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Original Article

Synthesis and Research of Sorbent Based on Sodium Metalsilicate and Thiourea

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Abstract *- The purpose of this study is to synthesize and research a new type of sorbent based on thiourea and sodium metasilicate, which has a wide range of applications and has selective sorption properties due to the presence of the thio group in the molecule and the presence of silicon in its composition. During the synthesis of the sorbent, 60 g of thiourea and 100 g of sodium metasilicate were taken from an aqueous solution and heated in a water bath at a temperature of 90 °C. The duration of the reaction is 25-30 minutes, after 30 minutes the reaction mass cools down to room temperature, and after 24 hours the mass comes to a brittle state in the form of a gel. The resulting mass was crushed to a size of 1 mm and thermally treated at a temperature of 200 °C. The synthesized sorbent was thoroughly washed in distilled water and stored at 105°C-110°C until the mass reached a constant state. The yield of the reaction product is 91%. The reaction mechanism of the organ hybrid sorbent based on thiourea and sodium metasilicate was compared and the IR-spectra of the initial reagents and the obtained product were analyzed and the reaction mechanism was proposed. At the same time, the results of the thermal analysis of the obtained sorbent and SEM analysis of the sorbent were presented.*

Keywords *- Corbent, Thiomochevine, Sodium metasilicate, IR spectrum, Thermogravimetric curve, Mass loss, Scanning electron microscopy.*

1. Introduction

At present, the synthesis of new types of ions, determination of sorption properties of metal ions, and creation of technology for extracting precious metal ions from solutions using them are urgent tasks. In addition, the synthesis of ions and sorbents with selective properties for the sorption of various precious metals and their implementation will bring high economic benefits to the precious metals industry. It is known from the literature that the condensation reaction of urea and thiourea with formaldehyde in an acidic environment has been studied. The obtained products were tested as ion exchange resins and the ion exchange capacity of these resins was investigated [1].

Metal cations are the main pollutants of the lithosphere. Accumulation of serum in natural water resources can cause serious environmental problems. Another aspect is the accumulation of metallurgical waste. Powdered and granular calcium aluminosilicates ($CaO-MgO-SiO_2-Al_2O_3$) capable of immobilizing cerium ions (Ce^{3+}) contained in metallurgical waste near Chelyabinsk, Russia, were studied as raw materials for the production of composite sorbents. During the experiments, aqueous solutions with a high concentration of Ce^{3+} ions (1000 mg/l) were used. The migration properties of calcium (Ca^{2+}), magnesium (Mg^{2+}), silicon (Si^{4+}) and aluminium (Al^{3+}) ions were studied. The separation rate of Ce^{3+} ions in 24 hours was 93.36% for powder sorbents and 99.98% for granular sorbents. The interaction of sorbents with sorbate was carried out by the exchange of Ca^{2+} and Mg^{2+} ions from the sorbent matrix to the aqueous solution. The desorption process was studied in distilled water. The results of the study showed a high degree of irreversible sorption of Ce^{3+} ions from aqueous solution to powder and granular sorbents. The low price, environmental aspects of storage and high sorption capacity of crushed metallurgical waste slag allow to process of this raw material and use it as a sorbent, and then clean large natural objects from Ce^{3+} ions [2-4].

Anthropogenic organic pollutants restore sensitivity to human ecosystems even at low or studied concentrations. Control of nanomaterials' (NM) unique structural and surface properties with good sorption capacity at the level of sensitivities for surfactants is associated with query communications. In this regard, various NMs (metal and mixed oxide nanoparticles (NZ), carbon NMs (fullerenes, carbon nanotubes, graphene and graphene oxide), polymer-based nanocomposites (organic polymers, inorganic and hybrid polymers, and dendrimers) are considered. Specific features of NM-based extraction methods include comprehensive analysis burdens, the vision of future indicators, and key challenges[5]. Two novel inorganic ion exchange materials magneso-silicate and magnesium

alumino-silicate have been synthesized under identical conditions. Magneso-silicate and magnesium alumino-silicate were found to have the formulas $MgSi_{5.59}O_{12.18}\cdot 5.93H_2O$ and $MgAl_{2.32}Si_{5.2}O_{14.88}\cdot 18.23H_2O$, respectively. The ion exchange capacities of these materials for some radionuclides and heavy metals Cs^+, Co^{2+}, Cd^{2+}, Zn^{2+} and Cu^{2+} were investigated and the data obtained showed that magnesium alumino-silicate has a higher capacity for these cations compared to magneso-silicate. Distribution coefficients in the nitric acid medium have been evaluated to explore the separation potentiality of magneso-silicate and magnesium alumino-silicate for Cs^+, Co^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+} and Fe^{3+} ions. Sorption isotherms for all cations were investigated and the data showed the applicability of Freundlich isotherms for all cases[6].

