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# XXIII

## INTERNATIONAL CONGRESS

# VII

ENGINEERING, ENVIRONMENT AND MATERIALS  
IN PROCESS INDUSTRY  
EEM2021

PROCEEDINGS



JAHORINA  
MARCH 17-19, 2021

REPUBLIC OF SRPSKA  
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# **PROCEEDINGS**

## **VII INTERNATIONAL CONGRESS**

### ***ENGINEERING, ENVIRONMENT AND MATERIALS IN PROCESS INDUSTRY***

***EEM2021***

**UNDER THE AUSPICES OF  
MINISTRY OF SCIENTIFIC AND TECHNOLOGICAL DEVELOPMENT, HIGHER  
EDUCATION AND INFORMATION SOCIETY OF THE REPUBLIC OF SRPSKA  
AND  
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## INVASIVE ACER NEGUNDO L. BIOMASS AS LEAD SORBENT FROM AQUEOUS SOLUTION

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### Abstract

*Acer negundo L. is recognized as an invasive deciduous tree species that tends to grow and spread out of its natural range posing a threat to the biodiversity of the impacted areas. Although invasive plant species are considered to negatively affect ecosystems, their biomass can be used for environmental benefits. In order to find abundant and inexpensive sorbent for wastewater purification and metal pollution minimization, A. negundo leaf biomass was selected and investigated for that purpose. Since lead is well known as a common pollutant occurring in various industrial effluents with harmful effects on biota, it was selected for the sorption experiments. Acer negundo L. (AN) samples were collected at the unpolluted area in Mountain Avala, Serbia. After milling and sieving, the prepared sorbent was used in batch system sorption experiments. Experiments were conducted under isothermal conditions, by adding a precise amount of sorbent in the lead solution of known initial concentration. The adsorption performance of the obtained biomass-derived material (AN) was evaluated by testing several operational parameters: initial pH value, initial sorbent dosage, contact time, and initial lead concentration. The experimental parameters were optimized in order to determine the most appropriate conditions for Pb(II) removal. Maximum lead uptake occurs at pH 5, with 2g/L of sorbent dosage. The kinetic study revealed very fast adsorption, with equilibrium occurring after an initial 15 min of contact between sorbent (A. negundo leaf biomass) and sorbate (1mmol/L of lead solution). After this contact time, the residual metal concentration started to increase indicating the desorption process. Data obtained from sorption experiments were subjected to equilibrium modeling: they were fitted by two-parameter models (Langmuir and Freundlich). Results showed that the maximal adsorption capacity of a sorbent is 109 mg/g. Obtained results suggest that Acer negundo leaf biomass can be successfully applied as a lead sorbent upon the optimization of the operating parameters. Studies of this type provide valuable information for future water remediation technology development.*

**Keywords:** *invasive plant, Acer negundo, lead, sorption, wastewater treatment*

### Introduction

Invasive plant species are alien species that become established in natural habitats worldwide, where they are threatening native biodiversity and alter ecosystem processes such as primary productivity, decomposition rate, soil water regime, nutrient cycling, etc. (Vitousek et al., 1997).

Many of them are currently subjected to various control mechanisms, including their chemical or mechanical removal from the environment. *Acer negundo* L. is deciduous tree species originating from North America that invades natural and semi-natural areas across Europe. The higher level of phenotypic plasticity in biomass allocation in response to light and nutrient availability enables *A. negundo* to spread by outperforming many native tree species (Porté et al., 2011). Additionally, species *A. negundo* has been identified as one of the most aggressive invasive tree species in Serbian forest ecosystems (Medarević et al., 2008), including some protected natural areas such as mountain Avala near Belgrade (Glišić et al., 2014). Although invasive plant species are considered to negatively affect the ecosystem functioning, recent investigations show that their biomass can be utilized to provide certain environmental benefits, like bioenergy production, water pollution remediation, or soil conditioning (Van Merbeek et al., 2015; Ahmed et al. 2020). Recently, biosorption as a way to treat polluted waters through removing metal ions by using the dead biomass became one of the prominent remediation approaches. Biosorption, as a sustainable remediation technology, could potentially aid in the solution to the global and widespread problem of water pollution, especially concerning some of the most commonly found heavy metals in wastewaters such as lead, cadmium, arsenic, copper, and zinc (Jaishankar et al., 2014). Various biomaterials and plant-derived materials (e.g. bark, leaves, fruits, and seeds) have been investigated in this sense, aiming to reveal their efficacy, underlying sorption mechanism, and optimal parameters for their use. Invasive plant species could represent a promising source of biomass for such purposes. The aim of this study was to investigate the efficiency of invasive *A. negundo* leaf biomass as an adsorbent for a lead from aqueous media for the purpose of its remediation by assessing main physico-chemical interactions between pollutant and biomass and factors that are influencing metal removal.

