GALAXY INTERNATIONAL INTERDISCIPLINARY RESEARCH JOURNAL (GIIRJ)
ISSN (E): 2347-6915
Vol. 9, Issue 9, Sep. (2021)

DETERMINATION OF THERMAL STABILITY AND SURFACE MORPHOLOGY OF THE OBTAINED IMMOBILIZED LIGAND

Kasimov Sherzod Abduzairovich,
Associate Professor at the Department of Inorganic and Analytical Chemistry, Termez State University, 190111, Republic of Uzbekistan, Termez, Barkamol Avlod str., 43.
E-mail: sh_kasimov@rambler.ru

Turaev Khayit Khudainazarovich,
Doctor of Chemistry, Professor, Dean of the Faculty of Chemistry, Termez State University, 190111, Republic of Uzbekistan, Termez, st. Barkamol Avlod, 43.
E-mail: hhturaev@rambler.ru

Djalilov Abdulakhat Turapovich,
Academician of the Academy of Sciences of the Republic of Uzbekistan, Professor, Doctor of Chemical Sciences, Director of the Tashkent Research Institute of Chemical Technology, Republic of Uzbekistan, 111116, Tashkent region, Zangiotinsky district, p / o Ibrat.
E-mail: gup_tniixt@mail.ru

Umbarov Ibragim Amonovich,
Doctor of Technical Sciences, Associate Professor, Dean of the Faculty of Termez State University, 190111, Republic of Uzbekistan, Termez, st. Barkamol Avlod, 43.
E-mail: i_umbarov@mail.ru

ANNOTATION

The article studies the thermal stability and surface morphology of the immobilized ligand obtained on the basis of in situ covalent attachment of O, O-di- (2-aminoethyl) -dithiophosphate potassium on a urea-formaldehyde matrix. As a result of the conducted thermal studies, it was determined that the resulting ligand is stable up to 128 ° C, and also has a high sorption capacity for non-ferrous metals due to its microporous surface structure.

Keywords: covalent immobilized ligand, O, O-di- (2-aminoethyl) -dithiophosphate potassium, thermal analysis, scanning electron microscopy, surface morphology.

INTRODUCTION

The synthesis of new covalently immobilized ligands, the separation with their help of transition metals from solutions by complex-forming sorption methods, the study of the composition, structure, physicochemical properties of coordination compounds formed in the process of sorption is one of the main tasks of inorganic chemistry.

Earlier in the literature it was shown that a complexing polyfunctional polymer sorbent based on polycondensation of urea, formaldehyde, phosphoric acid [1, 2] was synthesized; a chelating sorbent based on urea, formaldehyde and dithizone was also synthesized and investigated [3]. Immobilized metal complexes of some d and f elements with hetarylformazans were synthesized

[4], sorption characteristics were determined for Co (II), Cd (II), Ni (II), Cu (II), and Zn (II) ions on silica gel with covalently immobilized 1 - (2-pyridylazo) -2-naphthol [5], carried out the sorption-photometric determination of cobalt ions using the immobilized reagent 4-amyl-2-nitroso-1-naphthol [6]. A sorbent for the concentration of lanthanum from large-volume water samples has been proposed. The sorbent is stable under dynamic conditions and is based on hypercrosslinked polystyrene modified with 1-phenyl-3-methyl-4-benzoylpyrazol-5-one [7].

Chelating immobilized ligands are widely used for the concentration and separation of trace elements. Further development of their application is associated with high selectivity and effective effect of concentration of ions of elements from solutions of complex chemical composition. The use of immobilized ligands with high selectivity towards non-ferrous metal ions is one of the promising directions in the practice of wastewater treatment.

The aim of the study is to determine the thermal stability and surface morphology of the immobilized ligand obtained on the basis of in situ covalent attachment of O,O-di- (2-aminoethyl) -dithiophosphate potassium on a urea-formaldehyde matrix.