Hybrid ions were obtained based on anions containing manganese (IV) oxide, macroporous, strong base carrier. MnO_2 (26.4% Mn) is amorphous and uniformly distributed in the polymer matrix. The hybrid material was used to extract sulfides from deoxygenated aqueous solutions with concentrations of 100–500 $mgS^2–/dm^3$ and pH in the range of 12.0–14.0. The highest sorption capacity of the sorbent was 152.0 $mgS^2–/g$. About 60% of the dissolved sulfides were oxidized to S_2O_3 and returned from the ions composition to the aqueous phase of the studied system, while 15% was bound to the sorbent granules in the form of polysulfides. The mechanism of the extraction process of sulfides as a result of adsorption and oxidation using the synthesized hybrid material is proposed[22].

Tubular porous ceramic substrates based on crystalline SiO_2 were successfully functionalized with amino and mercapto functions using the one-step sol-gel approach. All the amino-functionalized membranes showed 95% retention of Ni(II) ions from an aqueous solution for two hours of filtration in the filtration unit (at room temperature). Whereas, maximum retention by mercapto-functionalized membranes was 30% of Pb(II) ions[8].

In the synthesis of ion-exchange polymers, the polycondensation method is widely used for the synthesis of a large group of valuable heat-resistant high-molecular compounds. The widespread use of this method in the chemical industry is also connected with the availability of raw materials (ammonia, urea, thiourea, aldehydes, and acrylonitrile) in our Republic. The process of polycondensation has been sufficiently analyzed in a large number of monographs and analytical scientific articles, and the technological parameters of this process have been developed [9].

A new design of inefficient sorbents for the removal of trace pollutants from the water was proposed: grafting the external surface of γ-alumina (γ-Al_2O_3) nanofibers with functional groups that have a strong affinity to the contaminants. The product sorbents could capture the pollutants selectively even when the concentration of the contaminants is extremely low. Two types of γ-Al_2O_3 nanofibers with different sizes were prepared via facile hydrothermal methods. Thiol groups were then grafted on the γ-Al_2O_3 fibers by refluxing the toluene solution of 3-mercaptopropyltrimethoxysilane (MPTMS). The thiol group modified fibers not only can efficiently remove heavy metal ions (Pb^{2+} and Cd^{2+}) from water at high flux, but also display high sorption capacity under sorption equilibrium conditions. A similar result was obtained from the nanofibers grafted with octyl groups which are employed to selectively adsorb highly diluted hydrophobic 4-nonylphenol molecules from water. This study demonstrates that grafting nanofibers is a new and effective strategy for developing efficient sorbents[23].

A high-performance novel extraction method for heavy metal ions from aqueous solutions is investigated using a microwave-assisted heating technique. The selected solid material is a functionalized nano-magnetic iron oxide with 3-aminopropyltriethoxysilane [Nano-Fe_3O_4–SiO_2–NH_2] which is synthesized using a microwave-assisted procedure. The metal sorption properties of this magnetic nano-sorbent with four different divalent metal ions, viz. Pb(II), Cu(II), Cd(II) and Hg(II) were evaluated by heating the interacting metal ion solution in contact with the sorbent in a microwave oven for 5–30 s. Pb(II) was found the highest extracted metal ion and the capacity value was identified as 1250 μmol g^{-1} after heating for 5 s, while the equilibrium capacity value was obtained as 1300 μmol g^{-1} at 15 s. The maximum capacity values of Cu(II), Cd(II) and Hg(II) were characterized as 1050, 350 and 350 after microwave heating for 25, 15 and 25 s, respectively. The functionalized Nano-Fe_3O_4–SiO_2–NH_2 sorbent was synthesized using the microwave-assisted preparation system and characterized by the FT-IR, SEM, HR-TEM, XRD-method and surface area determination[11-13].