## Materials and methods

Leaves of *Acer negundo* L. were harvested from the unpolluted area of Mountain Avala, Serbia. Collected biomass was thoroughly rinsed with tap and deionized water to remove impurities and air-dried for two weeks. The sample was grinded in a blade grinder (20,000-30,000 rpm) and sieved into particle size less than 0.2 mm. The obtained powder was labeled as AN.

A stock solution of lead (1 mmol/L) was prepared by dissolving a precise amount of  $\text{Pb}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  (analytical grade) in deionized water. From the stock solution, several solutions of different initial lead concentrations were prepared by dilution with deionized water. The surface functional groups in samples before and after lead sorption were analyzed by Attenuated Total Fourier Transform Infrared Spectroscopy using Thermo Nicolet 6700 FTIR. All the spectra were recorded in the range from 4000 to 400  $\text{cm}^{-1}$  with an average of 32 scans. Due to the strong absorption of diamond IR, the area between 1900 and 2200  $\text{cm}^{-1}$  was interrupted. Sorption experiments were performed in a batch system by adding a precise amount of sorbent into 100 mL glass flasks containing 50 mL of the lead solution of a specific concentration. Afterward, the mixture was shaken at 250 rpm in an orbital thermostatic shaker, followed by filtration. The filtrate was analyzed by using atomic absorption spectrophotometer AAS (Perkin Elmer AAnalyst 300). The effect of the initial pH value on lead sorption was investigated in the range from 2.0 to 5.0. The adjustment of the initial pH value of each solution was done by adding appropriate quantities of 0.01 mol/L  $\text{HNO}_3$  and 0.01 mol/L KOH. Effect of contact time on sorption capacity was investigated in the range from 2 to 180 min, while the effect of AN sorbent dosage was studied in the range from 1 to 20 g/L. Finally, the effect of initial lead concentration was investigated in the range from 5 to 325 mg/L. The sorption capacity was calculated using the following equation:

$$qe = \frac{(C_0 - C_e)V}{m} \quad (1)$$



$q$  - the amount of lead absorbed (mg/g);  $C_0$  and  $C_e$  - the initial and equilibrium lead concentrations (mg/L), respectively;  $V$  - the solution volume (L);  $m$  - the absorbent mass (g).

## Results and Discussion

In order to identify the functional groups on AN surface and potential active sites for lead binding, ATR-FTIR spectra had been recorded. The results of ATR-FTIR spectra of AN before and after sorption are presented in Figure 2. The peak located around 1730-1733  $\text{cm}^{-1}$  refers to the C=O stretching vibration (Rubeiro da Luz, 2006) and is commonly related to ester-containing compounds in the lipid membrane and cell walls pectin (Wu et al., 2020). Change of the absorption peak 1617  $\text{cm}^{-1}$  that refers to the amide I protein band (Gao et al., 2012) was noticed after lead binding. Distinct bands around 1000  $\text{cm}^{-1}$  indicated the involvement of the carbohydrates and C-O group in metal binding (Yang & Yen, 2002). Therefore, several functional groups and components, such as carbohydrates, esters, and proteins were involved in Pb binding of AN.

