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Prof. Dr Ana Radojević

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Original Research

SEM-EDS AND DTA-TGA ANALYSIS OF CORN STALKS USED FOR THE
BIOSORPTION OF COPPER IONS FROM SYNTHETIC SOLUTIONS

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Abstract

This paper presents the results of SEM-EDS and DTA-TGA analysis of corn stalks before and after the biosorption of Cu²⁺ ions. Certain differences in the adsorbent structure and in the elemental analysis of the sample surface were observed. In micrographs of raw corn stalks, pores and cavities can be seen, which facilitate the penetration of the aqueous phase into the adsorbent structure, where copper ions are adsorbed at the internal active sites. After the biosorption of Cu²⁺ ions, a distinctly different surface morphology of corn stalks can be observed. The absence of cavities and channels is evident, with a compact and uniformly ordered lamellar structure. The EDS spectra of corn stalks, before and after copper ion biosorption, indicate the possible exchange of potassium, calcium, and magnesium with copper during the biosorption process. Based on the DTA-TGA analysis, the degradation of the corn stalk sample occurs in several stages.

Keywords: copper ions, biosorption, corn stalks, SEM-EDS, DTA-TGA.

INTRODUCTION

Rapid technological development enabled humans to exploit the planet's natural resources more intensively, but it also caused a steady increase in environmental pollution. Major contributors to natural water contamination include wastewater from urban areas, ore deposits, non-ferrous and ferrous metallurgy, the textile and paint industries, and various agricultural activities. Consequently, the increasing discharge of heavy metals into aquatic ecosystems has become a serious threat to both the environment and human health, highlighting the urgent need to develop efficient and sustainable wastewater treatment methods [1].

Agricultural by-products, such as corn stalks, are promising, low-cost, and environmentally friendly adsorbents for removing metal ions from contaminated water. Because of their lignocellulosic structure and numerous functional groups, both modified and unmodified corn stalks can effectively bind heavy metal ions through biosorption [2].

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Biosorption is the ability of biomaterials to accumulate and concentrate heavy metals from even very dilute aqueous solutions. The biosorption process involves a solid phase (biological material) and a liquid phase (solvent, usually water) containing the dissolved metal [3].

It is a passive metabolic process, meaning it does not require energy, and the amount of pollutants adsorbed depends on the structure of the biosorbent and kinetic equilibrium [4]. Biosorption is determined by chemical equilibrium and is largely influenced by pH, ion concentration, and interactions between ions of different metals [5].

The main advantages of biosorption over conventional purification technologies, in addition to its efficiency in removing metal ions from aqueous solutions, are the availability and low cost of these adsorbents, which are usually waste products from the wood, agricultural, and food industries. Therefore, biosorption can serve as an alternative to existing conventional wastewater treatment technologies [3].

MATERIALS AND METHODS

Corn stalks collected from agricultural fields near Bor were used as the adsorbent material. Before use, the samples were mechanically ground and sieved, and the particle size fraction of $(-1 + 0.4)$ mm was selected for the biosorption experiments.

RESULTS AND DISCUSSION

The morphology of corn stalks before and after biosorption of Cu^{2+} ions was analyzed using a scanning electron microscope (SEM) (VEGA 3 LMU TESCAN) equipped with an integrated energy-dispersive X-ray detector (X-act SDD 10 mm², Oxford Instruments). Before SEM analysis, the samples were coated with a thin layer of chromium in a sputter coater (QUORUM Q150T ES) to ensure conductivity. The prepared samples were then transferred to the microscope chamber and observed at an accelerating voltage of 8 kV.

SEM-EDS analysis of the corn stalks was performed both before and after Cu^{2+} ion biosorption. Certain differences in the structure of the adsorbent and in the elemental composition of the sample surface were observed. Figure 1 shows the surface morphology of corn stalks before and after copper ion biosorption, along with the corresponding EDS spectra.

