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ELECTRON MICROSCOPIC INVESTIGATION OF ION-EXCHANGE MATERIALS SYNTHESIZED BASED ON DIMETHYLOL CARBAMIDE, DIMETHYLOL THIOCARBAMIDE, AND DIGLYCIDYL THIOCARBAMATE

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Abstract. *In many studies, the scanning electron microscopy (SEM) method is widely used to determine the morphological and surface structure of various adsorbent materials [104; 204–208]. This is because the morphological structure of sorbents significantly influences their physicochemical properties.*

Keywords: *adsorbent, SEM, sorbent, elemental analysis.*

The widespread use of porous ion-exchange polymer materials requires the study of the regularities governing the formation of the structure of network polymers. The mechanism of formation of porous and film structures of the samples mainly depends on the nature of the inert diluent and the amount of the second monomer. In this process, cyclization of the polymer chains at the initial stages of polymerization plays a key role, increasing the homogeneity of the system at low conversion degrees. The type of the second monomer and its medium and high concentrations lead to the formation of micro-coils of the polymerized product due to such cyclization. In subsequent stages, these micro-coils ensure bonding between coils and form sparsely cross-linked regions of the polymer network. In turn, this may lead to the formation of network polymers with different spatial structures, including heterogeneous polymers with high internal molecular mobility.

Scanning electron microscopic studies were carried out in order to investigate the influence of the second monomer content on the morphology of ion-exchange resins synthesized based on DMC. The experiments were mainly performed on porous and film-type DMC+FC polymer ion-exchange resins prepared in a 1:1 ratio, as well as on DMC+FC+bentonite-based ion exchangers.

Figure 1 presents the results of scanning electron microscopy of DMC+FC ion-exchange resins obtained with different amounts of the second monomer.

The obtained results indicate a pronounced heterogeneity of the polymer ion-exchanger both in terms of particle size and shape, as well as in structural features (surface and internal region morphology). It can be seen that the ion-exchanger particles have a predominantly circular (oval) shape, and most of them correspond in form to ellipsoids with varying aspect ratios. Fully spherical particles are observed much less frequently. A significant variation in particle size is also observed, ranging from one-tenth of a micron to several hundred and even up to one thousand microns, i.e., reaching the millimeter scale.

In the study of the regularities governing the formation of the morphological structure of ion-exchange materials, researchers increasingly emphasize the importance of electron microscopy methods. The mechanism of pore and film formation in ion-exchange materials is mainly determined by the nature of the inert diluent (pore-forming agent) and the amount of crosslinking agent in the reaction system [105; pp. 30–55, 106; pp. 38–43, 107; pp. 40–43, 108; pp. 513–518]. In this process, cyclization of polymer chains at the initial stages of polymerization plays a key role in increasing the homogeneity of the system morphology. To determine the morphological structure of the synthesized ion-exchange resins, their scanning electron microscopy images were analyzed (Figures 1–4) [109; p. 103].

The elemental analysis of the obtained ion-exchange resins was performed using an EDX (energy-dispersive X-ray) system. The results of the analysis are presented in Tables 1–3.

