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Evaluation of the structural and compositional properties of carbon adsorbents derived from local waste by the SEM-EDX technique

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Abstract. Activated carbon is one of the most widely used adsorbents due to its high specific surface area and well-developed porosity. Its performance in various applications, such as environmental, industrial, and biomedical fields, is directly related to its morphological structure and the elemental composition of its surface. In this context, scanning electron microscopy (SEM), coupled with energy-dispersive X-ray spectroscopy (EDX), represents an essential technique for the characterization of activated carbons. The article provides a detailed interpretation of the information obtained by SEM–EDX in the study of activated carbon, analyzing SEM images and EDX spectra in correlation with carbonization, activation, and chemical modification processes.
Keywords: activated carbons, citric acid, SEM-EDX.

Evaluarea proprietăților structurale și compoziționale ale adsorbanților carbonici din deșeuri locale prin tehnica SEM-EDX

Rezumat. Cărbunele activ este unul dintre cei mai utilizați adsorbanți datorită suprafeței specifice mari și porozității bine dezvoltate. Performanța sa în diferite aplicări, cum ar fi cele de mediu, industriale și biomedicale sunt direct legate de structura morfologică și de compoziția elementală a suprafeței. În acest context, microscopia electronică de baleiaj (SEM), cuplată cu spectroscopia de raze X cu dispersie de energie (EDX), reprezintă o tehnică esențială pentru caracterizarea cărbunilor activi. Articolul oferă o descifrare detaliată a informațiilor obținute de SEM-EDX în studiul cărbunelui activ, interpretând imaginile SEM și spectrele EDX în corelație cu procesele de carbonizare, activare și modificare chimică.
Cuvinte-cheie: cărbune activ, acid citric, SEM-EDX.

1. Introduction

Activated carbon represents a class of carbon-based materials with an extremely high specific surface area and a well-developed pore network, which makes it widely used as an adsorbent in environmental, industrial, and analytical applications — from water and air purification to metal extraction or carbon dioxide capture. The structural and chemical characteristics of these materials directly influence their performance in adsorption processes; therefore, characterization of their morphology and composition is essential for understanding and optimizing their functional properties.

The Scanning Electron Microscopy (SEM) technique combined with Energy-Dispersive X-ray Spectroscopy (EDX) is one of the most widely used instrumental methods for the characterization of activated carbons. SEM provides high-resolution images of the surface, allowing investigations of texture, pore development, and topographical structure, while EDX delivers information on the elemental composition of the samples, which is crucial for identifying residual heteroatoms or elements introduced during chemical modification of activated carbons [1].