Figure 1a shows a micrograph of the surface of corn stalks before copper ion biosorption, where cavities and channels are visible. These features facilitate the movement of the aqueous phase through the structure. It is assumed that copper ions penetrate these cavities and channels into the adsorbent structure, where they are adsorbed onto internal active sites. After copper ion biosorption (Figure 1b), the surface morphology of the stalks changes significantly. Cavities and channels are no longer observed, and the surface of the corn stalks becomes more compact, most likely due to the incorporation of copper ions into the structure.

EDS imaging was performed at several points on the sample surfaces to detect the heterogeneous distribution of elements. In both untreated corn stalks and corn stalks after copper ion biosorption, peaks for Cr are observed in the EDS spectrum. These Cr peaks occur because the adsorbent surface was coated with chromium in the vaporizer before imaging to make it conductive. The EDS spectrum of corn stalks before biosorption (Figure 1c) shows a high content of Ca, C, O, K, Si, Mg, and P. After copper ion biosorption (Figure 1d), the

peaks for K, Mg, Ca, and Si disappear, while peaks for Cu appear. Therefore, it can be assumed that K, Mg, and Ca are most likely exchanged with Cu by an ion exchange mechanism during the biosorption process.

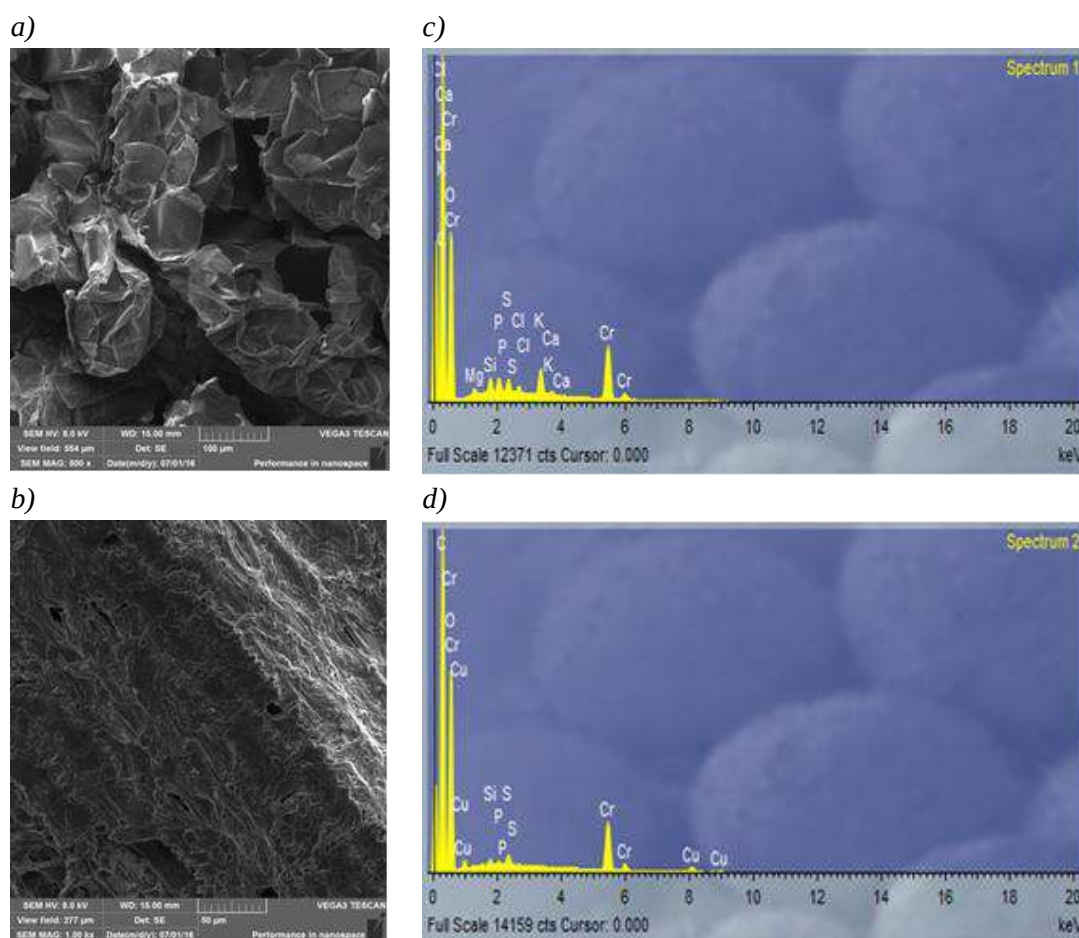


Figure 1 SEM micrograph of the surface of corn stalks: a) before biosorption of copper ions (magnification 500×); b) after biosorption of copper ions (magnification 1000×); c) EDS spectrum of untreated corn stalks; d) EDS spectrum of corn stalks after biosorption of copper ions