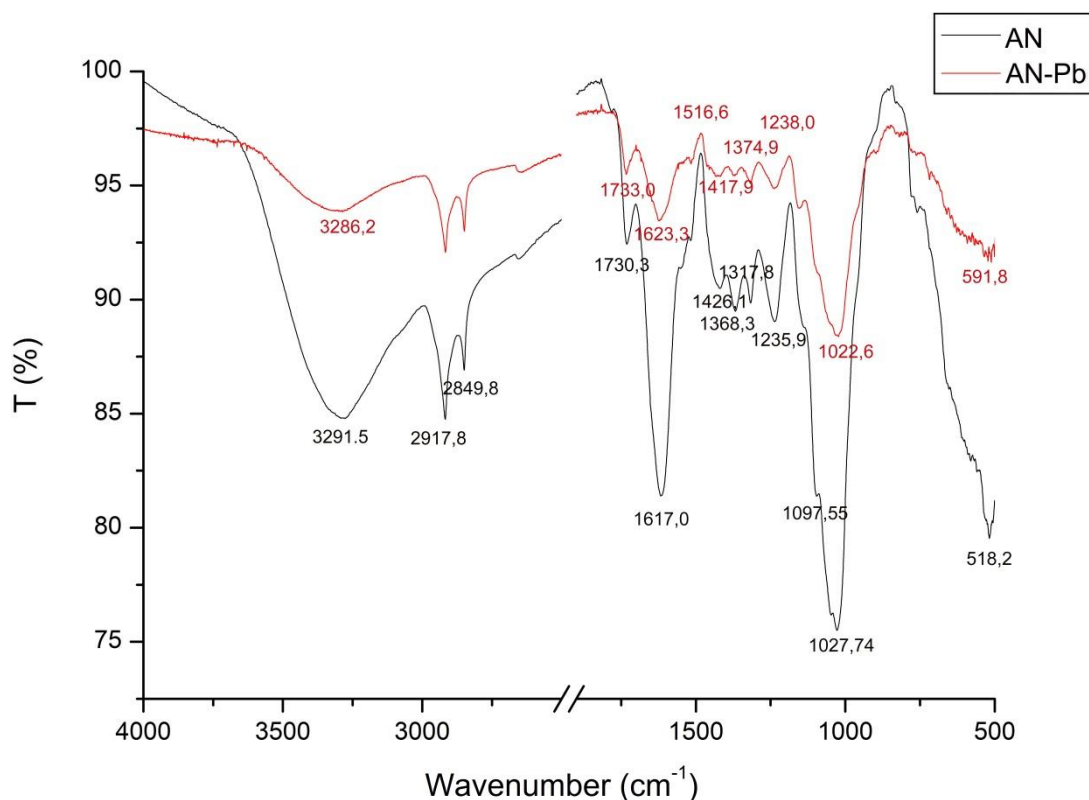


Figure 2. ATR-FTIR spectrum of AN before (black line) and after Pb(II) sorption (red line)

### Sorption experiments

Figure 3a shows that the sorption capacity of AN dramatically increases with increasing of pH values: from 20 mg/g at initial pH value 2.0 to 95.3 mg/g at initial pH value 5.0. At low pH, the concentration of protons in the solution is high and functional groups are protonated (Lopčić et al., 2013). Therefore, due to the strong competition between hydrogen and lead ions for the same active sites on the sorbent, AN affinity to bind lead ions is very low. The increase of solution pH leads to more available active sites on the surface resulting in higher sorption capacity. The effect of contact time on sorption capacity between AN and lead ions is presented in Figure 3b.

Very fast lead sorption phase occurs at initial several minutes, where the maximum capacity has been reached within 15 min. The first phase of the sorption process is very fast due to the large concentration gradient and the large numbers of available active sites on the surface (Stanković et al., 2019). Also, there was a noticeable decrease of initial pH value, which was 5.0 in the first 15 min of lead sorption, indicating strong release of  $H^+$  ions and strong competition between hydrogen and lead ions for the same active sites on the AN surface. However, in the second phase, sorption capacity started to decrease (from 95.5 mg/g in the 15<sup>th</sup> min to 81.5 at 180<sup>th</sup> min). Simultaneously the pH value of the solution started to decrease, from an initial 5.02 to 3.71 (after 15 minutes), indicating the presence of hydronium ions that compete with lead ions, and occurring of desorption process. The desorption process might be the consequence of weak physical sorption of electrostatic nature between AN and lead ions. With further contact time increase, the present hydrogen ions compete with the lead ions, forming more stable bonds with the sorbent material. Thus, the optimal contact time is considered to be 15 minutes for this sorbent type. The effect of sorbent dosage was investigated by varying the concentration of sorbent AN in solution and the results are presented in Fig.3c. The highest sorption capacity was achieved with a sorbent dosage of 1 g/L (101.5 mg/g), but the sorption efficiency is the lowest (near 60%). In literature, the decrease of sorption capacity with increasing the sorbent dosage can be explained by overlapping or aggregation of particles (Nadem et al. 2008). For further experiments, we have chosen a sorbent dosage of 2 g/L because both the sorption capacity and efficiency are sufficiently high (both are near 90%). As can be seen from Figure 3d sorption capacity increased together with an increase of initial lead concentration (from 2.4 mg/g at an initial lead concentration of 5mg/L to 96.25 mg/g at an initial lead concentration of 230 mg/L). The highest biosorption capacity was at the initial concentration of 230 mg/L for a sorbent dosage of 2 g/L.