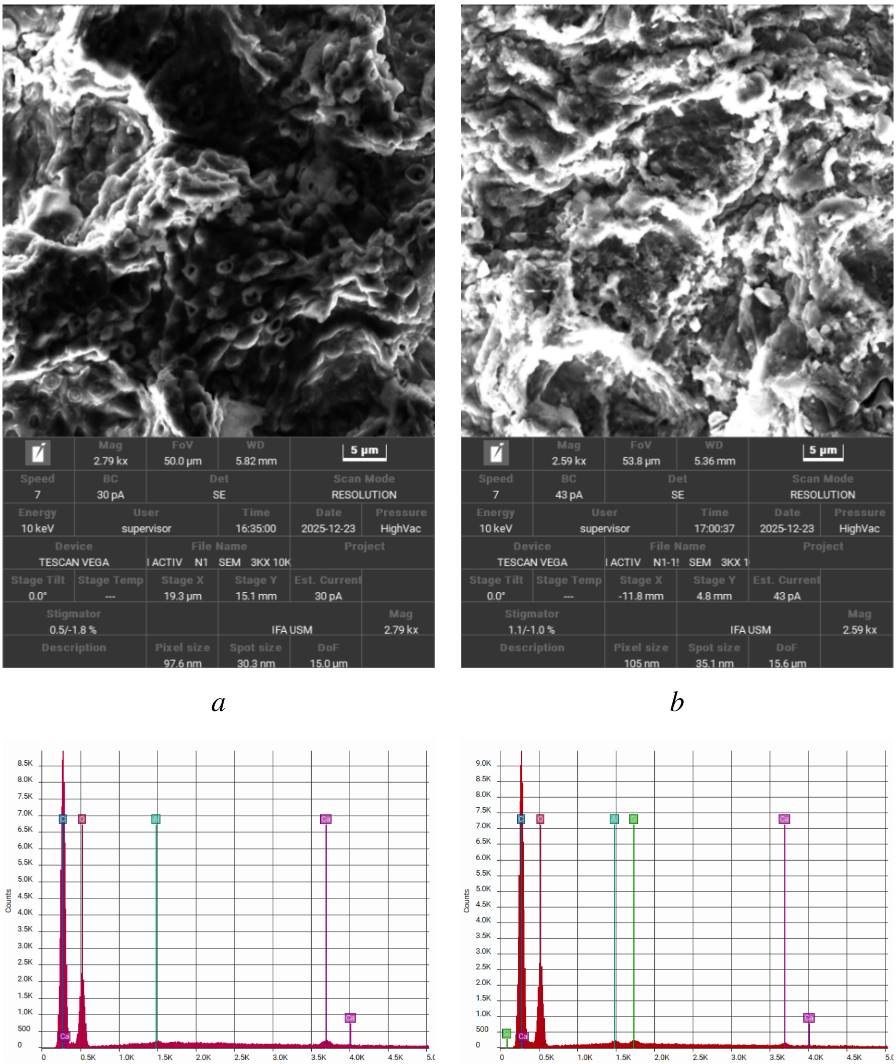
In carbon materials research, SEM–EDX analysis is frequently used to correlate porous morphology with the distribution of chemical elements, thus providing a comprehensive view of the structural and chemical characteristics of adsorbents. Through this combination, it is possible, for example, to assess the increase in carbon content after activation or the presence of metal atoms (Ca, Fe, etc.) from natural precursors or applied chemical treatments [2]. The scientific literature contains numerous studies in which SEM–EDX has been used to analyze activated carbons obtained from various raw materials (coconut shell, mango seed, agro-industrial waste), highlighting significant variations in pore texture and elemental composition determined by activation conditions and subsequent treatments [1], [3], [4].

Thus, the use of SEM–EDX not only provides essential data on the physical and chemical structure of activated carbons but also serves as a basis for optimizing synthesis processes and adapting materials for specific applications, such as pollutant removal or gas adsorption with climate impact. Samples used in this study was the biomass percussor consisted of Junglas regia cv. Chandler walnut shells collected from Tirsitei village, Telenesti district, Republic of Moldova.

2. Results and discussions

SEM micrographs reveal a well-developed porous structure, characteristic of chemically activated materials (Figure 1). Walnut shells were chemically impregnated with aqueous citric acid solutions at concentrations of 30%, 50% and 70%, producing two series of samples for each impregnation level. One series was washed with distilled water to neutral pH prior to carbonization, while the other was carbonized directly at 350 °C and 450 °C for 2 h using a heating rate of 10 °C min^{-1} in a SNOL laboratory muffle. Citric acid served as an environmentally friendly activating agent, promoting the development of oxygen-containing surface groups that improve the adsorption properties of the resulting activated carbons.

The sample treated at 350 °C exhibits moderate porosity, with relatively compact walls and a non-uniform pore distribution. At 450 °C, a more pronounced development of the porous network can be observed, along with a clearer opening of cavities. The surface becomes rougher and more fragmented at the higher temperature.



a *b*

c *d*

Figure 1. SEM-EDX analysis of activated carbon samples 1 (*a, c*) and 1' (*b, d*), washed and treated at (1) 350 °C and (1') 450 °C, 30% citric acid

The EDX spectra indicate the predominance of carbon and oxygen, with a slight increase in carbon content for the sample treated at 450 °C, suggesting an intensification of the carbonization process. The absence of significant inorganic impurities confirms the efficiency of the washing step.

