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SYNTHESIS AND RESEARCH METHODS OF SULFUR-CONTAINING SORBENTS

Abstract: This article consists of 6 pages and includes raw materials and research methods, synthesis of sulfur-containing sorbent ligands, polymer sorbent synthesis, obtaining coordination compounds of Ni (II) and Fe (II) ions with synthesized polymer ligands. consists of. The article provides information on the method of synthesis of several sorbents containing sulfur and the sorption of metal ions.

Key words: EXG – epichlorohydrin, F – formaldehyde, K – urea, IQ – infrared, SAS - static exchange capacity.

Language: English

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Introduction

Reagents of "t" and "kt" brands were used in the research. Solutions of reagents were prepared by dissolving their exact weights in a certain volume of solvent.

Potentiometric titration was performed on a pH-meter OP-211/1 and a universal ionomer EV-74. In this case, a decreasing portion of 0.11 N NaCl solution and an increasing portion of 0.11 N HCl solution were added to the same (0.25 g) weight of OH- and H-form ionite, bringing the volume of solution in each flask to 25.1 ml. tartar solutions were left for 8 days. Potentiometric titration of the solutions was then performed, and a potentiometric titration curve of the dependence of the pH of the equilibrium solution on

the volume of acid added for titration was constructed and described.

The IR absorption spectra of the starting materials, sorbents and their coordination compounds with metals were expressed and recorded on an IRTracer-100 IK-Fure spectrometer in an area of 400-4000 cm⁻¹.

Thermal stability of polymer ligands and their coordination compounds with metals by differential-thermal and thermogravimetric methods on Paulik-Erdey system derivatograph at a speed of 10 degrees / min, T-900, TG-200, DTA - 1/10, DTG - 1/10 galvanometer sensitivity, was studied by automatically recording the derivatogram on photographic paper. A sample of 60 to 80 mg was

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placed in a 10 mm diameter platinum crucible without a lid. Aluminum oxide was used as a standard. The dynamic heating mode was carried out in atmospheric conditions.

Netzsch Simultaneous Analyzer STA 409 PG (Germany) with K-type thermocouple (Low RG Silver) and aluminum crucible was also studied on a differential scanning calorimeter at a flow rate of 50 ml / min in an inert nitrogen atmosphere (Institute of Bioorganic Chemistry, Academy of Sciences of Uzbekistan). Measurement temperature range 20-390°C, heating rate 5K / min. The sample size is 5-10 mg. The measurement system was calibrated with standard sets KNO₃, In, Bi, Sn, Zn, Mg.

The method of quantitative and qualitative radiographic analysis of compounds was carried out on a powder diffractometer XRD-6100 (Shimadzu, Japan) to determine the crystalline properties of geological deposits. CrKa was performed under the influence of radiation (b-filter, Ni, $\lambda = 1.54178\text{\AA}$, current and voltage in the X-ray tube 30 mA, 30 kV). The constant rotation speed of the detector was 4 degrees / min, in 0.02° steps ($\delta / 2\text{th-coupling}$), and the scanning angle was from 4° to 80°. The samples were analyzed in a rotating chamber with a rotational speed of 30 rpm.

The morphology and element composition of polymer ligands and their coordination compounds were determined and analyzed using SEM-scanning electron microscopy at the High Technology Training and Experimental Center.

0.1 N. solutions of Ni, Fe elements "k.t." The exact masses of the soluble salts of the brand were dissolved in distilled water and nitric acid solution was added until the pH = 1-2.1. Concentrations of the prepared solutions were standardized using a complexometric titration method.

An aqueous solution of 4- (2-pyridylazoe) resorcinol (PAR) was used as a reagent in the photometric determination of Ni and Fe elements.

The desired medium in the solution was set using KCl-HCl [155] for pH = 1-3.1 and CH₃COOH-NH₄OH buffer solutions for pH = 3-11. The pH of the solutions was monitored at ± 0.05 pH using a universal ionometer EV-74 and an OP-211/1 pH meter. The solutions were stirred and tested in MM-5 type magnetic stirrers.

