

## REMOVAL OF COPPER (II) FROM AQUEOUS SOLUTION BY BIOSORPTION ONTO POWDER OF JERUSALEM ARTICHOKE

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**Abstract.** Stalks' powder of Jerusalem artichoke (*Helianthus tuberosus L.*) was tested as an agricultural waste product for the removal of Cu (II) ions from aqueous solution. Batch experiments were carried out to evaluate the effects of pH, biosorbent dosage, initial metal concentration and contact time. The maximum removal efficiency of about 71 % was reached at pH 4.0 by using of 5 g/dm<sup>3</sup> biosorbent for 120, 90 and 60 min contact time, at 10, 50 and 100 mg/dm<sup>3</sup> initial Cu (II) concentration, respectively. Pseudo-first-order, pseudo-second-order, Elovich's equation and intraparticle diffusion models were applied to describe the obtained kinetic data. The pseudo-second-order model provided the best fit for experimental data with coefficient of determination greater than 0.99. Freundlich and Langmuir isotherm models were used to describe metal adsorption. Equilibrium data agreed well with Freundlich isotherm with  $R^2 = 0.968$ .

**Keywords:** biosorbent, heavy metals, copper ions, kinetics, isotherms.

### INTRODUCTION

Solid wastes generated due to agricultural by-products are high volume and creating essential disposal problem. So, it will be a significant contribution in the field of environmental protection if suitable methods are not developed for utilization of agricultural by-products there by minimizing the disposal problem [18]. On the other hand, in recent years increasing concern about the effect of toxic metals in the environment has resulted in more stringent environmental regulations for industrial operations that discharged metal-bearing effluents. It is well proved that copper is essential for good health, but it is also potentially toxic for humans when taken in excessive amounts [1]. Copper may be found as a contaminant of food, water and drinks since it is a widely used material. There are many actual and potential sources of copper pollution [2].

Nowadays, the removal of heavy metal ions from aqueous solutions based on the biotechnological approaches is consistent to green chemistry, simple and comparatively inexpensive. By-products or waste biomaterials are low cost and highly abundant materials which can be used as efficient biosorbents [6]. Biosorbents derived from plants constitute a wide range of materials that include visible parts of plants (shoots, bark, fruit, seed, stones, cones, hulls, husks, stalks, etc.). These materials are composed of biopolymers mainly various forms of lignocellulose and tannins that enable binding chemical compounds due to the presence in their structure of specific function groups [Witek-Krowiak, 2012]. Different types of biomass have been investigated for biosorption of heavy metals [21]. Biosorption of Cu (II) have been described in a wide range of biomass, like

microorganisms [19, 20], agricultural wastes and by-products [3, 4, 5, 10, 11, 12, 14, 15, 22].

It is observed that the heavy metal biosorption capacities depends on the type of biomass and is related to the metal ion chemical speciation in solution [13]. The existing knowledge on various aspects of the fundamentals and applications of biosorption and critically reviews the obstacles to commercial success and future perspectives have been made recently [8]. The biosorption mechanism may be complex and each material has its peculiarities like chemical structure and porosity, which preconditions its sorption capacity of the sequestered compounds. The effectiveness of biosorption depends also on the physical and chemical parameters, such as pH, temperature, etc. Each biosorbent reacts in its own way under given conditions.

The interest to the Jerusalem artichoke (*Helianthus tuberosus L.*) as an agricultural product has become considerable recently. Unfortunately, there are not observed many investigations concerning biosorption properties of the Jerusalem artichoke by-products upon heavy metal ions removal from aqueous solution [5, 16].

This paper puts forward the use of Jerusalem artichoke stalks, as an agricultural waste material, for biosorption of Cu (II) ions from aqueous solution. Research was made into the impact of different process conditions (pH, adsorbent dosage, initial metal concentration and reaction time) on the biosorption. The biosorption kinetics was mathematically described by means of models of the order of reaction and intraparticle diffusion. Two of the most popular models (Langmuir and Freundlich isotherm) were applied for describing the equilibrium.

## MATERIALS AND METHODS

### Biosorbent preparation

In the present study the powder obtained from stalks of Jerusalem artichoke by cutting, drying (40°C), milling and sieving (laboratory sieve 0.80 mm) was used. For the removal of certain interfering components (pigments, etc.) the plant material was extracted twice with distilled water (1:7) for 45 min at 25°C under continuously stirring. After that the material was dried at 40°C.