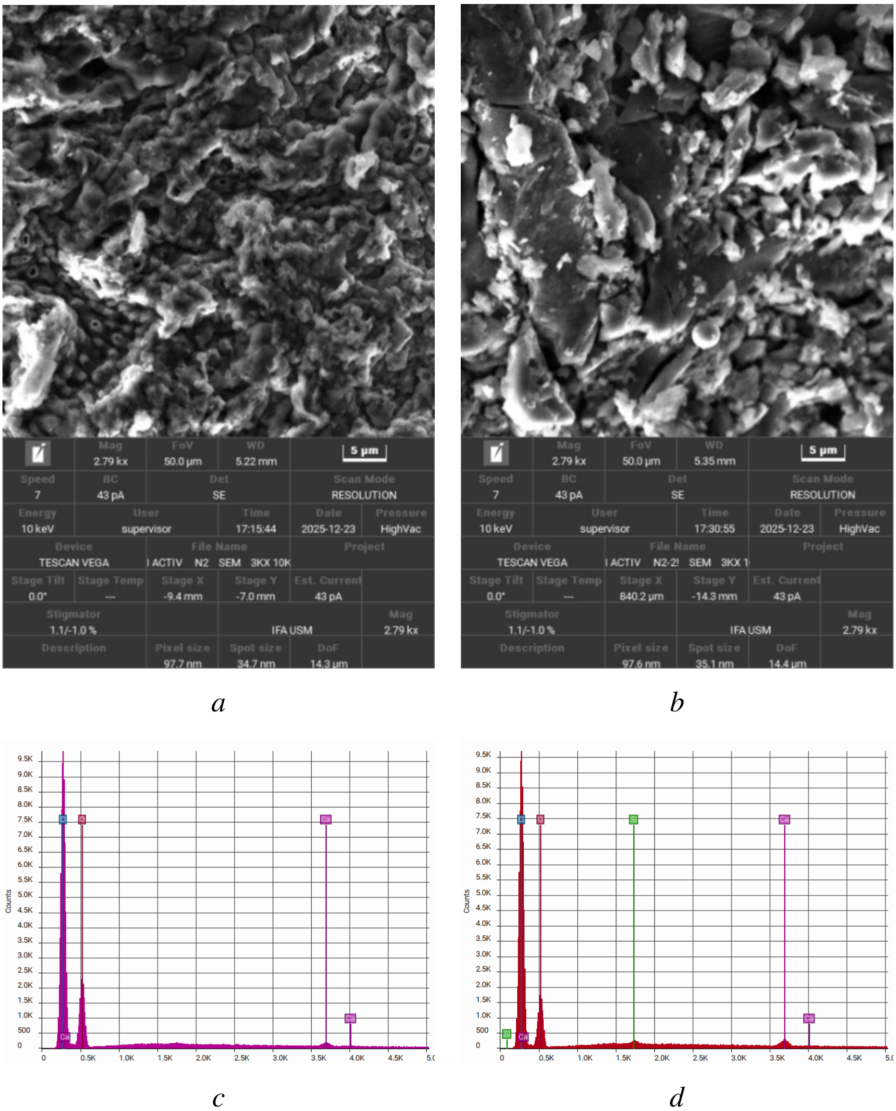
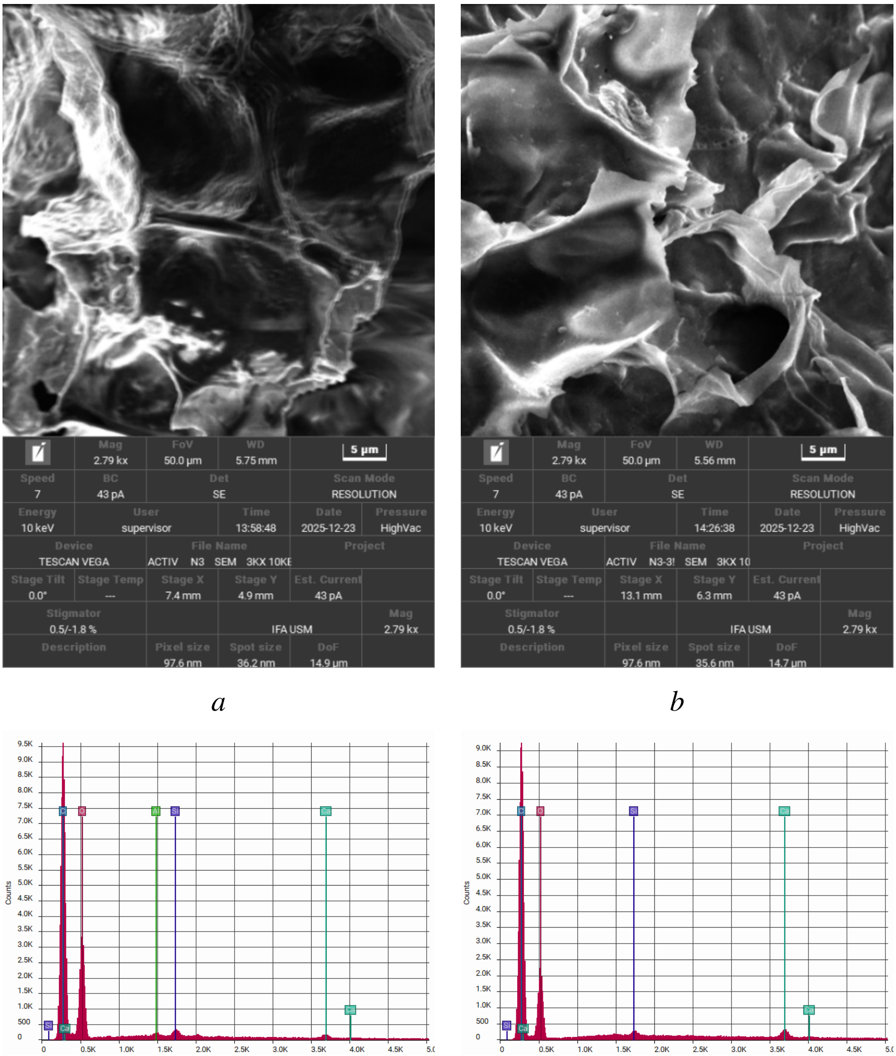