EXPERIMENTAL PART

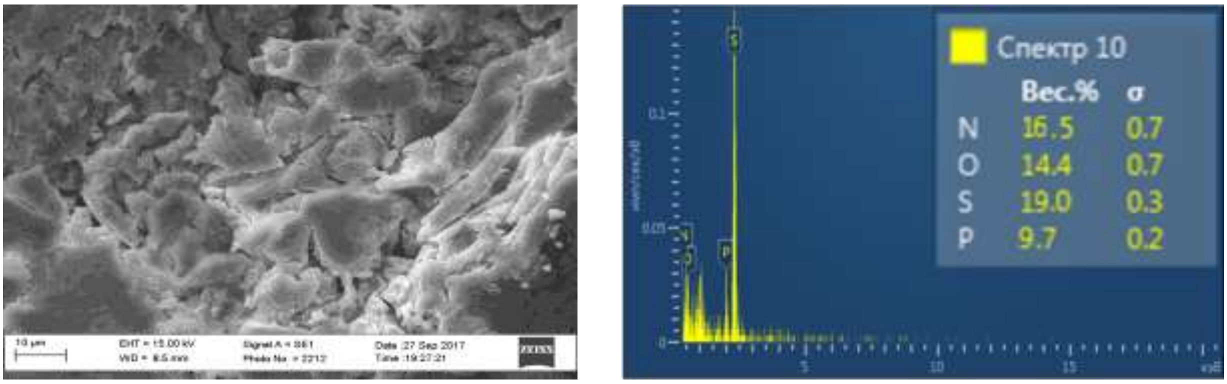
The study of the phase morphology and the change in the structure of the obtained ligand, depending on the preparation conditions and the type of additives, was carried out using a scanning electron microscope from Jeol Interactive Corporation, Japan JSM-6460LA. To decipher the images, literature sources were used, as well as microprobe data on an Oxford X-ray microanalyzer, which makes it possible to determine the elemental composition of the ion exchanger phases with an accuracy of 0.5%. The scanning electron microscopy image was taken at the Center for Advanced Technologies under the Ministry of Innovative Development of the Republic of Uzbekistan.

The thermal stability of polymer ligands and their coordination compounds with metals was determined by differential thermal and thermogravimetric methods on a Paulik-Paulik-Erdey derivatograph at a rate of 10 deg / min, T-900, TG-200, DTA - 1/10, DTG - Galvanometer sensitivity 1/10. A sample weighing 60–80 mg was placed in a platinum crucible without a lid 10 mm in diameter. Aluminum oxide was used as a standard. The dynamic heating mode was carried out in atmospheric conditions (Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan).

RESULTS AND ITS DISCUSSION

The process of covalent immobilization of O, O-di- (2-aminoethyl) -dithiophosphate potassium with urea-formaldehyde resin was carried out in molar ratios 1: 2 of the starting materials at a temperature of 90 ° C, the reaction time was 1 hour. The resulting reaction product with a yield of 93% is a resin a figurative mass of yellow color.

Using scanning electron microscopy (SEM) of the synthesized polymer ligand (photography, elemental analysis), the microscopic structure was studied. As can be seen in the photograph taken at 100 μm, the resulting immobilized ligand has a microporous structure. This indicates that the resulting ligand has a fairly high sorption capacity (Fig. 1).



(a) (b)

Rice. 1. Scanning electron microscopic image of the surface (a) and the results of elemental analysis (b) of the ligand

Based on the literature data [8] to study the thermal stability of polymer ligands and according to the results of derivatographic analysis, various exothermic and endothermic effects observed when changing the mass as a result of the destruction of the structure of compounds upon heating the ligand were analyzed (Fig. 2, table).

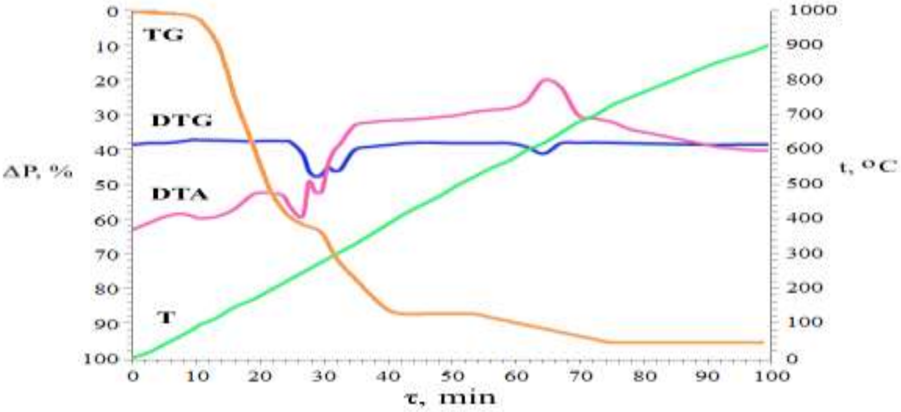


Fig. 2. Derivatogram of the immobilized sulfur-containing ligand.