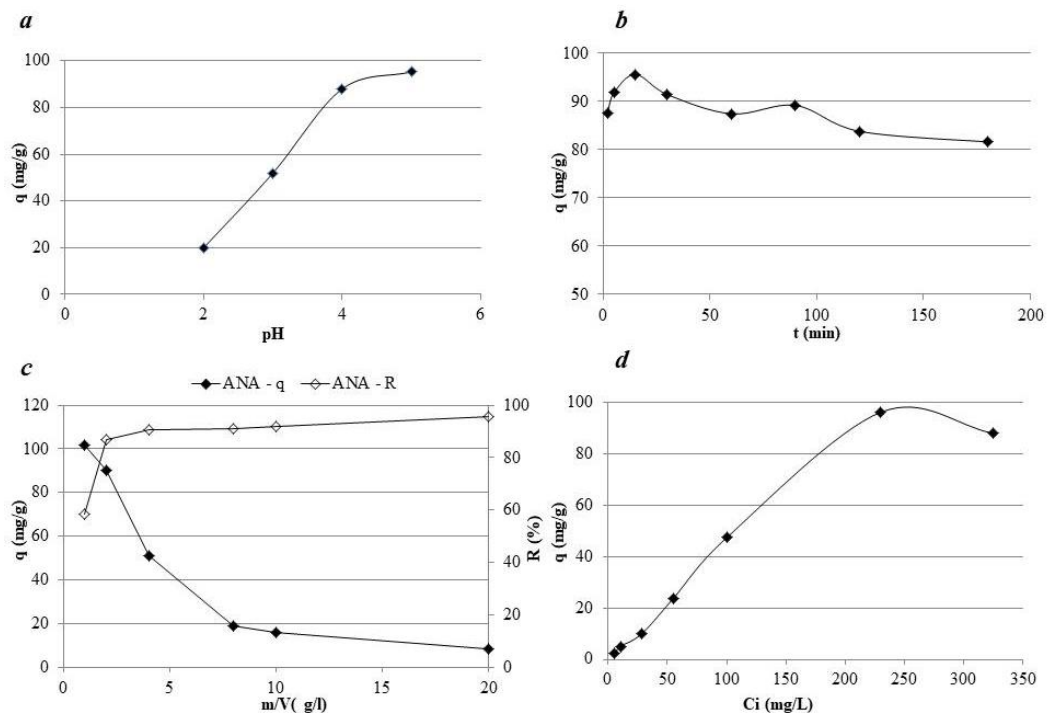


Figure 3. Process operative parameter: a) pH, b) contact time, c) biosorbent dosage and d) initial lead concentration

The involvement of the ion-exchange mechanism in the lead sorption process by AN was examined by following the release of exchangeable cations ( $Ca^{2+}$ ,  $Mg^{2+}$ ,  $Na^+$ ,  $K^+$ , and  $H^+$ ) from AN after the sorption process. The ratio between adsorbed lead ions and realised cations from AN was calculated according to a well-known equation (Šoštarić et al. 2015) and the results are



presented in Figure 4.

Results showed an almost equal release of cations from AN and bonding of lead ions from solution after the process of sorption, indicating the displacement of these cations by lead ions. Since the ratio between bonded and realised ions (Rb/r) is 1.07, the ion exchange mechanism can be the dominant mechanism in the lead sorption process by AN.

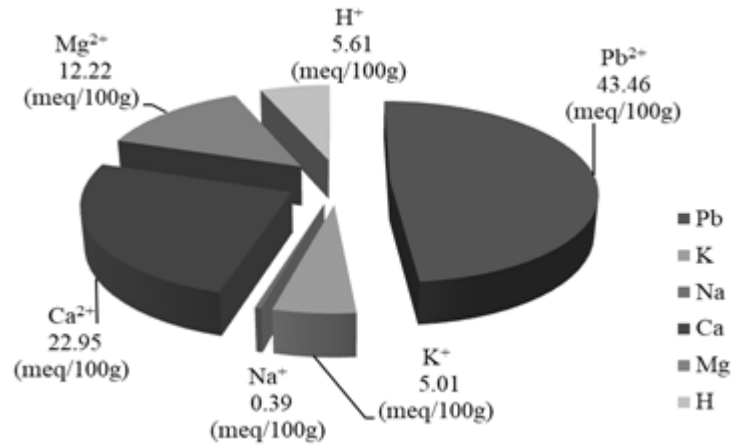


Figure 4. Ion exchange mechanism vs lead sorption ( $C_i=1\text{mMol/L}$ ,  $t=120\text{ min}$ ,  $m/V=2\text{g/L}$  and  $\text{pH } 5.0$ )

For a more profound understanding of the sorption mechanism, experimental data obtained from sorption experiments were fitted by two-parameter isotherm models (Langmuir and Freundlich) and the results are presented in Figure 5. Calculated parameters from isotherm models are presented in Table 1.