The reaction of thiourea with various substances is reported in the literature, one of which, thiourea, reacts violently with epichlorohydrin at room temperature with heat evolution. To keep the rate of reaction of epichlorohydrin with thiourea uniform and to reduce exothermicity, the condensation reaction is carried out in the presence of a solvent. Ethyl, isoamyl alcohols, dimethylformamide, and toluene are used as solvents[14,15].

A high yield in obtaining polymer sorbents is achieved when using isoamyl alcohol and dimethylformamide. The reaction rate and polymer yield depend on the amount of solvent used. The effect of thiourea and solvent mass ratio (0.5:1.0:1.5:2.0:2.5) on the duration of the condensation reaction and the properties of the anions was studied in detail. When 0.5-0.6 parts by mass of solvent are used per 1 part by mass of thiourea, the condensation reaction proceeds at a rapid rate and a brittle polymer with low mechanical strength is formed. An increase in the amount of solvent from 1.5 to 2.5 parts by mass leads to an increase in the reaction mass solidification time (100-120 hours). When one part by mass of solvent and 1 part by mass of thiourea are used, the condensation process proceeds smoothly and the resulting product has good mechanical strength and

sufficient exchange capacity[16,17]. In the IR spectrum of the product obtained in a strongly acidic and neutral environment, there are almost no absorption lines corresponding to the epoxide group, which indicates that the interaction of thiourea with epichlorohydrin is due to the opening of the chlorine atom and the epoxide group in epichlorohydrin, and the amine group in thiourea is due to the interaction with the mobile hydrogen [18,19]. A novel sorbent based on UVM-7 mesoporous silica doped with Ti has been synthesized and used for solid-phase extraction of several organophosphorus pesticides in environmental water samples followed by gas chromatography coupled to a nitrogen-phosphorus selective detector. Thus, mesoporous silica materials doped with Ti and Fe as well as immobilized cyclodextrin silica-based supports were prepared and morphologically characterized by several techniques such as transmission electron microscopy, nitrogen adsorption-desorption and X-ray diffraction[20,21].

2. Experimental Part

In the article, the processes of synthesis of sorbent based on the interaction of thiourea with sodium metasilicate and various properties of the obtained sorbent were studied. In the process, raw materials were withdrawn in predetermined mass proportions. 100 g of thiourea was taken from an aqueous solution of sodium metasilicate (instead of an aqueous solution of sodium metasilicate, a solution with a silicate modulus of 1.8 was used) and heated to 90 °C in a stainless steel reactor with a water bath and a stirring mechanism and a temperature controller. The duration of the reaction is 25-30 minutes, and the reaction is carried out continuously at a rotation speed of 60 times per minute, in the process, thiourea and sodium silicate are interconnected by hydrogen bonding in an alkaline medium. After 30 minutes, the reaction mass is cooled to room temperature and kept at this temperature for 24 hours, after 24 hours the mass becomes a brittle state in the form of a gel. The resulting mass was crushed to a size of 1 mm and thermally treated at a temperature of 200 °C. As a result of the breaking of not very strong hydrogen bonds during heat treatment, ammonia gas is released and silicon-oxygen and oxygen form a carbon bond. The synthesized sorbent was thoroughly washed in distilled water, placed in a drying oven at 105°C-110°C, and kept at this temperature until the mass reached a constant state. The yield of the reaction product is 91%. Excess Na^+ ions contained in the sorbent are washed away during the washing of the obtained sorbent in distilled water.

3. Results and its Discussion

In order to obtain information about what substance or substances are formed as a result of the reaction or what functional groups are formed in the resulting substance, as well as the changes occurring in the existing functional groups, the sample is 4000 cm^{-1} to 400 cm^{-1} (wavelength 10^{-6} - 10^{-3} m) in the area where the IR-spectrum is obtained and analyzed. As a result, it is possible to conclude the class of the sample and its theoretical and practical properties.

The total energy of the molecule consists of the sum of the energies of these movements, that is, the energies of electronic, vibrational, and rotational movements:

$$E_{full} = E_{elec} + E_{vibr} + E_{rota} + E_{forw}$$

In the IR-spectroscopy method, only actions in the 2nd and 3rd cases are considered.

An IR-Fure spectrometer manufactured in Japan (SHIMADZU) was used during the research. The analysis of the spectra was carried out with the help of the main software, which performs automatic measurement of the spectra, and organizes work with a spectrum library with a very wide base for displaying the graph of the spectra and their parts.