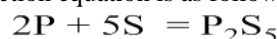
Concentrations of elements in the analyzed solutions were determined using optical analysis methods SF-46 spectrophotometer and KFK-2MP

photometer. Ni (0.05 n, pH = 3.5-3.8), Fe (0.1 n, pH = 6.5) and sulfate solutions were used to determine the exchange capacity of ligands for metal ions. All measurements were performed under static and standard conditions. The amount of metal cations in the initial and equilibrium solutions was determined using trilonometric titration, photocolometry (Fe²⁺, Ni²⁺) and atomic absorption spectrometry (Ag⁺). The duration of contact time of solutions with polymer ligands was 1 day, and the pH of the solution was determined potentiometrically.

Synthesis of sulfur-containing sorbent ligands.

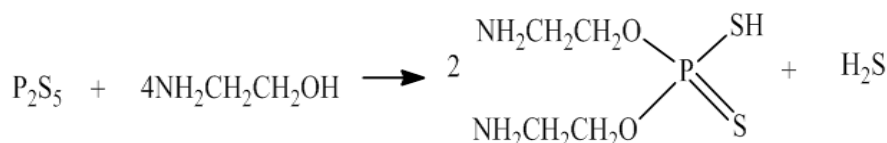
Extraction of phosphorus (V) sulfide. 1.51 g (0.05 mol) of powdered red phosphorus was mixed with 4 g (0.125 mol) of sulfur powder. This mixture was placed in a test tube made of hard liquefied glass and heated under a weak stream of dry carbon monoxide. The temperature was slowly increased until a homogeneous liquid was formed (300-350°C). The test tube was then cooled to form a yellow-green crystalline substance. The resulting substance was separated from the test tube. Product 5.38 g, reaction yield 98%.

The reaction equation is as follows.



i (2-aminoethyl) dithiophosphate ether synthesis. Heat to 0,11 mol or 22,2 g of phosphorus (V) sulfide with 0,2 mol or 12,2 g of monoethanolamine. The reaction is carried out in a heat-resistant vessel. When these two substances are mixed, the reaction begins rapidly. The reaction produces a thick smoke-like white gas, which is hydrogen sulfide gas with a very pungent odor, after which the gas slowly begins to separate from the vessel. After the gas is completely drained, we can see that a black viscous liquid has formed in the vessel. Heat the resulting black liquid in a water bath at a temperature of 50-60°C, continue heating until the separation of the black liquid from the container is complete. Then use a glass tube to mix the substance in the container, if there is an insoluble sediment at the bottom, add 2-3 ml of monoethanolamine and continue the process, ie heating. Continue heating until the sediment and gas separation at the bottom is complete. When the sediment melts in the bottom of the pot, stop heating. Before use, the sorbent is cooled.

The reaction equation is as follows.



Polymer sorbent synthesis. 4 g of powder from Zn (II) O, O-di- (2-aminoethyl) dithiophosphate

complex was placed in a round-bottomed flask with a return cooler, a thermometer and an automatic stirrer.