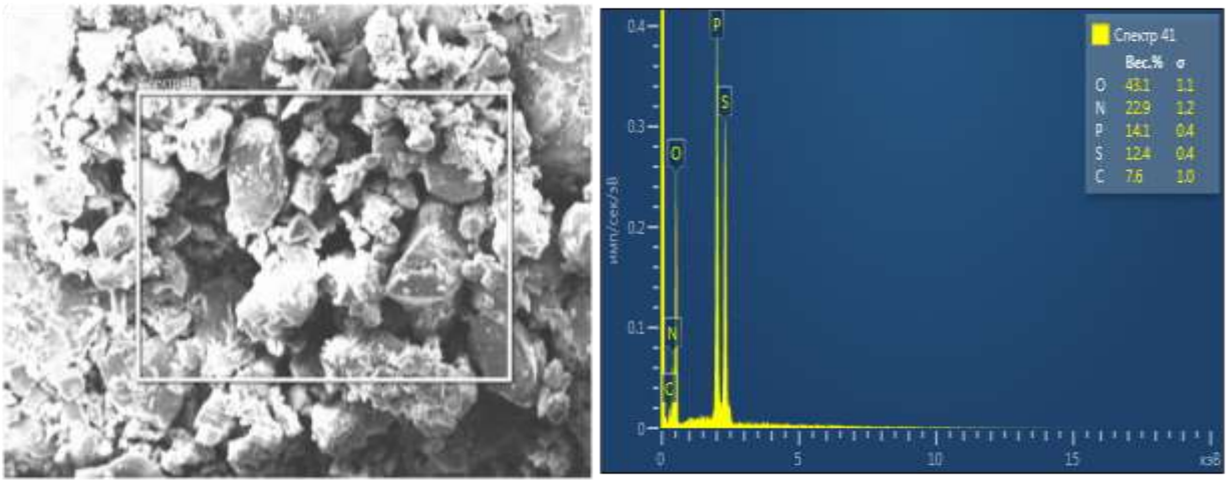

Figure 1. Scanning electron microscopy image of the surface of the DMC+FC ion-exchange resin and the elemental composition (%).

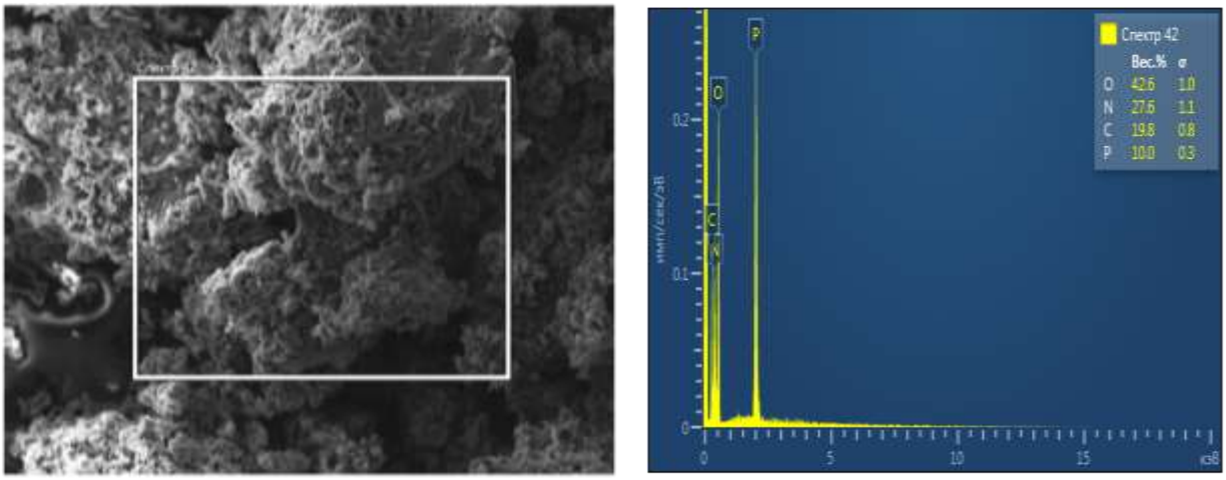

Figure 2. Scanning electron microscopy image of the surface of the DMT+FC ion-exchange resin and its elemental composition (%).

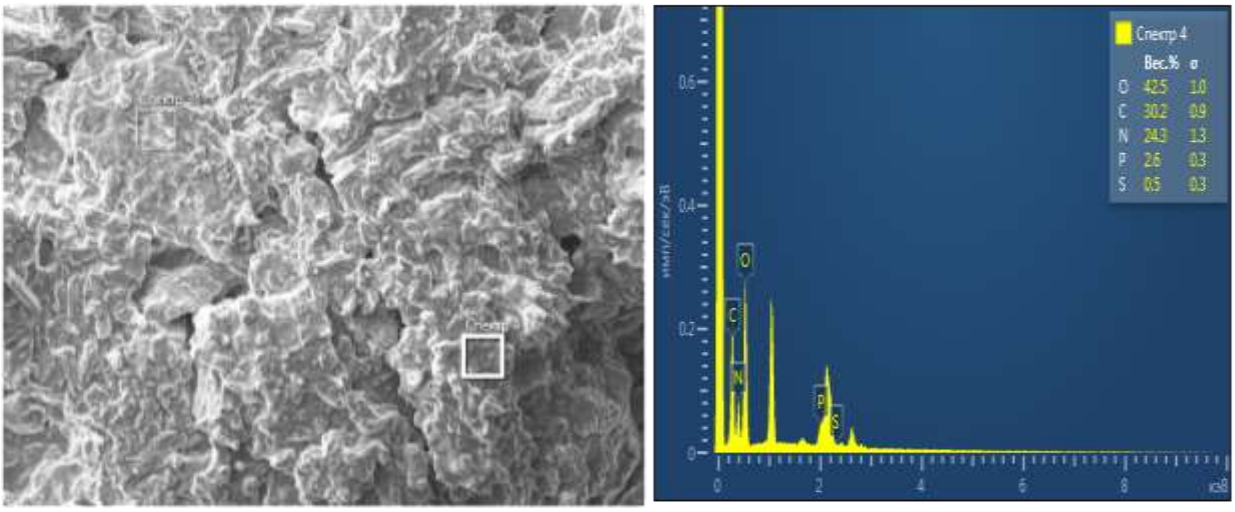

Figure 3. Scanning electron microscopy image of the surface of the DGT+FC ion-exchange resin and its elemental composition (%).