### Preparation of copper solution

Stock solution (1000 mg/dm<sup>3</sup>) of Cu (II) was prepared by dissolving of required quantity of CuSO<sub>4</sub>·5H<sub>2</sub>O in distilled water. It was further diluted to obtain desired concentrations of working solutions for the batch experiments study.

### Biosorption batch experiments

The pH value of the samples was adjusted by adding 0.1 M NaOH or HCl solutions. All chemical reagents used in the experiments were of analytical grade. Biosorption experiments were carried out in 250 cm<sup>3</sup> Erlenmeyer glass flasks with 100 cm<sup>3</sup> volume of copper solution. Batch experiments were conducted by varying the pH value (2, 3, 4, 5, 6, 7, 8, 9), initial copper (II) concentration (10, 50 and 100 mg/dm<sup>3</sup>) and amount of biosorbent (from 1.0 to 40.0 g/dm<sup>3</sup>). Experiments were carried out at contact time of 24 h in order to reach equilibrium, agitation speed 100 rpm and ambient temperature 20.0±0.5°C. All experiments were performed in triplicate. The data were analyzed and presented as mean values.

### Analytical methods

For determination of Cu (II) concentration in the solutions before and after biosorption, samples were withdrawn, filtered and filtrate was analyzed. Metal concentration was determined spectrophotometrically at 605 nm wavelength.

The metal uptake  $q$  (mg/g) was determined by employing the mass balance. If  $C_0$  and  $C_e$  are the initial and final metal concentration (mg/dm<sup>3</sup>), respectively;  $V$  (dm<sup>3</sup>) is the initial volume of copper solution and  $m$  (g) is the mass of biosorbent material, the equilibrium metal uptake  $q_e$  (mg/g) can be calculated as:

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

The performance of biosorption was evaluated in terms of its removal efficiency as RE (%), estimated by the following equation:

$$RE = \frac{(C_0 - C_t)}{C_0} \cdot 100 \quad (2)$$

where  $C_t$  is the copper (II) concentration at time  $t$ .

### Kinetic experiments

Batch kinetic experiments were carried out at pH 4.0 and 20.0±0.5°C. For this purpose, 0.5 g of biosorbent were contacted with 100 cm<sup>3</sup> of copper (II) aqueous solution with initial metal concentration 10, 50 and 100 mg/dm<sup>3</sup> in 250 cm<sup>3</sup> Erlenmeyer glass flasks on a magnetic stirrer at 100 rpm. At different time intervals ranging from 10 to 150 min the concentrations of copper (II) in the treated solutions were determined as described in analytical methods.

### Isotherm experiments

Equilibrium sorption experiments were performed as follow. Biosorbent (0.5 g) were exposed to 100 cm<sup>3</sup> copper (II) solution with an initial concentrations from 5.0 to 100.0 mg/dm<sup>3</sup> at constant pH 4.0, agitation time 100 rpm and ambient temperature 20.0±0.5°C for 24 h in order to reach equilibrium. Sorption isotherm is plotted of the sorbate uptake ( $q_e$ ) versus the equilibrium concentration of the residual sorbate remaining in the solution ( $C_e$ ).

## RESULTS AND DISCUSSION

*The influences of batch study parameters, such as pH of solution, biosorbent dosage, contact time and initial copper concentration on the biosorption process were investigated.*

The effect of pH in the solution on the Cu (II) removal efficiency was studied at different pH ranging from 2.0 to 9.0 and results are illustrated in Fig. 1.

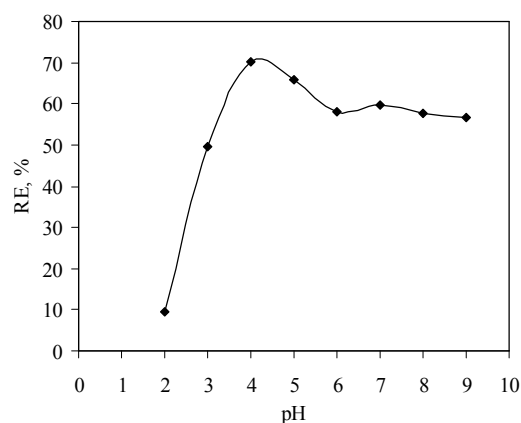


Fig. 1. Effect of pH on the removal of Cu (II) from aqueous solution by powder of Jerusalem artichoke stalks (initial copper concentration 10 mg/dm<sup>3</sup>, 20°C, biosorbent dosage 2 g/dm<sup>3</sup>)

Hydrogen ion concentration (pH) of the aqueous solutions affects the surface charge of sorbent and the degree of speciation and ionization of sorbent during adsorption. The maximum removal efficiency (70.2 %) was reached at pH 4.0 (Fig. 1). Similar result was obtained by other research for biosorption of copper onto treated rubber leaves [14].