Table Derivatographic data of ligand thermolysis

Temperature range of thermolysis, °C	Peak effect, °C	Mass change, %	Total mass change, %	The nature of the effect
60-136	128	15,7	15,7	endothermic
143-187	170	22	37,5	endothermic
190-205	200	15,4	52,9	exothermic
204-250	212	2,9	55,8	endothermic
300-340	330	19,2	75	endothermic
340-360	348	3,6	78,6	endothermic
360-400	380	3,7	82,3	endothermic
400-430	430	1,8	84,1	exothermic
450-500	482	1,1	85,2	exothermic
570-605	595	2,8	88	exothermic
605-625	613	1	89	exothermic
738-805	780	4	93	exothermic

In the derivatogram of the ligand shown in Figure 2, six endothermic effects were observed at 128, 170, 212, 330, 348, 380 °C and six exothermic effects at 200, 430, 482, 595, 613, 780 ° C. The first endothermic effect at 128 °C was associated with the release of hygroscopic water containing the ligand, with a weight reduction of 15.7%. Endo effects at 170 and 212 °C, as well as exo effects at 200 °C, by their nature participate in the protonation of amino groups of the ligand and are associated with the release of water, ammonia and hydrogen sulfide from hydrodithiophosphate groups. At these temperatures, the overall weight loss of the ligand is 55.8%. The decrease in the total mass of the ligand in the temperature range 60–900 °C was 93.59%.

CONCLUSIONS

Thus, the resulting immobilized ligand, based on in situ covalent attachment of O, O-di- (2-aminoethyl) -dithiophosphate potassium on a urea-formaldehyde matrix, is stable up to 128 ° C, also has a high sorption capacity for nonferrous metals due to its microporous surface structure.

REFERENCES

1) Kasimov SH.A., Turayev KH.KH., Dzhalilov A.T. Issledovaniye protsessa kompleksoobrazovaniya ionov nekotorykh dvukhvalentnykh 3d-metallov s khelatoobrazuyushchim sorbentom // Universum: Khimiya i biologiya: elektron. nauchn. zhurn. -2018. -№3 (45).
2) Kasimov SH.A., Turayev KH.KH., Dzhalilov A.T., Choriyeva N.B., Amonova N.D. IK spektroskopicheskiye issledovaniye i kvantovo-khimicheskiye kharakteristiki azot i fosforsoderzhashchego polimernogo liganda // Universum: Khimiya i biologiya: elektron. nauchn. zhurn. 2019. № 6(60). URL: http://7universum.com/ru/nature/archive/item/7400
3) Chorieva N., Ermuratova N., Turaev Kh., Kasimov Sh. (2021) "Synthesis and research of chelate forming sorbent based on carbamide, formaldehyde, ditizone //Chemistry and Chemical Engineering: Vol. 2020 : No. 4 , Article 4. DOI: 10.51348/RWHC6586
4) Pervova I.G. Immobilized metal complexes of some d and f elements with hetarylformazans: synthesis, structure and properties: dis. Doctors of Chemistry: 02.00. 02 / Pervova Inna Gennadievna // Ekaterinburg .–2006 .–288 p. - 2007.
5) Openko V. V. et al. Study of sorption of Co (II), Cd (II), Ni (II), Cu (II) and Zn (II) on silica gel with covalently immobilized 1- (2-pyridylazo) - 2-naphthol // News of higher educational institutions. Chemistry and chemical technology. - 2014. - T. 57. - No. 10. - S. 57-61.
6) Inatova, M. S., Smanova, Z. A., Nurmukhamadov, Zh., Gafurov, A. A. Sorption-photometric determination of cobalt ions using an immobilized reagent 4-amyl-2-nitroso-1-naphthol // European research. - 2016. - No. 8 (19).
7) Arkhipova A. A. et al. Sorbents with non-covalently immobilized β-diketones for preconcentration of rare earth elements //Talanta. – 2016. – T. 161. – C. 497-502.
8) Kopylova V.D., Portnykh N.V., Karimova L.V., Shabanova L.N. Issledovaniye termostoykosti kompleksnykh form ionitov.// Zhurn.prikl.khimii. -1979. -№3. -S.533-537.