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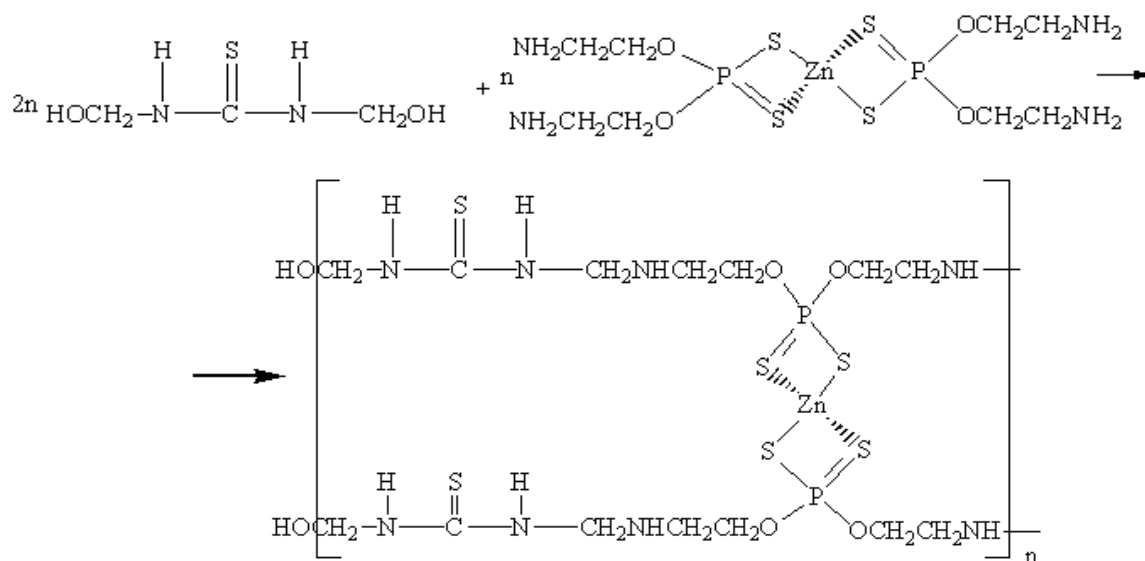
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In a separate container, take 6 grams (0.2 mol) of formalin (CH₂O) and 15.2 grams (0.2 mol) of thiourea CS (NH₂)₂ and mix at 35-40°C until a homogeneous mixture is formed. The mixture was then added dropwise to the Zn (II) O, O-di- (2-aminoethyl) dithiophosphate product in the flask. The mixture was heated at 80-90°C for 4 hours, stirring, until a solid resinous substance was formed. The resulting solid resin mixture was poured into a porcelain bowl and dried in a drying oven at a temperature of 50-60°C for 24 hours. The dried polymer was crushed and the mixture was first washed with a 3% aqueous solution

of KOH and then with distilled water and air-dried until it reacted neutrally with phenolphthalein. The resulting ionite solid white compound after drying. Air-dried ionite mass 21 grams reaction yield 83%. If the temperature is normal.

The synthesized complex sorbent is partially soluble in water, insoluble in organic solvents and does not suffocate.

The reaction equation for the production of ionite based on thiourea, formaldehyde, Zn (II) O, O-di- (2-aminoethyl) dithiophosphate is as follows



Obtaining coordination compounds of Ni (II) and Fe (II) ions with synthesized polymer ligands

Coordination compounds of Ni (II) and Fe (II) ions with the synthesized sorbent are 0.1 n of water-soluble sulfate and nitrate salts of the corresponding metals. To 55 ml of solution (Fe (II) ion is a very rare ion) add 1 g of sorbent and stir for 2 hours. The resulting black liquid forms a black precipitate with Ni (II) salts, and with Fe (II) forms a light orange

precipitate, which is filtered and dried. Drying is carried out at a temperature of 500°C. The results of elemental analysis of the composition of the obtained coordination compounds and some physicochemical properties are given in Table 2.1 below. A photograph of the obtained ligands and their coordination compounds was taken under a scanned electron microscope and the amount of elements in them was determined.

Table 1. Elemental analysis and some physical properties of the obtained coordination compounds

Metall	color	t _{liquid} , °C	Found, %			Gross formula	Calculated, %		
			C	H	Me		C	H	M
[ML ³] ₂ ; [AgL ³]									
Ni (II)	dark black	174	32,8	5,27	8,78	(C ₁₀ H ₂₀ N ₂ O ₄ PS ₂) ₂ Ni	33,44	5,61	8,85
Fe (II)	light yellow	168	32,53	5,09	8,45	(C ₁₀ H ₂₀ N ₂ O ₄ PS ₂) ₂ Fe	33,36	5,6	9,08

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Cd (II)	yellow	163	31,26	5,12	14,56	(C ₁₀ H ₂₀ N ₂ O ₄ PS ₂) ₂ Cd	31,31	5,26	14,65
Ag (I)	black	192	27,35	4,56	23,73	C ₁₀ H ₂₀ N ₂ O ₄ PS ₂ Ag	27,6	4,63	24,78
[ML ⁴] ₂ ; [AgL ⁴]									
Ni (II)	dark blue	165	25,92	4,04	11,15	(C ₆ H ₁₂ N ₂ O ₂ PS ₂) ₂ Ni	26,59	4,46	11,72
Fe (II)	light yellow	142	25,88	4,25	11,46	(C ₆ H ₁₂ N ₂ O ₂ PS ₂) ₂ Fe	26,5	4,45	12,02
Cd (II)	yellow	155	24,28	3,87	18,96	(C ₆ H ₁₂ N ₂ O ₂ PS ₂) ₂ Cd	24,39	4,09	19,02
Ag (I)	black	176	20,49	3,55	30,78	C ₆ H ₁₂ N ₂ O ₂ PS ₂ Ag	20,76	3,48	31,07

