The 56th International October Conference on Mining and Metallurgy
22-25 October 2025, Bor Lake, Bor, Serbia
www.ioc.tfbor.bg.ac.rs

DOI: 10.5937/IOC25607K
Original scientific paper

TREE LEAF BIOCHAR AS POTENTIAL ADSORBENT FOR LEAD ION REMOVAL FROM AQUEOUS SOLUTION

Marija Koprivica[1a], Marija Simić[1b], Jelena Petrović[1c], Jelena Dimitrijević[1d], Marija Ercegović[1e], Marija Marković[1f]

[1]Institute for Technology of Nuclear and Other Mineral Raw Materials, Franchet d'Esperey 86, 11000 Belgrade, Serbia
[1a] m.koprivica@itnms.ac.rs, 0000-0002-1451-5959
[1b] m.petrovic@itnms.ac.rs, 0000-0003-1717-7796
[1c] j.petrovic@itnms.ac.rs, 0000-0003-4841-1899
[1d] j.dimitrijevic@itnms.ac.rs, 0000-0002-3830-2392
[1e] m.ercegovic@itnms.ac.rs, 0000-0002-7145-8671
[1f] m.markovic@itnms.ac.rs, 0000-0001-6787-1933

Abstract

Industrial wastewater is a type of environmental pollution and, in the 21st century, is one of the major problems for humanity and nature. Lead is a metal that negatively impacts the environment and human health. Therefore, great attention has been paid to removing this metal from waste effluents generated during mining activities and various industrial processes. Adsorption technology is a powerful tool for the removal of pollutants from wastewater. During past years, there has been an increase in the use of plant waste and its products for this application. In this study, Paulownia tree leaves (PL) were structurally changed by the pyrolysis process and converted to biochar (PLB), which was used as a Pb ion adsorbent. The obtained material was characterized via FTIR and SEM-EDX analysis before and after Pb ion removal. The results of the FTIR analysis showed differences in the intensity of the surface functional groups, especially the aromatic and oxygen functional groups. These changes confirm that behind ion exchange, the complexation, and/or cation-π electron interaction done too. The SEM-EDX also showed changes in the surface structure and elemental content. The surface exhibited newly formed cracks and pores during Pb ion adsorption. Also, the EDX confirmed that the percentages of O, N, K, Mg, and Ca decreased while the lead content increased significantly. The summarized results from this work revealed that leaves from trees are valuable biomass for sustainable biochar production, supporting waste valorization, and contributing to pollution control efforts, making them suitable for adsorption of metal ions.

Keywords: *pyrolysis, biochar as adsorbent, Pb(II) ion removal, FTIR analysis, SEM-EDX analysis*

1. INTRODUCTION

Mining and industrial activities are responsible for why lead (Pb) is one of the most abundant and toxic heavy metals that occurs in nature. Since it can result in a range of health problems not only for humans but also for plant and animal life, it is imperative to remove it from the environment as effectively as possible [1,2]. One of the best solutions for removing heavy metals from wastewater is adsorption using various effective materials. Potential materials for adsorption can be obtained from waste biomass [1,2,3,4]. In this regard, this work aims to evaluate the possibility of obtaining an efficient lead ion (Pb^{2+}) adsorbent from aqueous solutions by pyrolysis of Paulownia tree leaves (PL), which represent a cheap and economical source of raw materials. The FTIR and SEM-EDX analyses of the obtained biochar (PLB) before and after lead ion adsorption were performed for characterization.

2. EXPERIMENTAL

2.1 Materials preparation

The initial feedstock of obtained biochar was leaves from the Paulownia tree (PL). It was collected in a Belgrade park, washed with distilled water, air-dried for three weeks, and crushed with a grinder to obtain a finer sample. The biochar was obtained from 10g of the tree leaf powder via the slow pyrolysis process at 400 °C, 2 hours under argon gas flow in a laboratory furnace (Nabertherm, Germany). After pyrolysis, the biochar (PLB) was stored in a desiccator until adsorption tests were performed.