Figure 2. SEM-EDX analysis of activated carbon samples 2 (*a, c*) and 2' (*b, d*), washed and treated at (2) 350 °C and (2') 450 °C, 50% citric acid

At a citric acid concentration of 50% (Figure 2), the microstructure exhibits a more developed porosity compared to Figure 1. At 450 °C, an increase in pore size and interconnectivity is observed, indicating a more intense activation process. The structure becomes more open and heterogeneous.

EDX analysis shows a C/O ratio favorable for advanced carbonization, while maintaining the presence of oxygen-containing surface functional groups. The elemental composition suggests efficient functionalization without mineral contamination.



a *b*

c *d*

Figure 3. SEM-EDX analysis of activated carbon samples 3 (*a, c*) and 3' (*b, d*), washed and treated at (3) 350 °C and (3') 450 °C, 70% citric acid

At a citric acid concentration of 70% (Figure 3), a significant modification of the morphology can be observed. At 350 °C, the structure presents well-defined pores, but with localized areas of partial wall collapse. At 450 °C, a pronounced development of macropores and a thinning of the internal structure appear. The surface is intensely fragmented, indicating a high degree of chemical activation. EDX spectra confirm the predominance of carbon and oxygen, while the relatively higher oxygen content reflects the introduction of carboxylic and phenolic functional groups. The results suggests a balance between porosity development and mechanical stability.