Another important parameter is biosorbent dosage. To investigate the effect of biosorbent dosage on biosorption, the experiments were conducted with constant copper concentration (10 and 100 mg/dm<sup>3</sup>), and samples with different biosorbent dosage ranging from 1 to 40 g/dm<sup>3</sup> (solid/liquid) were used under the constant temperature 20°C and pH 4.0. Data are given in Figure 2. The obtained results show that removal efficiency of Cu (II) ions increased as the biosorbent amount increases and then decreases and finally becomes constant. The maximum removal efficiency of 70.6 % and 71 % was obtained by using of 5 g/dm<sup>3</sup> biosorbent dose at 10 mg/dm<sup>3</sup> and 100 mg/dm<sup>3</sup> initial metal concentration, respectively.

These results are in good accordance to those obtained by another research, who studied the copper biosorption on chemically modified orange peels [12].

Concerning the effects of initial copper concentration and contact time, the plots (Fig. 3) showed that the biosorption of copper can be divided into two main stages. The initial rapid stage where biosorption was fast and second slow stage which refers to gradual biosorption before copper uptake reached equilibrium. A similar phenomenon was observed by others also [4, 14, 20].

As shown in Figure 3, the biosorption capacities of Jerusalem artichoke stalks powder at equilibrium increased from 1.412 to 14.180 mg/g as the copper concentrations increased from 10 to 100 mg/dm<sup>3</sup>. The times taken to reach equilibrium for copper concentrations of 10, 50 and 100 mg/dm<sup>3</sup> were 120, 90 and 60 min and the maximum removal efficiencies were 70.6, 70.6 and 70.9 %, respectively.

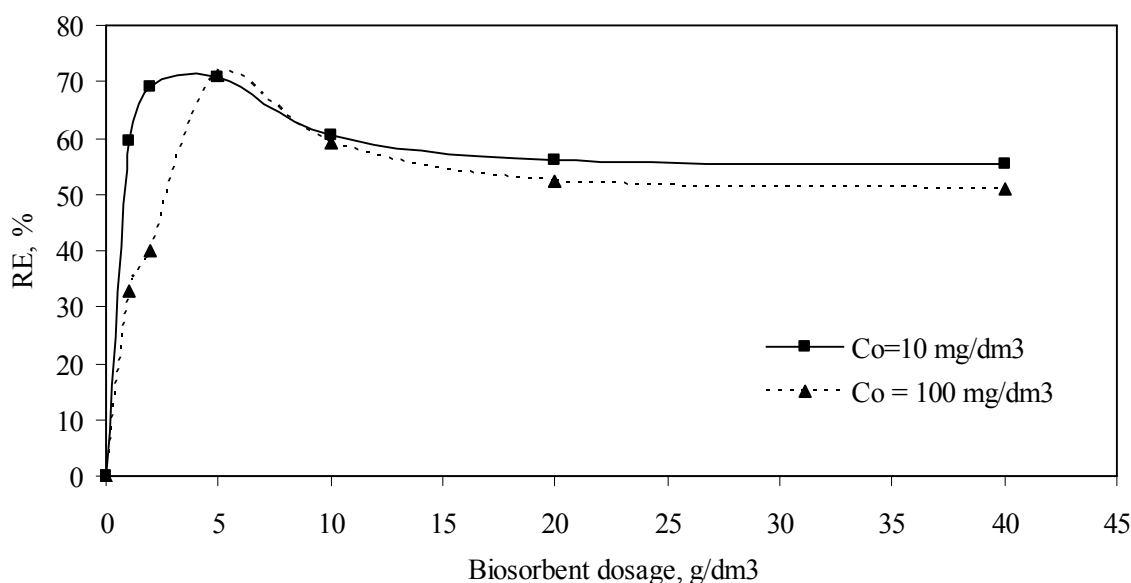


Fig. 2. Effect of biosorbent dose on the Cu (II) removal from aqueous solution by powder of Jerusalem artichoke stalks (20°C, pH 4.0)

