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STUDY OF THE MORPHOLOGY AND THERMOSTABILITY OF POLYAMPHOLYTE PRODUCED ON THE BASIS OF AMINOACETIC ACID IMMOBILIZATION IN EPOXY MATRIX

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ANNOTATION

The purpose of this study is the synthesis and study of a covalently immobilized chelate-forming ligand for the extraction of non-ferrous and noble metal ions. To achieve this goal, a nitrogen-containing immobilized ligand based on aminoacetic acid, epoxy resin, and polyethylene polyamine was synthesized and studied. The article studies the thermal stability and surface morphology of an immobilized ligand obtained on the basis of covalent fixation of aminoacetic acid on an epoxy matrix. As a result of thermal studies, it was found that the resulting ligand is stable up to 195°C. The microscopic structure and elemental analysis of the ligand and its coordination compound with Cu^{2+} ions were studied using a scanning electron microscope. The microstructure of the surface of the obtained ligand was measured at the level of 100 μm; it was found that it is porous and has high sorption properties.

Keywords: covalent immobilized ligand, aminoacetic acid, microstructure, thermal stability, scanning electron microscopy.

INTRODUCTION

Ion-exchange polyion exchangers are used in all areas of industry. They are widely used in thermal and nuclear power plants for water softening, in chemical plants for water

desalination, hydrometallurgy to isolate rare, valuable and heavy metals from process solutions, as well as to reduce environmental damage, to clean wastewater from poisonous ions (1). The nature of the interaction is determined by the nature of the functional groups, the metal ion, and depends on the sorption conditions. Polyampholytes with nitrogen and oxygen-containing functional groups (PEARg, PABA-EP) were used as sorbents [2, 3]. Polyampholytes are polymeric substances that simultaneously contain both anionic and cationic groups in their composition, which, depending on the pH of the medium, can adsorb cations and anions. Ions of heavy metals such as nickel, cobalt, zinc, chromium, copper, lead, cadmium cause serious environmental problems for animals, plants and humans due to their extreme toxicity [4]. Heavy metals are generally toxic in nature and are not easily degraded by simple biological treatment, like other organic materials [5,6].]. The removal of these contaminants from discharged water is a necessary step in wastewater treatment processes [7]. Extensive work is being done in this area to purify toxic metals using a range of advanced technologies such as polymer-enhanced ultrafiltration [8], flocculation [9], adsorption [10-12], ion exchange [13], and various membrane separation technologies [14.15]. Of great interest is the adsorption process, a surface phenomenon in which a multicomponent liquid (gas or liquid) mixture is attracted to the surface of a solid adsorbent and forms bonds through physical or chemical bonds, the most effective, promising and widely used fundamental approach in wastewater treatment processes is recognized [16], mainly depends on its simplicity, economically viable, technically feasible and socially acceptable [17]. There are also many theories for evaluating the ion exchange process [18,19]. In the present work [20], the use of a new environmentally friendly aminopolycarboxylate chelating agent of the trisodium salt methyl glycine diacetic acid (MGDA) for the inactivation of various metal ions by complexation in microporous anion exchangers was considered, and the sorbents were tested. And in this review [21], the modern de-development in the field of application of biosorbents for water purification is critically assessed with an emphasis on adsorption. Earlier in the literature, a complexing polyfunctional polymer sorbent based on the polycondensation of urea, formaldehyde, phosphoric acid [22, 23] was synthesized; and polyethylenepolyamine [25], the physicochemical properties of a chelating polymeric sorbent synthesized on the basis of epoxy resin, glycine and polyethylenepolyamine (EGP-1) [26] were also studied.