In the study, thiourea was used as the starting organic substance and its IR-spectroscopic analysis was carried out. The IR spectrum of thiourea is presented in Fig. 1.

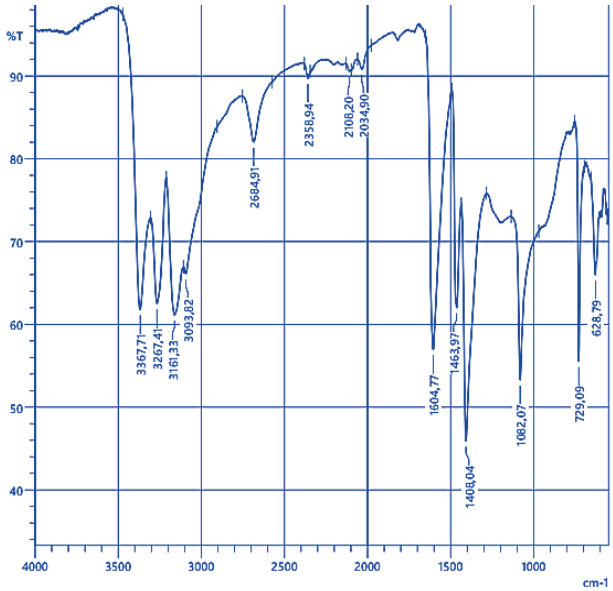


Fig. 1 IR spectrum of thiourea

As can be seen from the above figure, the absorption peaks in the region of 3367.71 cm^{-1},-3267.41 cm^{-1} and 3161.33 cm^{-1} have a high-intensity triplet of thiourea belonging to the -NH_2 group and were caused by valence vibrations. In addition, the absorption peaks at 2684.91 cm^{-1} are bonds caused by asymmetric and symmetric valence vibrations of the thiourea -NH_2 group.

The next bonds are 2358.91cm^{-1}- 2108.20cm^{-1}, absorption peaks at 2034.90 cm^{-1} belong to the -NH_2 group, absorption at 1064.77 cm^{-1} and 628.79 cm^{-1} belong to the -NH_2 group and are caused by vibration and intensity is moderate. The vibrational frequency belonging to the S=S group appears in the areas of 1463.97 cm-1, 1408.04 cm^{-1} and 1082.07 cm^{-1}.

Figure 2 shows the IR spectrum of the sorbent (4-c) synthesized based on sodium metasilicate and thiourea.

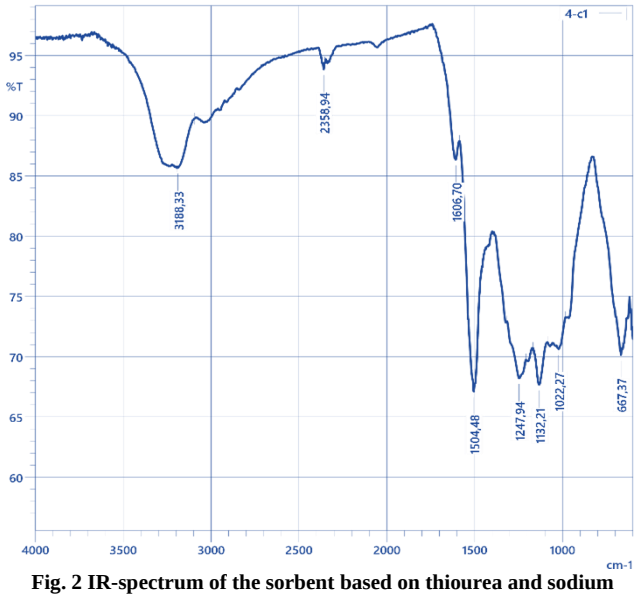


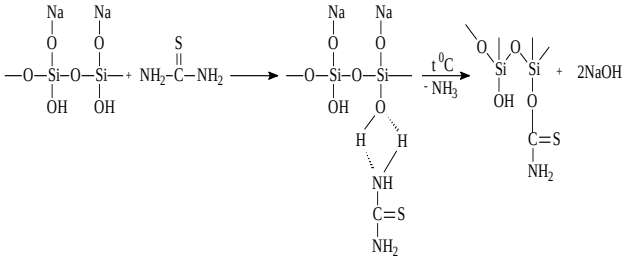
Fig. 2 IR-spectrum of the sorbent based on thiourea and sodium metasilicate

Depending on the absence of absorption bands in the high-frequency part of the IR spectrum (3600-3100 cm^{-1}), it is concluded about the presence or absence of -OH or -NH_2 (>NH) groups in the composition of the molecule. Absorption bands caused by valence vibrations of the hydroxyl group are observed in the range of 3200-3600 cm^{-1} of the spectrum.

