔS. J/mol	ΔG. kJ/mol
TKFQ	-16.0	24.0	-29.5

Table 8 shows that the Cu(II) ion is self-sorbed on sorbents with a decrease in free energy and enthalpy and an increase in entropy. The increase in entropy of the system with the formation of a coordination compound in the sorbent phase can be explained by the decomposition of the solvate clouds of the active functional groups of the ligand.

Conclusion

Optimal conditions for the synthesis of the TKFQ sorbent were established, namely a temperature of 130 °C, a reaction time of 2.5–3 h, and a molar ratio of initial reagents of 2:5:1. Under these conditions, the static exchange capacity of TKFQ toward 0.1 N NaOH was found to be 5.1 mg-eq g^{-1}. The static exchange capacities with respect to various metal ions in 0.1 N solutions were also determined. The structure of the metal–TKFQ complexes was elucidated using Raman and IR spectroscopy in combination with quantum-chemical calculations performed with Avogadro, HyperChem 8.01, and GaussView 6.0.16. Surface morphology and elemental composition of the resulting complexes were investigated by thermal analysis and SEM–EDX techniques. The effect of the solution environment on metal ion sorption was systematically studied. It was established that the sorption capacity of TKFQ reaches a maximum in the pH range of 3–6, indicating that moderately acidic conditions are optimal for efficient metal uptake. The degree of sorption of studied metal ions on sorbents increases in the following order:

$$TKFQ \rightarrow Cu(II) < Zn(II) < Ni(II).$$

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