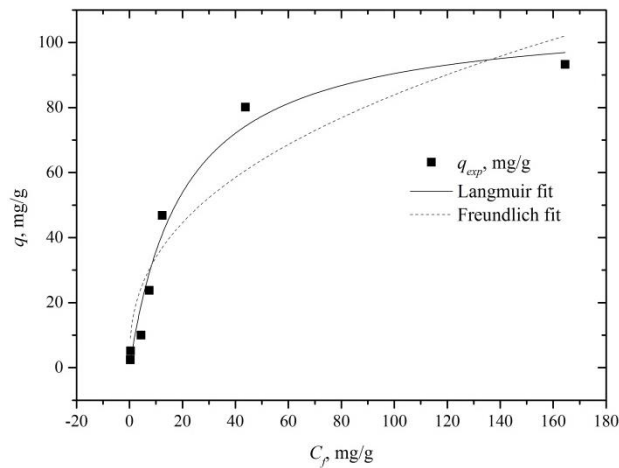


Figure 5. Langmuir and Freundlich isotherm models for the AN lead sorption (initial lead concentration: 5 - 325 mg/L, AN dosage: 2g/L, pH 5.0, contact time: 120 min, room temperature and stirring speed of 250 rpm).

Table 1. Isotherm parameters for Pb (II) sorption onto AN

Models	Parameters	AN
Langmuir	$q_m$ (mg/g)	108.9
	$K_L$ (L/mg)	0.05
	$\chi^2$	40.4
	$R^2$	0.970
Freundlich	$K_f$ (mg/g)(L/mg) <sup>1/n</sup>	13.7
	$1/n$	0.4
	$\chi^2$	174.1
	$R^2$	0.873

Due to the values of  $R^2$  it may be concluded that the adsorption of lead ions onto AN can be described with Langmuir models, indicating monolayer sorption and the heterogeneous surface of the sorbent. Results showed that the maximal adsorption capacity of a sorbent is 108.9 mg/g. Sorbents based on plant leaves powders are among the widely studied sorbents for the removal of various metal ions, due to their affordable availability, high sorption efficiency (capacity), and low cost. In Table 2, AN sorbent with other examined leaf sorbents for lead ions removal is compared.

Table 2. Comparison of the maximum sorption capacities of sorbents based on different plant leaves (Anastopoulos et al., 2019)

Plant leaves	Sorption capacity (mg/g)	Isotherm model	Ref.
<i>Syzygium cumini</i> L.	32.47	Langmuir	King et al., 2007
Phoenix tree	71.0	Langmuir	Liang et al., 2016
Black tea	19.7	Freundlich	Mohammed et al., 2016
<i>Ficus religiosa</i> L.	37.45	Langmuir	Qaiser et al., 2009
<i>Solanum melongena</i> L.	71.42	Langmuir	Yuvaraja et al., 2014
<i>Cinnamomum camphora</i> L.	75.82	Langmuir	Chen et al., 2010
<i>Dicerocaryum eriocarpum</i> Decne	41.9	Langmuir	Edokpayi et al., 2015
Cabbage	6.08	Langmuir	Kamar et al., 2017
Arborvitae	35.84	Langmuir	Shi et al., 2016
<i>Viscum album</i> L.	68.53	Langmuir	Suc and Son 2014
<i>Acer negundo</i> L.	<b>108.9</b>	Langmuir	<b>This study</b>

## Conclusions

In this study, the invasive plant *Acer negundo* L. was used as a sorbent for the removal of lead ions from the aqueous solution. FTIR analysis showed that several functional groups and components, such as carbohydrates, hydroxides, pectin, and proteins are involved in lead binding. Analysis of the cations released after the sorption shows that the number of cations released from AN was almost equal to the amount of sorbed lead ions indicating an ion-exchange mechanism. During the process of sorption Ca, Mg, and K ions were exchanged with the lead ions. This study provides valuable information for future water remediation technology development. Since the AN is a widely spread invasive plant, application of its leaf biomass as sorbent could present a feasible and sustainable remediation solution.

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