As can be seen from the above picture, the absorption bands caused by the valence vibrations of the ON- group belonging to the obtained sorbent polysilicon acid show a wide absorption in the spectrum of 3188.33 cm-1. These bands are distinguished from other bands by their greater width and intensity. As a result, the broad absorption band belonging to the –OH group closes the absorption peaks belonging to the –NH_2 groups. In addition, the doublet of absorption peaks at 2358.94 cm^{-1} and adjacent to it are bands caused by asymmetric and symmetric valence vibrations of the -NH_2 functional group of thiourea. At the same time, the absorption peaks at 1606.70 cm^{-1} and -1504.48 cm^{-1} are scissor-shaped of the -NH_2 functional group, and the wave absorptions at 667.37 cm^{-1} show these peaks due to the valence vibrations of the -NH_2 group.

The frequency of vibration related to the C-O group appearing in the field of 1247.94 cm^{-1} allows us to assume that ammonia is released and silicon-oxygen and oxygen-carbon bonds are formed in the resulting sorbent. As a result of valence vibrations belonging to the Si-O-Si group, the 1132.21 cm^{-1} wave shows its intense, characteristic peak in the absorption region. A theoretical conclusion can be made that the thio-group belonging to thiourea is preserved in the sorbent and that the obtained sorbent is selective for some metals, as the vibration frequency of the S=S group appears in the region of 1022.27 cm^{-1}.

Based on the IR-spectroscopic analysis data, theoretical calculation books and comparisons with the IR-spectroscopic data of the original reagent, the reaction mechanism in obtaining the synthesized sorbent can be realized as follows and the approximate formula of the sorbent can be proposed as follows.



In order to study the physicochemical properties of the obtained sorbent and conduct research on the thermal stability of the resulting sorbent, at the same time, to clarify the approximate formula of the new sorbent, thermal analyzes of this sorbent were carried out. The network gravimetric analysis data of the new sorbent are presented in Figure 3.

The thermal analysis of the sorbent (4-c) obtained based on thiourea was studied. A sample of 7,765 mg was taken for the thermal analysis of the organohybrid sorbent synthesized based on thiourea, and the process was studied in the temperature range of 20-800°C. Thermal analyzes were carried out in an atmosphere of argon inert gas. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of the synthesized sorbent are presented in Figure 3.

In the derivatogramma of the sorbent based on thiourea, two endothermic effects were observed at temperatures of 218.97-263.26 ^{0}C and two exothermic effects at temperatures of 191.49 ^{0}C and 333.44 ^{0}C. The analysis of the thermogravimetric curve of the sorbent synthesized on the basis of thiourea and sodium metasilicate shows that the TGA curve mainly takes place in the temperature range of 3 intensive mass losses.

The 1st mass loss range corresponds to the temperature of 19.21-171.93 °C, the 2nd mass loss range corresponds to the temperature of 171.93 - 333.44 °C, and the 3rd mass loss range corresponds to the temperature of 333.44 - 801.80 °C. The analysis shows that mass loss in the 1st mass loss interval is 0.375 mg, i.e. 4.97%, due to the moisture retained on the surface of the sorbent in this temperature range, an intensive decomposition process occurs in the 2nd mass loss interval.

The main amount of mass loss in this range is 5.076 mg, i.e. 67.098%. Based on the literature analysis, it can be said that the mass loss in this temperature range is due to the decomposition of amino- and thio-groups in the sorbent and as a result of the separation of the organic part from the sorbent. 3 - uniform mass loss in the range of mass loss, i.e. from 400 oS to 800 oS, is 1.206 mg, i.e. 15.942%. The analysis of the TGA and DTA curve results of the organohybrid sorbent is tabulated in Table 1.