2.2 Preliminar adsorption test

Adsorption isotherms studies were conducted by stirring 0.02 g of the PL or the PLB powder with 20 mL of Pb solutions at a concentration of 100 mg/L for 24 hours at room temperature. The solution was separated from the adsorbent by filtration, and Pb^{2+} concentrations before and after adsorption were determined using an atomic adsorption spectrophotometer (AAS, PerkinElmer, PinAAcle 900T).

The amount of Pb^{2+} ions adsorbed on the PL and PLB surface was calculated using equation:

$$q = (C_0 - C_{eq})/m \times V \qquad (1)$$

Where q [mg/g] was the adsorbent capacity, C_0, and C_{eq} [mg/L] were initial and after adsorption concentration of Pb ions, m [g] was the amount of adsorbent and V [L] was volume of the Pb^{2+} solutions.

2.3 Characterization of material before and after lead ions adsorption

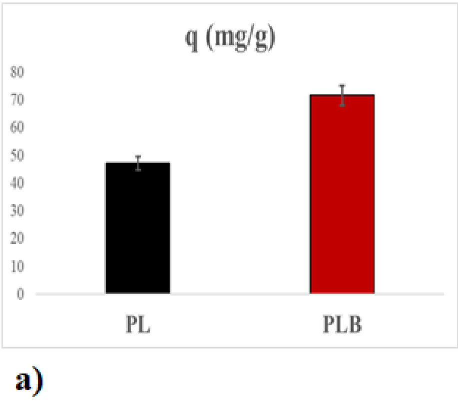
FTIR analysis was used to determine the presence of functional groups on the surface of the PLB before and after the lead sorption process. The samples' FTIR analysis was recorded on a Thermo Fisher Scientific Nicolet IS-50 spectrophotometer in the spectral range of 400–4000 cm^{-1} in transmission mode. All samples were converted into pellet form using KBr.

The surface morphology of the PLB before and after lead ions adsorption was observed using SEM-EDX analysis (JEOL JSM-6610LV SEM, JEOL Inc., USA). All samples were cathodic-coated with gold, placed on an adhesive carbon disc, and recorded in a vacuum under a voltage of 20 kV.

3. RESULTS AND DISCUSSION

3.1 Preliminar adsorption test

The preliminary adsorption test revealed that the PL and PLB exhibited different adsorption affinity toward Pb^{2+} (Figure 1a). As can be observed in Figure 1a, the pyrolysis process improved the adsorption properties of PL (from q= 45.18 mg/g to q=71.67 mg/g). Similar findings were showed with the results for Ginkgo tree leaf and it biochar obtained at 400°C, where pyrolysis improved adsorption efficiency for Pb ions removal from q=24.81 mg/g to q=51.13 mg/g [4].



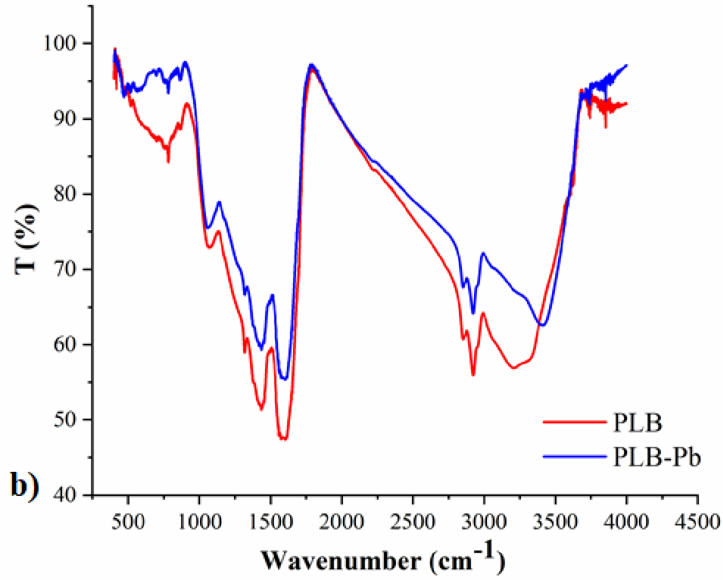


Figure 1. a) Comparison at Pb^{2+} ions adsorption effectiveness between PL and PLB, b) FTIR spectrum before and after Pb ion adsorption at PLB surface.