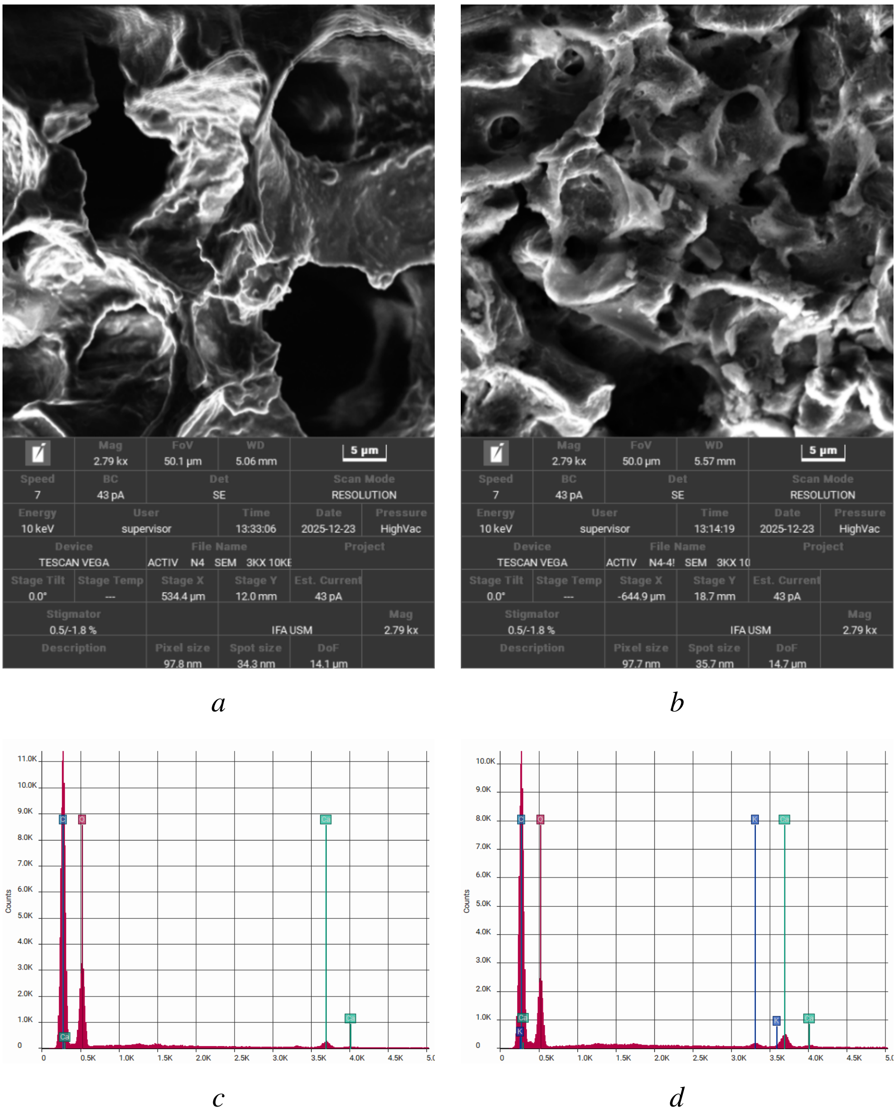
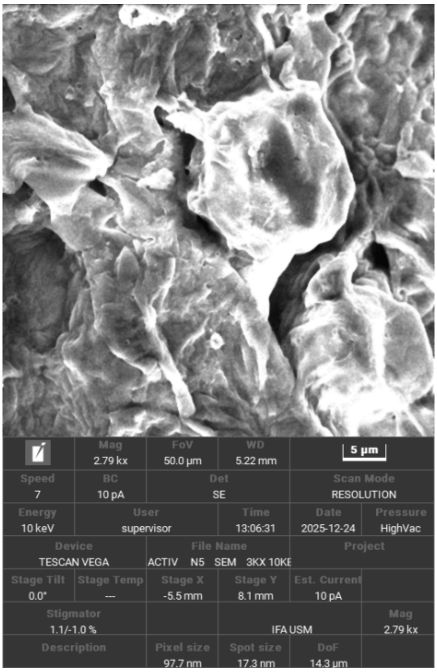
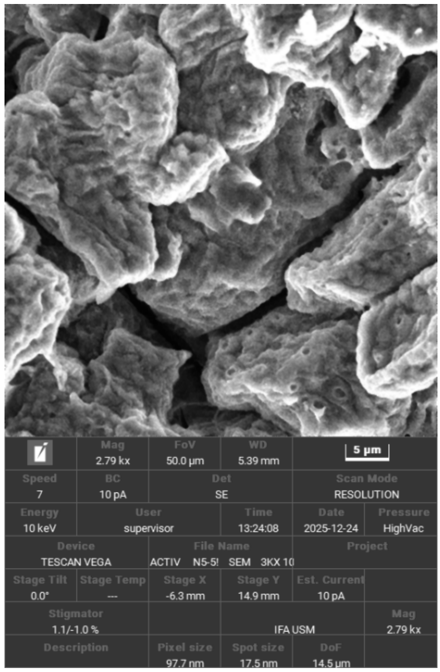