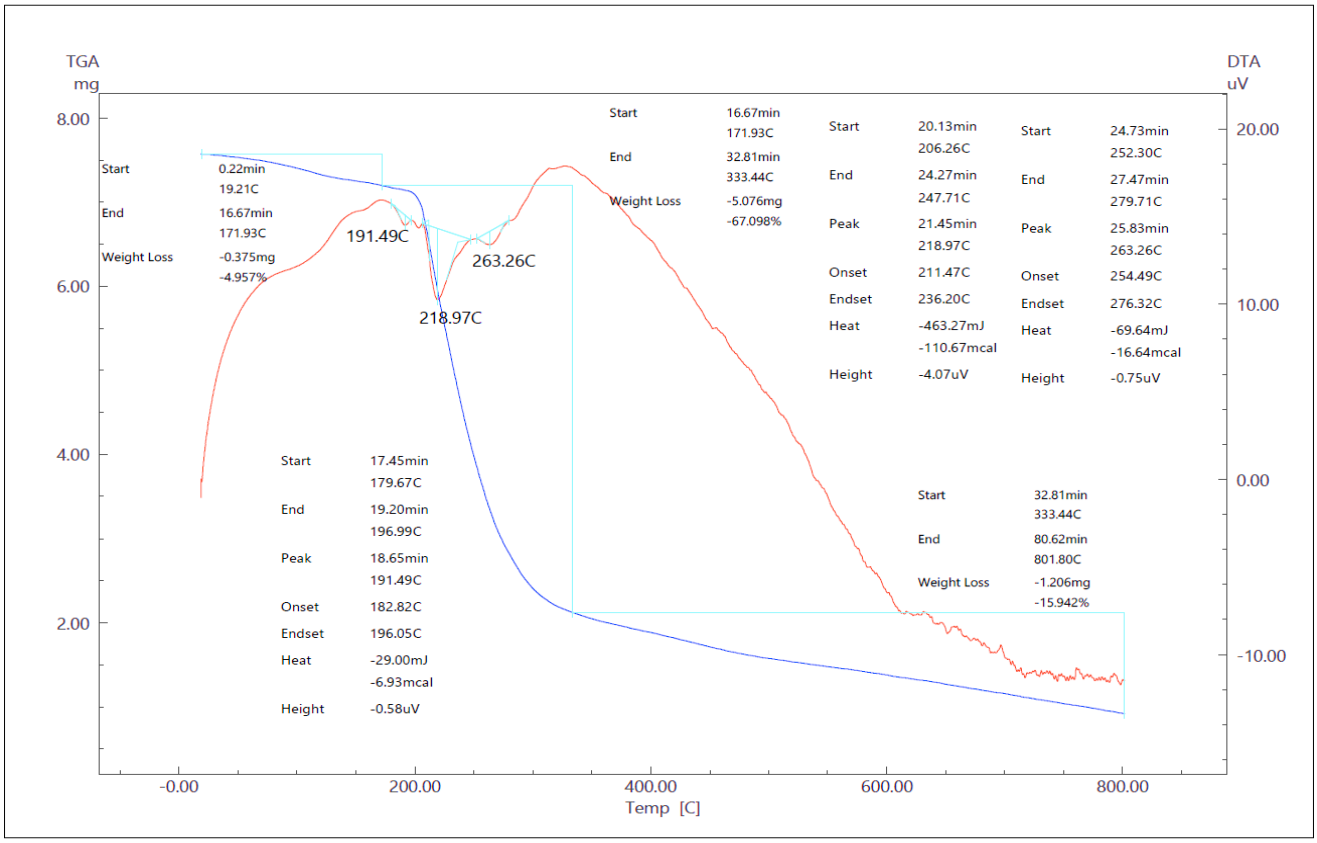


Fig. 3 Thermogravimetric (TGA) and differential thermal analyzes (DTA) dervotogramma of the sorbent were obtained based on thiourea

Table 1. Analysis of TGA and DTA curve results of sorbent

No	Temperature, °C	Lost mass, mg (7,565)	Mass lost, %	The amount of energy consumed (µV*s/mg)	Spent time (min)	dw(mg)	dw/dt (mg/min)
1.	100	0.165	2,18	12,149	9.41	7.40	0.017
2.	200	0.485	6,245	14.594	19.5	7.08	0.024
3.	300	5.165	13,2	16.693	29.4	2.40	0.175
4.	400	5.687	75.17	13.621	39.5	1.878	0.143
5.	500	5,997	79.27	4,750	49.7	1.568	0.120
6.	600	6.192	81,8	-6,473	59.9	1.373	0.103
7.	700	6.414	84,7	-10.11	70.1	1.151	0.091
8.	800	6.65	87,9	-11.44	80.4	0.915	0.082