CONCLUSION

The article focuses on the synthesis of starting materials and research methods, sorbents that form complex compounds with sulfur-containing metals, and describes in detail the research methods used in the analysis of starting materials and synthesized substances. Also:

1. Extraction of phosphorus (V) sulfide.
2. Synthesis of a sorbent that forms a complex from the interaction of phosphorus (V) sulfide and monoethanolamine;

Topics such as are also covered

Coordination compounds of the synthesized complex-forming sorbent ligands with Ni (II) and Fe (II) ions are obtained, and acid-base ionization constants of sorbents, stability constants of complex compounds of metal ions with sorbent ligands are given.

References:

1. Antokolskaya, I.I., Bolshakova, L.I., Myasoedova, G.V., Kolesov, G.M., Kravsova, R.P., Savin, S.B., Shvoeva, O.P., & Sherbinina N.I. (1992). Avtorskoe svidetelstvo № SU 1086756 A1, kl. C 08 G 8/16, C 08 J 5/20, 1992, Sposob polucheniya kompleksobrazuyushchego ionita.
2. Wołowicz, A., & Hubicki, Z. (2014). Adsorption characteristics of noble metals on the strongly basic anion exchanger Purolite A-400TL. *J. Mater Sci.*, V.49, pp.6191–6202
3. Sottikulov, E.S., Beknazarov, X.S., & Eshkurbanov, F.B. (2017). Synthesis and research of sorption properties of modified orthochemical acids with ethanolamines. *Composite materials*, №1, pp. 79-81.
4. Kulyuxin, S. A., Gorbacheva, M. P., Krasavina, Ye. P., Mizina, L. V., Rumer, I. A., & Konovalova, N. A. (2011). Extraction of 137Cs, 90Sr, 90Y and ion d-elements from water solutions with the help of sorbents, containing triethylenediamine. *Radiochemistry*, T.53, -№ 3, pp. 252-256.
5. Paxnutova, Ye. A., & Slijov, Yu. G. (2013). Synthesis and research of structural and chromatographic characteristics of xelatsoderjashchix sorbents on the basis of silica. *J. prikl. ximii*, T. 86, № 4, pp. 567-572.
6. Wang, Z., Yin, P., Qu, R., & Xu, Q. (2012). Heterogeneous synthesis of chelating resin organophosphonic acid-functionalized silica gel and its adsorption property of heavy metal ions from fuel ethanol solutions. *J. Appl. Polym. Sci.*, -V.126, -№ 2, pp. 544-551.
7. Gao, B., Jiang, G., & An, F. (2012). Preparation of iminodiacetic acid-type composite chelating material IDAA-PGMA / SiO [2] and preliminary studies on adsorption behavior of heavy metal ions and rare earth ions. *J. Appl. Polym. Sci.*, - V.125, -№ 4, pp. 2529-2538.
8. Zub, Yu.L. (2010). *Novye mesoporistye sorbenty s kompleksobrazuyushchim poverxnostnym*

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- sloem. Materialy III mezhdunarodnyy symposium po sorbsii i ekstraktsii, (pp.67-84). Vladivostok, September 20-24, 2010.*
- Skopenko, V.V., Trofimchuk, A.F., Bruskina, A.M., Simonova, L.N., & Tryashin, A.S. (1990). *Author's certificate № SU 1590096 AI, kl. B 01 D 15/08, 1990, The ability to obtain sorbents for the extraction of ionic metals from solutions.*
 - Yuferova, I.B., Fadeeva, V.I., Tikhomirova, T.I., Kudryavsev, G.V., & Lisichkin, G.V. (1988). *Author's certificate № SU 1437776 AI, kl. G 01 N 30/48, B 01 J 20/10, 1988, Help for the extraction of ionic metals from rasvorov and chromatography.*