Thermal analysis provides important information about the thermal stability and degradation of the material. In this paper, DTA-TGA analysis of corn stalks was performed using a simultaneous DSC-TGA device (SDT Q600). A 4 mg sample was heated in an inert nitrogen atmosphere at a constant heating rate of 10 °C/min over a temperature range from 20 °C to 900 °C, and the results are shown in Figure 2.

Examining the TGA curve in Figure 2, a mass loss of about 7.6% is observed between 20 °C and 200 °C, resulting from the loss of physically bound (adsorbed) water from the sample. Additionally, Figure 2 shows that this process is accompanied by an endothermic peak on the DTA curve, with a minimum at 64.39 °C. In the temperature range from 200 °C to 900 °C, a mass loss of 77% is observed on the TGA curve, originating from the decomposition of lignocellulosic components in the adsorbent structure and the formation of volatile products such as CO, CO₂, and others [6]. In the same temperature range, an exothermic peak appears on the DTA curve, with a maximum at 300 °C, which corresponds

to the decomposition temperature of hemicellulose. In summary, the total mass loss in the temperature range from 20 °C to 900 °C was 84.6%.

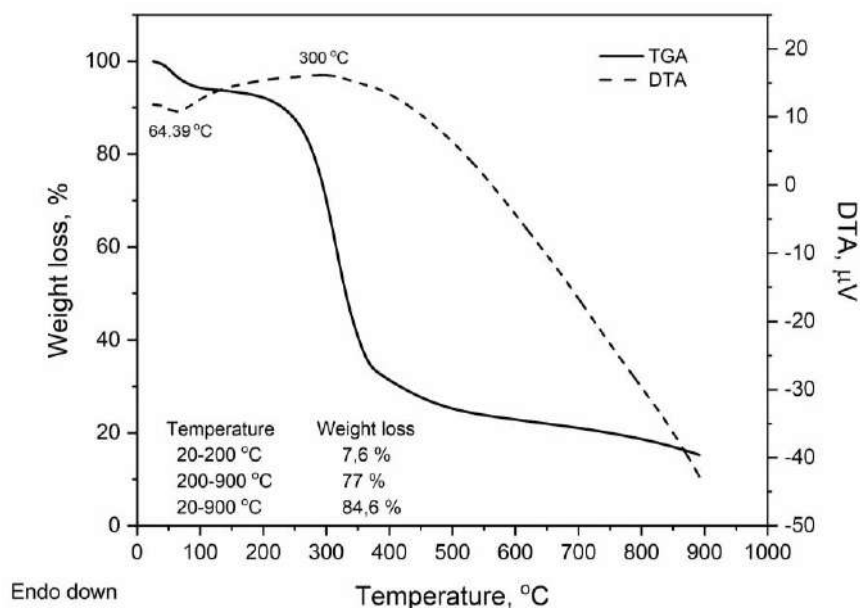


Figure 2 DTA-TGA analysis of corn stalks

CONCLUSION

This study presents the findings of SEM-EDS and DTA-TGA analyses conducted on corn stalks before and after the biosorption of Cu^{2+} ions. SEM analysis of corn stalks before and after copper ion biosorption shows that the surface morphology of corn stalks significantly changes after copper ion biosorption. The EDS spectra of corn stalks, before and after copper ion biosorption, indicate the possible exchange of potassium, calcium, and magnesium with copper during the biosorption process.

Based on the DTA-TGA analysis, the degradation of the corn stalk sample occurs in several stages.

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