METHODOLOGY

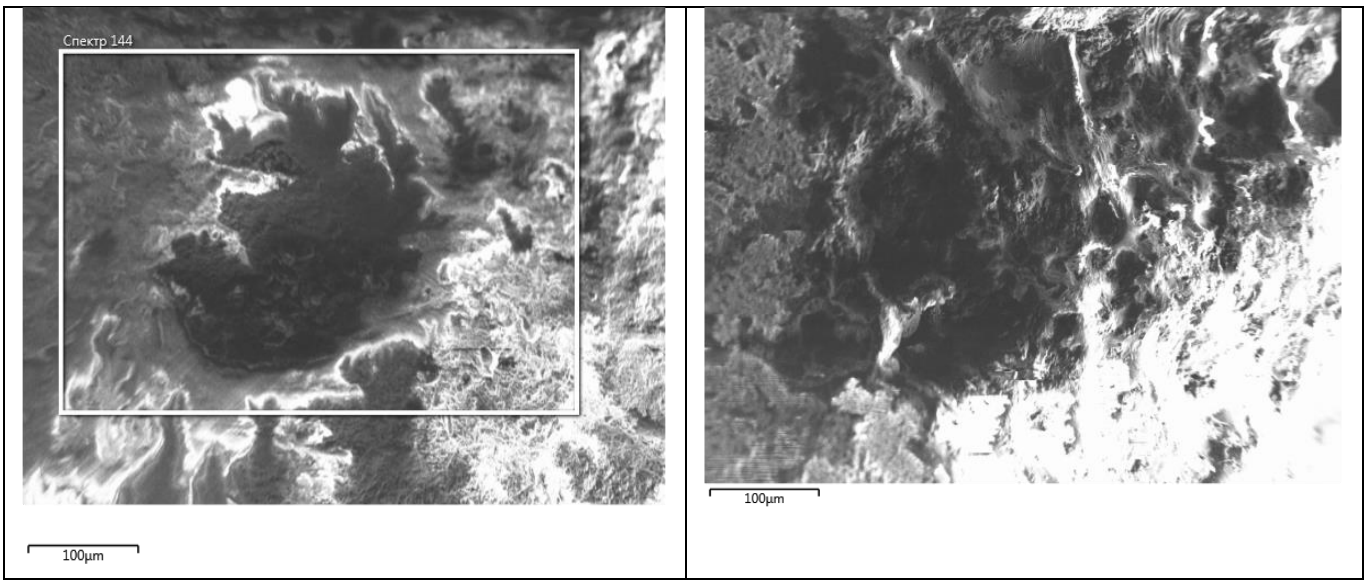
Morphological studies of the surface of the samples were carried out using a scanning electron microscope SEM - EVO MA 10 (Zeiss, Germany). During the measurement, an accelerating voltage (EHT - Extra High Tension) of 20.00 kV was applied, the working distance (WD-working distance) was 8.5 mm. The measurement was carried out in the mode of detecting secondary electrons (SE1-secondary electrons detector). The image was acquired at various scales using the SmartSEM software.

Thermoanalytical studies were carried out on a Netzsch Simultaneous Analyzer STA 409 PG instrument (Germany), with a K-type thermocouple (Low RG Silver) and aluminum crucibles. All measurements were carried out in an inert nitrogen atmosphere with a nitrogen flow rate of 50 ml/min. The temperature range of measurements was 20-600°C, the heating rate was

5K/min. The amount of sample per measurement is 5-10 mg. The measuring system was calibrated with a standard set of substances KNO3, In, Bi, Sn, Zn.
Thermal analytical studies were carried out in analyzers at the Tashkent Scientific Research Institute of Chemical Technology, the image of scanning electron microscopy was taken at the center of advanced technologies at the Ministry of Innovative Development of the Republic of Uzbekistan.

RESULTS AND ITS DISCUSSION.

The process of covalent immobilization of aminoacetic acid with epoxy resin was carried out in molar ratios of 1.5:1 starting materials, a little polyethylenepolyamine was added as a hardener, at a temperature of 50°C, the reaction time was 20 minutes. The resulting resinous mass was poured into a porcelain bowl and dried in an oven at 70-80°C for 24 hours. The obtained product of the EAP (epoxy resin: aminoacetic acid,: polyethylenepolyamine) reaction with a yield of 88.2% is a resin-like mass of yellow color.
Scanning electron microscopy (SEM) (photographs, elemental analysis) was used to study the microscopic structure of the immobilized polymeric ligand and the coordination compound of the EAP sorbent with Cu^{2+} copper ions.
Figure 1 shows photographs of the microstructure of the surface of the immobilized ligand before (a) and after (b) sorption of copper (II) ions taken at 100 μm The image shows that the surface has a heterogeneous structure, the ion exchanger has a microporous structure, in which spherical, slit-like pores are traced, and there are larger and smaller particles. This indicates that the resulting ligand has a sufficiently high sorption capacity.



a b

Figure 1. Scanning electron microscope image surfaces of the immobilized ligand before (a) and after (b) sorption of ions copper(II)

Also in table 1 shows the elemental analysis of the obtained ligand and its coordination compound with Cu(II).