The following kinetic models were used to model experimental data:

The pseudo-first-order model is expressed as:

$$\frac{dq}{dt} = K_{1,ads} (q_e - q) \quad (3)$$

where  $q_e$  (mg/g) and  $q$  are amounts of adsorbed metal ions on the biosorbent at the equilibrium and any time  $t$ , respectively; and  $K_{1,ads}$  is the Lagergren

rate constant of the first-order biosorption (min<sup>-1</sup>). The model is based on the assumption that the rate is proportional to the number of free site [7]. Integrating Eq. (3) between the limits,  $t=0$  to  $t=t$  and  $q=0$  to  $q=q$  yields the linearized version of this model:

$$\log(q_e - q) = \log q_e - \frac{K_{1,ads} \cdot t}{2.303} \quad (4)$$

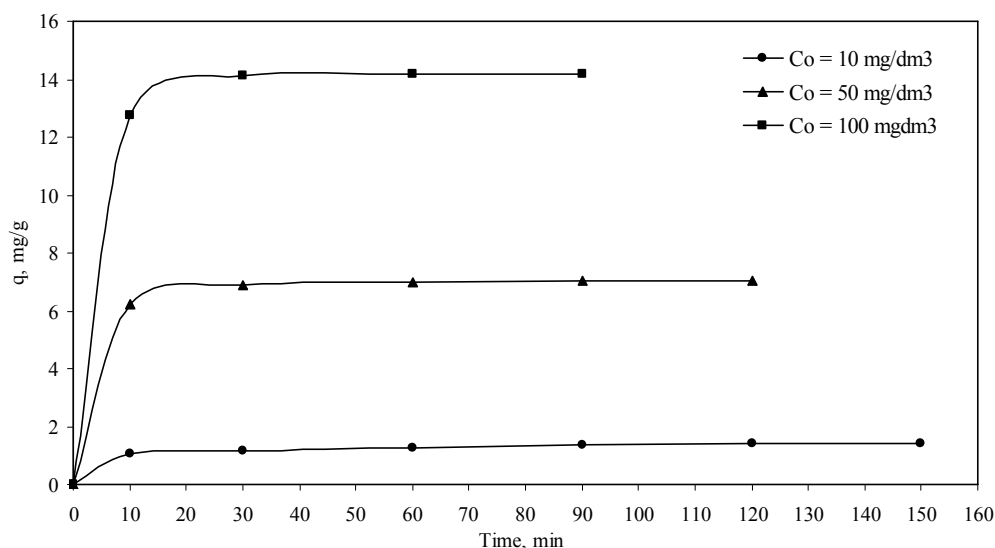


Fig. 3. Effects of initial metal concentration and contact time on the Cu (II) biosorption by powder of Jerusalem artichoke stalks (biosorbent dosage 5 g/dm<sup>3</sup>, 20°C, pH 4.0)

Linear plots of  $\log(q_e - q)$  versus  $t$  were plotted to evaluate this kinetic model and to determine rate constant and  $q_e$  from the slope and intercept, respectively.

The pseudo-second-order model is based on the assumption that biosorption follows a second-order mechanism, whereby the rate of sorption is proportional to the square of the number of unoccupied sites [7]:

$$\frac{dq}{dt} = K_{2,ads}(q_e - q)^2 \quad (5)$$

where  $K_{2,ads}$  is the rate constant of second-order biosorption (g/mg.min). Integrating Eq. (5) from  $t=0$  to  $t=t$  and  $q=0$  to  $q=q$  and linearization yields:

$$\frac{t}{q} = \frac{1}{K_{2,ads} \cdot q_e^2} + \frac{t}{q_e} \quad (6)$$

The parameters  $q_e$  and  $K_{2,ads}$  are calculated from the slope and the intercept of the plot  $t/q$  versus  $t$ . It is not necessary to independently determine  $q_e$  to apply this model.

A linearized plots for the pseudo first-order Lagergren and pseudo second-order models are shown in Fig. 4 and 5.

The Elovich's equation is of general application to chemisorption kinetics. This equation is often valid for systems in which the adsorbing surface is heterogeneous, and is formulated as:

$$q = \frac{1}{b} \ln(a \cdot b) + \frac{1}{b} \ln t \quad (7)$$

where  $a$  (mg/g/min) is the initial adsorption rate and  $b$  is related to the extent of surface coverage and the activation energy involved in chemisorption (g/mg).