In the SEM micrographs presented above, Figure 1 shows that the surface of the DMC+FC ion-exchange resin has a smooth structure, whereas Figure 2 illustrates that the surface of the DMT+FC ion-exchange resin, which contains sulfur, differs significantly. It can be clearly observed that the surface of the first ion-exchanger differs from that of the second one in terms of a larger number and size of macropores. This may be attributed to the presence of sulfur atoms in the ion-exchange material.

In addition, it can be seen that the pore characteristics remain unchanged even after treatment with acid and alkali solutions. This indicates that the ion-exchange sorbent based on DMC+FC possesses a large specific surface area and such a morphological structure, which may exhibit high sorption capacity toward various metal ions [110; pp. 435–446].

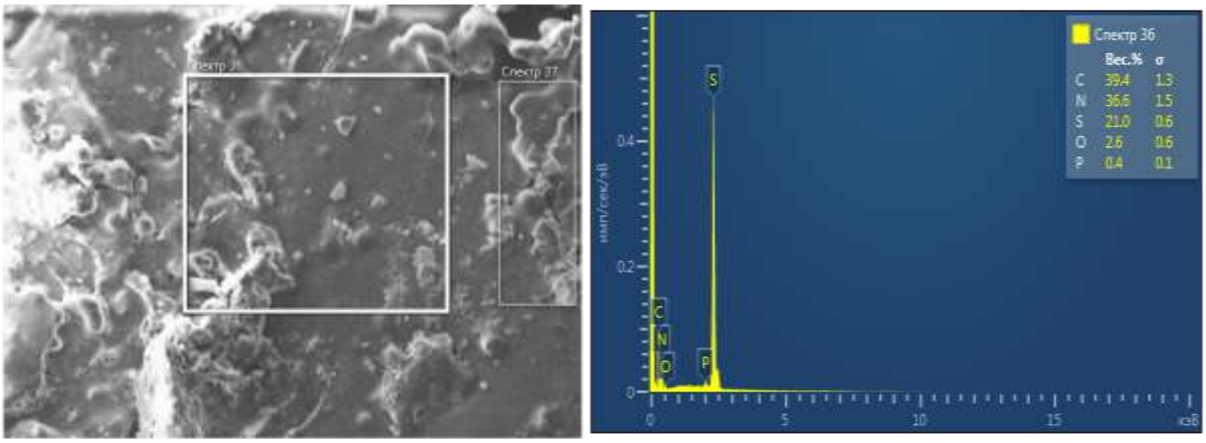

Figure 4. Scanning electron microscopy image of the surface of the DGT+AF ion-exchange resin and its elemental composition (%).

In addition, quantitative microanalysis of elements by energy-dispersive X-ray spectroscopy (EDS) showed that the mass fractions of the assumed phosphorus and nitrogen elements in the ion-exchange material were 14.08% and 22.91%, respectively. This indicates that the phosphorus content obtained by elemental analysis using the EuroEA elemental analyzer was relatively lower compared to the above results. The reason for this is that amination is the final stage of the reaction, and therefore amino groups are predominantly present on the material surface. It should be noted that energy-dispersive X-ray (EDS) microanalysis reflects the elemental composition only on the surface of the granules. The mass fraction of nitrogen (N) in the polycomplex was found to be almost identical in both elemental analysis methods.

Furthermore, in this chapter, optimal conditions for obtaining a new ion-exchange material based on DMC+FC resin modified with Khavdak bentonite were determined. The structure and composition of the ion-exchange sorbent obtained under these conditions were studied using modern physicochemical methods for identification [109; pp. 435–446].

The results of the study confirmed that the product obtained based on DMC+FC contains phosphorus and nitrogen, has a porous structure, and possesses both cation-exchange and anion-exchange properties due to the presence of phospho- and amino groups. In order to determine the possible applications of this ion-exchange sorbent, its investigated physicochemical properties are presented in the following chapter.

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