Figure 4. SEM-EDX analysis of activated carbon samples 4 (*a, c*) and 4’ (*b, d*), unwashed and treated at (4) 350 °C and (4’) 450 °C, 30% citric acid

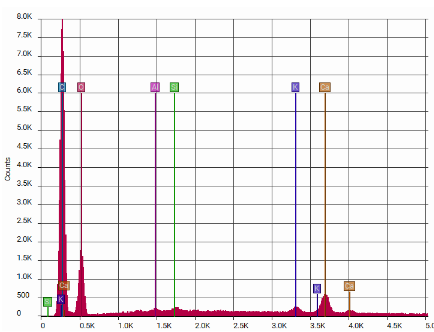
SEM micrographs reveal a less uniform surface compared to the washed samples (Figure 4). At 350 °C, adherent particles and deposits can be observed on the pore surface, associated with inorganic residues. At 450 °C, the porosity becomes more evident; however, the presence of aggregates persists. EDX analysis indicates, in addition to carbon and oxygen, the presence of residual mineral elements, suggesting the retention of salts or inorganic compounds. The lack of the washing step negatively affects the purity and structural homogeneity of the material.



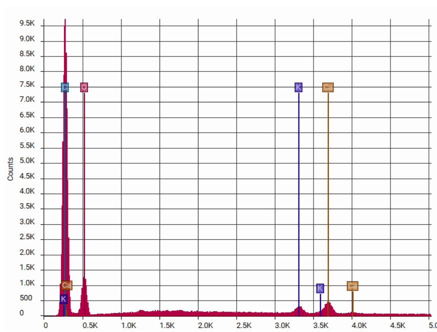
a



b



c



d

Figure 5. SEM-EDX analysis of activated carbon samples 5 (*a, c*) and 5' (*b, d*), unwashed and treated at (5) 350 °C and (5') 450 °C, 50% citric acid

The morphological structure exhibits developed porosity, but with areas of partial pore blockage due to residual deposits (Figure 5). At 350 °C, a relatively uniform distribution of cavities can be observed, whereas at 450 °C larger pores and structural cracks appear. EDX spectra highlight a higher content of secondary elements compared to the washed samples. Increasing the temperature leads to a partial reduction of residues, though not complete removal. The material demonstrates effective activation, but with reduced purity.