3.2 Characterization of material before and after lead ions adsorption

A decrease and shift in the frequencies of characteristic vibrations, such as -OH groups around 3300-3500 cm^{-1}, suggests interactions between functional groups on the adsorbent surface and Pb^{2+} ions (Figure1b) [1,2,4]. These appearances indicated bond weakening due to Pb^{2+} ion binding, implying hydrogen exchange, complexation, or covalent interactions [2,5]. Besides, spectral changes in the form of a decrease of intensity at about 1000-2000 cm^{-1} may indicate metal ion binding through interactions with aromatic and oxygen functional units on the adsorbent surface [1,2,4]. Also, the peak at about 800 cm^{-1}, which originated from aromatic C-H vibration, was reduced after adsorption [1,4,6]. Changes in the FTIR spectrum after Pb^{2+} ion adsorption suggest that the primary binding mechanisms involve ion exchange and complexation with functional groups on the adsorbent (Figure 1b) [5,6]. These findings align with the results of Zhao et al., where ion exchange mechanisms and interactions with surface groups were identified as key for Pb^{2+} removal from wastewater [6].

After the adsorption of Pb^{2+} ions onto the PLB surface, significant structural changes were observed in the SEM images (Figure 2). In addition to the bound Pb ions visible at specific locations, the surface exhibited more pronounced cracks and deeper pores, indicating modifications due to adsorption. The EDX analysis revealed that the PLB surface predominantly contained carbon (approximately 70%) and oxygen (around 20%), highlighting its carbon-rich structure and the presence of oxygen-containing functional groups, which are key contributors to metal ion adsorption, as supported by previous studies [1,3,5].

The EDX analysis further confirmed the adsorption mechanism involving ion exchange (Figure 2). The adsorption of Pb^{2+} ions led to a slight increase in the carbon content on the PLB surface, while the percentages of K, Mg, Ca, O, and N decreased. These changes in elemental composition provide valuable insights into the mechanisms of ion binding on the biochar surface [5,6]. The lead content increased significantly, from 0% to an average of 1.50%, demonstrating the PLB's adequate capacity for Pb^{2+} ion adsorption.