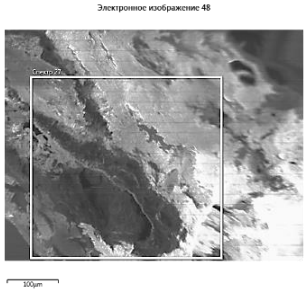
Scanning electron microscopic (SEM) analyses of the sorbent obtained based on thiourea and sodium metasilicate were carried out. A 100µm image of the analyzed sorbent was taken, and the vibration waves generated as a result of passing high-frequency waves from its parts in several areas were electronically analyzed with the help of very high-sensitivity sensors. Basic analyzes were carried out in a state-of-the-art SEM facility located in the High-Tech Center. The data obtained in SEM are presented in Figure 4 and the accompanying tables.

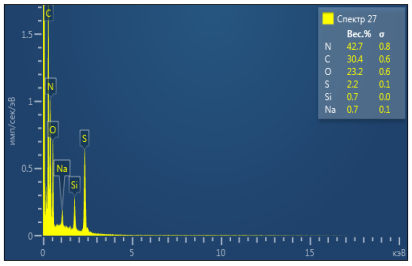
It can be seen from Figure 4 that there are pores in the structure of the obtained sorbent and that these pores are different in different areas of the sorbent. As a result of the analysis, the cases where the sigma mass percentages of the elements are lower than 1 indicate a high probability that the detectors belong to the waves associated with this particular element.

In addition, based on SEM images, analysis results, and calculation books, it can be seen that the bonds between thiourea and the silicate base were formed with different levels of order during the synthesis process.

As a result, it can be assumed that the structure of the resulting organohybrid sorbent is more complex. However, the analyzes provided more confidence in the presence of functional groups in the synthesized sorbent, indicating that the above-proposed formula is correct.

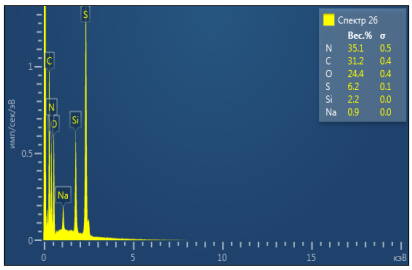
1.





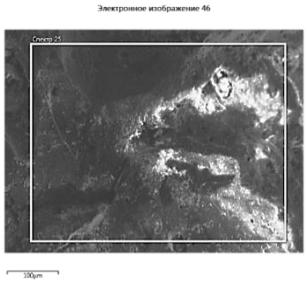
Element	Weight.%	Sigma weight.%
C	30.40	0.58
N	42.71	0.83
O	23.24	0.65
Na	0.67	0.06
Si	0.74	0.04
S	2.24	0.06
Total:	100.00	

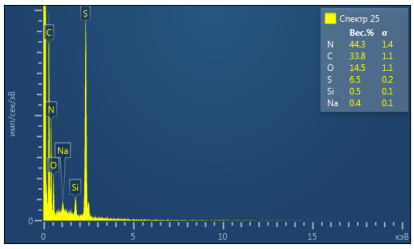
2.



Element	Weight.%	Sigma weight.%
C	31.15	0.38
N	35.14	0.52
O	24.42	0.37
Na	0.95	0.04
Si	2.19	0.04
S	6.15	0.07
Total:	100.00	

3.





Element	Weight.%	Sigma weight.%
C	33.82	1.13
N	44.33	1.40
O	14.48	1.06
Na	0.39	0.11
Si	0.52	0.06
S	6.46	0.22
Total:	100.00	

Fig. 4 SEM analysis results of sorbent based on sodium metasilicate and thiourea

4. Conclusion

It can be said that a sorbent with selective properties was synthesized based on sodium metasilicate and thiourea. The IR-spectroscopic analysis of the synthesized sorbent was carried out, and the approximate and theoretical formula of the sorbent obtained as a result of the analysis of the functional groups belonging to the synthesized sorbent was proposed. As a result of the thermal analysis of the sorbent, the approximate formula of the sorbent was clarified, the decomposition of functional groups related to thiourea in the sorbent was calculated, and it was confirmed that the sorbent can be used in high-temperature solutions. Electron microscopic and elemental analyzes of the synthesized sorbent in SEM showed the presence of nitrogen and sulfur in the composition, and the approximate formula of the synthesized product was proposed with more confidence.

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