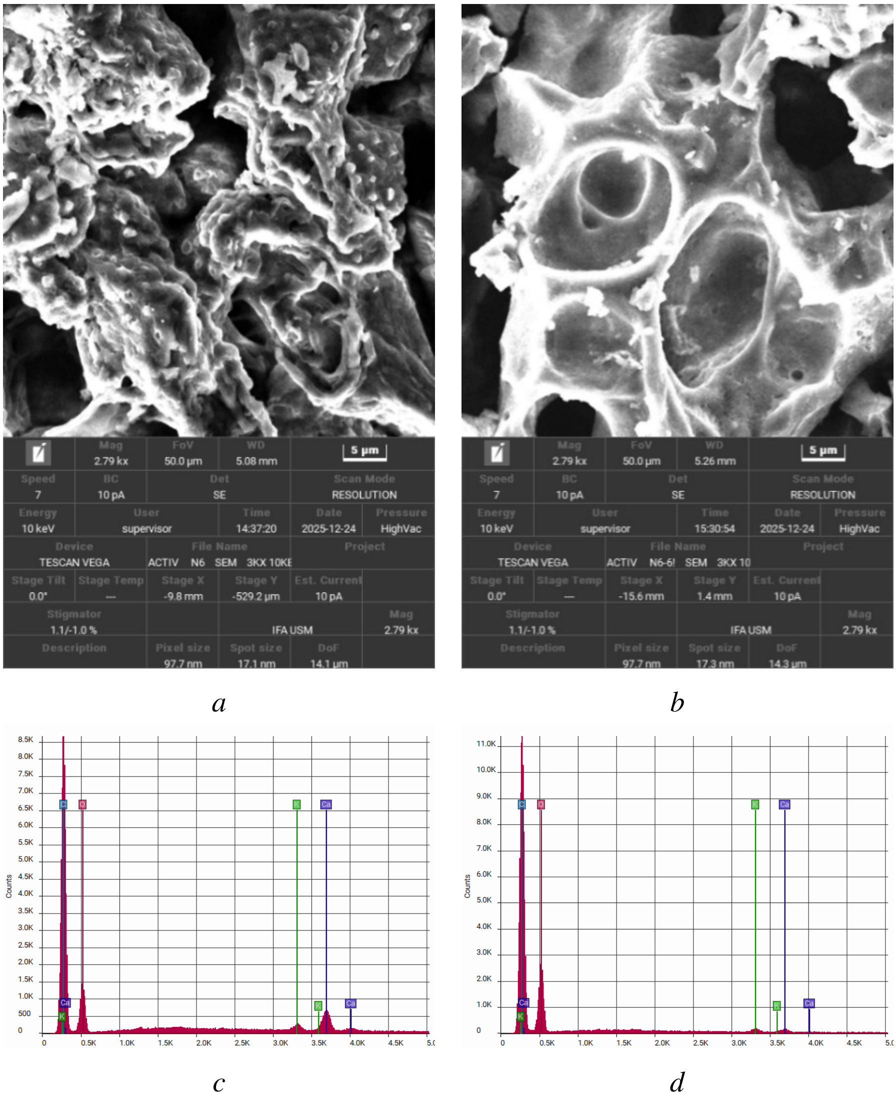


Figure 6. SEM-EDX analysis of activated carbon samples 6 (*a, c*) and 6’ (*b, d*), unwashed and treated at (6) 350 °C and (6’) 450 °C, 70% citric acid

At high concentration and without washing, the structure exhibits pronounced but highly heterogeneous porosity. At 350 °C, visible aggregates and inorganic deposits can be observed. At 450 °C, the structure becomes more open; however, areas of structural collapse appear. EDX analysis indicates carbon and oxygen as the dominant elements, along with traces of persistent mineral elements. Intense activation combined with the absence of purification leads to a disordered morphology and a more complex chemical composition (Figure 6).

In all cases, increasing the temperature leads to a more pronounced development of porosity and a greater degree of surface fragmentation. At 450 °C, a more open structure and a higher degree of carbonization can be observed. An increase in acid concentration promotes pore development and the introduction of oxygen-containing functional groups; however, at 70% it may result in partial structural collapse. The washed samples exhibit superior purity and the absence of mineral elements detectable by EDX. In contrast, the unwashed samples contain inorganic residues that may negatively affect their adsorptive performance.

3. Conclusions

The SEM–EDX analysis demonstrates that both the activation temperature and the citric acid concentration significantly influence the morphology and elemental composition of the activated carbons. Increasing the temperature to 450 °C leads to the development of a better-defined porous network and a higher degree of carbonization. High citric acid concentrations intensify pore formation but may compromise structural stability. The washing step is essential for obtaining a material with high chemical purity and a uniform pore distribution. Overall, the optimal conditions appear to be treatments at 450 °C with intermediate citric acid concentrations and prior washing.

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