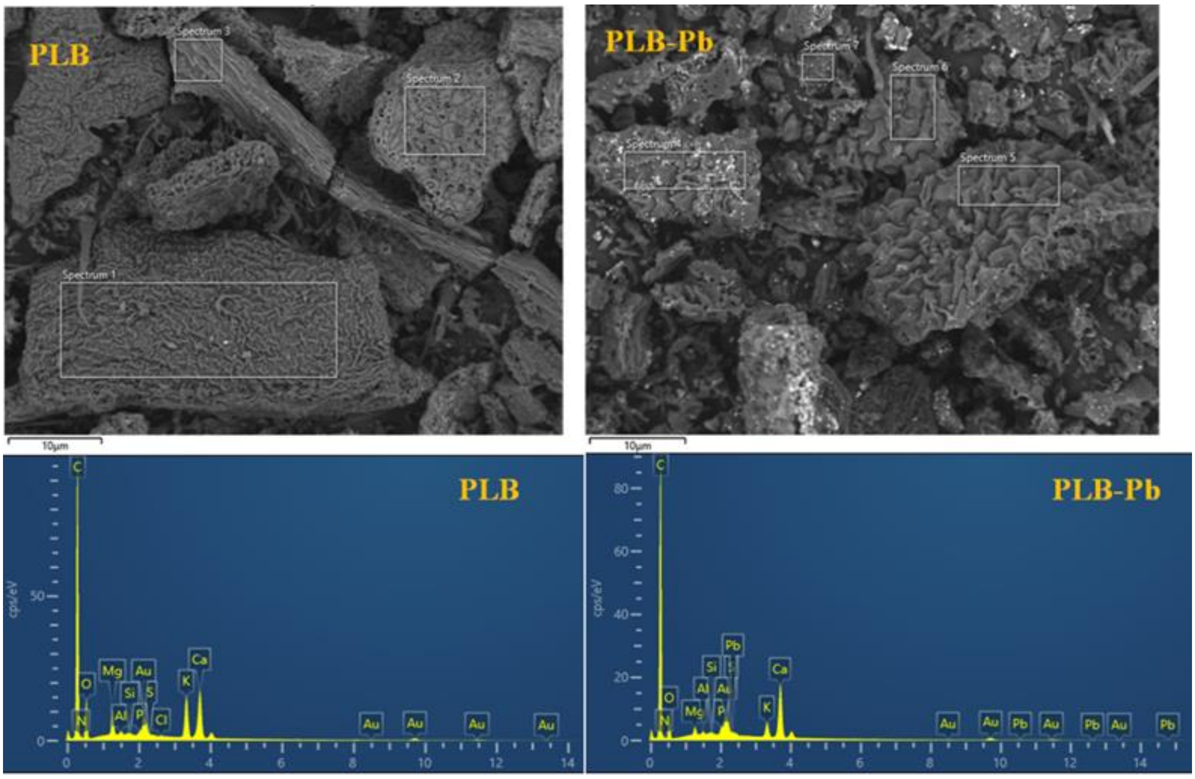


Figure 2. SEM-EDX analysis of the PLB surface before and after Pb^{2+} ions adsorption.

4. CONCLUSION

The results of the FTIR analysis showed differences in the intensity of the surface functional groups, especially the aromatic and oxygen functional groups. These changes confirm that the complexation and/or cation-π electron interaction are also done behind ion exchange. The SEM-EDX showed changes in the surface structure and elemental content. The surface exhibited newly formed cracks and pores during Pb^{2+} ion adsorption. Also, the EDX confirmed reduction of the O, N, K, Mg, and Ca amounts, while the Pb content increased significantly. The summarized results from this work revealed that leaves from trees could be valuable biomass for sustainable biochar production, and making them suitable for the adsorption of metal ions.

ACKNOWLEDGEMENTS

The authors are grateful to the Ministry of Education, Science and Technological development of the Republic of Serbia for financial support according to the contract with the registration number (e.g. 451-03-136/2025-03/200023).

REFERENCES

[1] M. Koprivica, M. Simić, J. Petrović, M. Ercegović, J. Dimitrijević, Processes, 11 (5) (2023) 1327.
[2] J. Petrović, M. Stojanović, J. Milojković, M. Petrović, T. Šoštarić, M. Laušević, M. Mihajlović, J. Environ. Manag., 182 (2016) 292-300.
[3] M. Ercegović, J. Petrović, M. Koprivica, M. Simić, M. Grubišić, N. Vuković, J. Krstić, Water, 16 (15) (2024) 2177.
[4] M-E. Lee, J.H. Park, J.W. Chung, Int. J. Environ. Res. Public Health, 14 (2017) 1528.
[5] C. Wang, X. Wang, N. Li, J. Tao, B. Yan, X. Ciu, G. Chen. Clean Technol., 4 (3) (2022) 629-652.
[6] M. Zhao, Y. Dai, M. Zhang, C. Feng, B. Qin, W. Zhang, N. Zhao, Y. Li, Z. Ni, Z. Xu, D. Tsang, R. Qiu, Sci. Total Environ., 717 (2020) 136894.