Table 1. Results of elemental analysis of the immobilized ligand and its complex compound with copper (II) ions

Element	Quantity, %	Sigma weight, %	Quantity, %	Sigma weight, %
	$[C_{54}H_{72}N_6O_{12}]n$		$[C_{54}H_{68}N_6O_{12}Cu_4]n$	
C	65,6	0,65	52,06	0,62
O	26,86	0,78	21,75	0,89
N	7,54	0,18	5,93	0,15
Cu	-		20.26	0,33
Sum	100,0		100,0	

An experiment was carried out to study the thermal stability of the ligand. Analysis based on the result of derivatographic analysis of various exothermic and endothermic thermal effects obtained during the mass installation in the process of destruction and the mass formation of compounds during the heating of ligands (Fig. 2). The derivatogram of the obtained polymeric ligand showed three endothermic effects at 195.66, 401.58, 445.53°C. The first shows the melting of the ligand at 195.6°C. Decomposition started at 401.58 °C. Endoeffects at temperatures of 401 and 445°C are due to the release of water involved in the protonation of amino groups in the ligand, as well as the release of water and ammonia from amino acids. At these temperatures, the overall reduction in ligand mass is 71%. In the temperature range 26-700°C, the reduction in the total weight of the ligand was 83.5%. Thus, the thermal stability of the synthesized ligand (EAL) indicates its stability up to a temperature of 195°C.

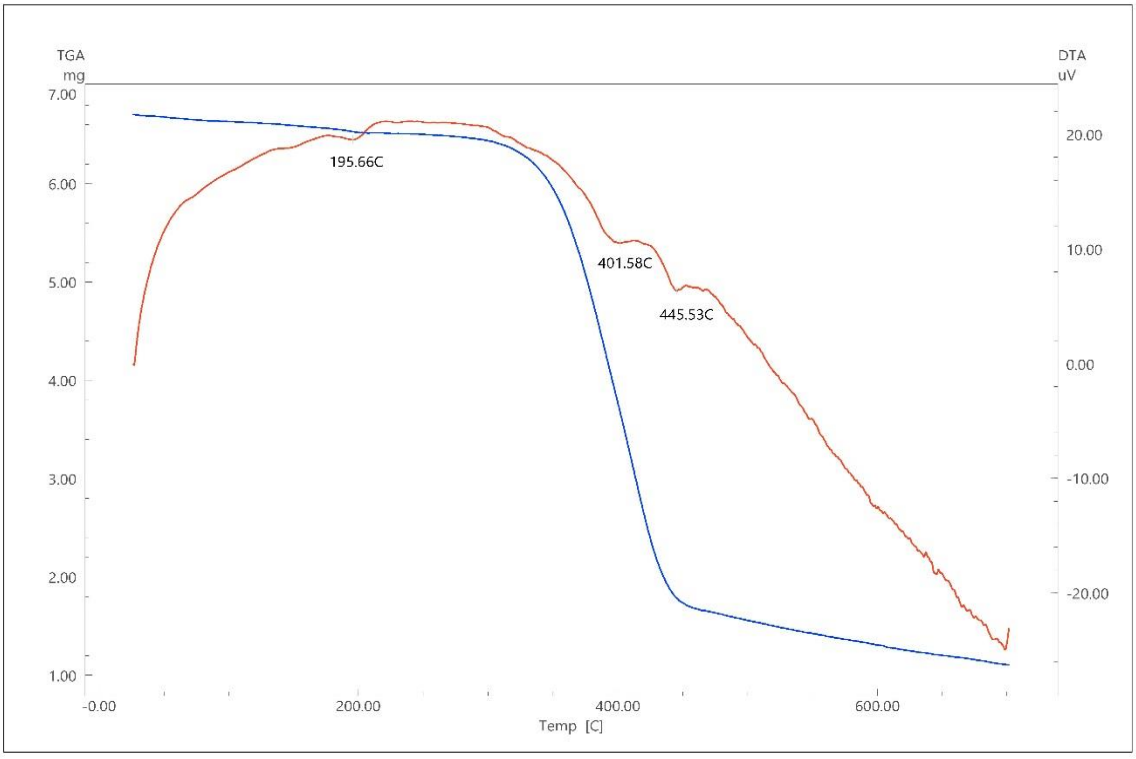


Figure 2. Thermogravimetric (TA) and differential thermal analysis (DTA) of the synthesized ligand

CONCLUSIONS

Thus, EAP polyampholyte was obtained by immobilizing aminoacetic acid in an epoxy resin matrix; it is stable up to 195°C; it is recommended for use in the sorption of some d-metal ions from solutions of higher temperatures. It also has a high sorption capacity for non-ferrous metals due to its microporous surface structure.

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