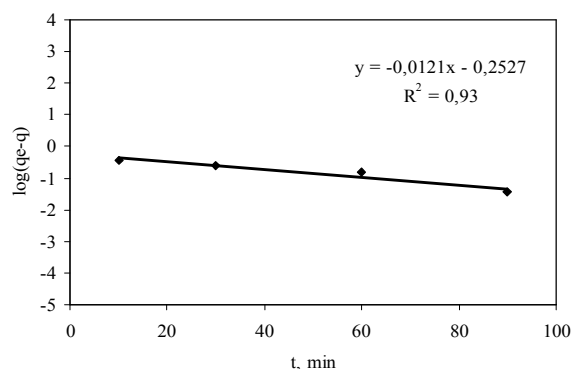


Fig. 4. Linearized pseudo first-order model for the biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks ( $C_0 = 10$  mg/dm<sup>3</sup>)

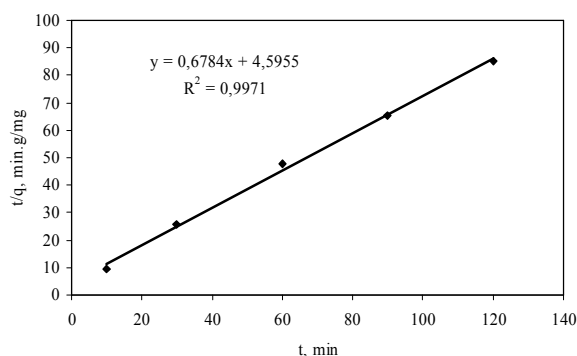


Fig. 5. Linearized pseudo second-order model for the biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks ( $C_0 = 10$  mg/dm<sup>3</sup>)

The intraparticle diffusion equation is given as:

$$q = K_d \sqrt{t} + C \quad (8)$$

where  $K_d$  (mg/g/min<sup>-1</sup>) is the intraparticle diffusion rate constant and  $C$  (mg/g) is a constant that gives

idea about the thickness of the boundary layer.

The Elovich's model plot is presented in Figure 6, while Figure 7 represents the plot of intraparticle diffusion model.

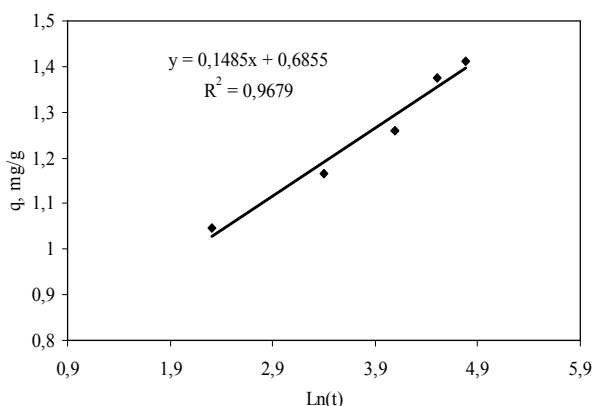


Fig. 6. Elovich's model plot for the biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks ( $C_0 = 10 \text{ mg/dm}^3$ )

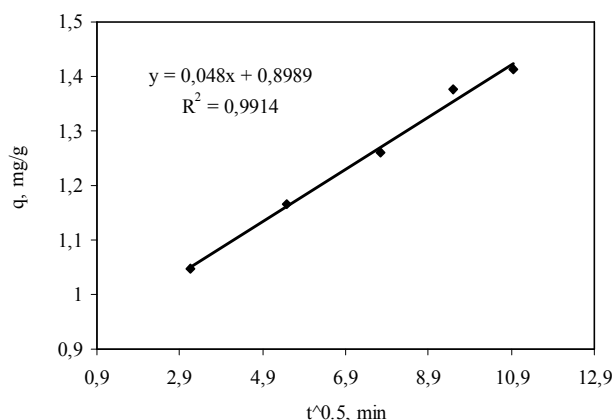


Fig. 7. Plot of the intraparticle diffusion model for the biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks ( $C_0 = 10 \text{ mg/dm}^3$ )

Figure 8 plots metal uptake rate versus time. Kinetic data was fitted onto four kinetic models with acquired parameters listed in Table 1.

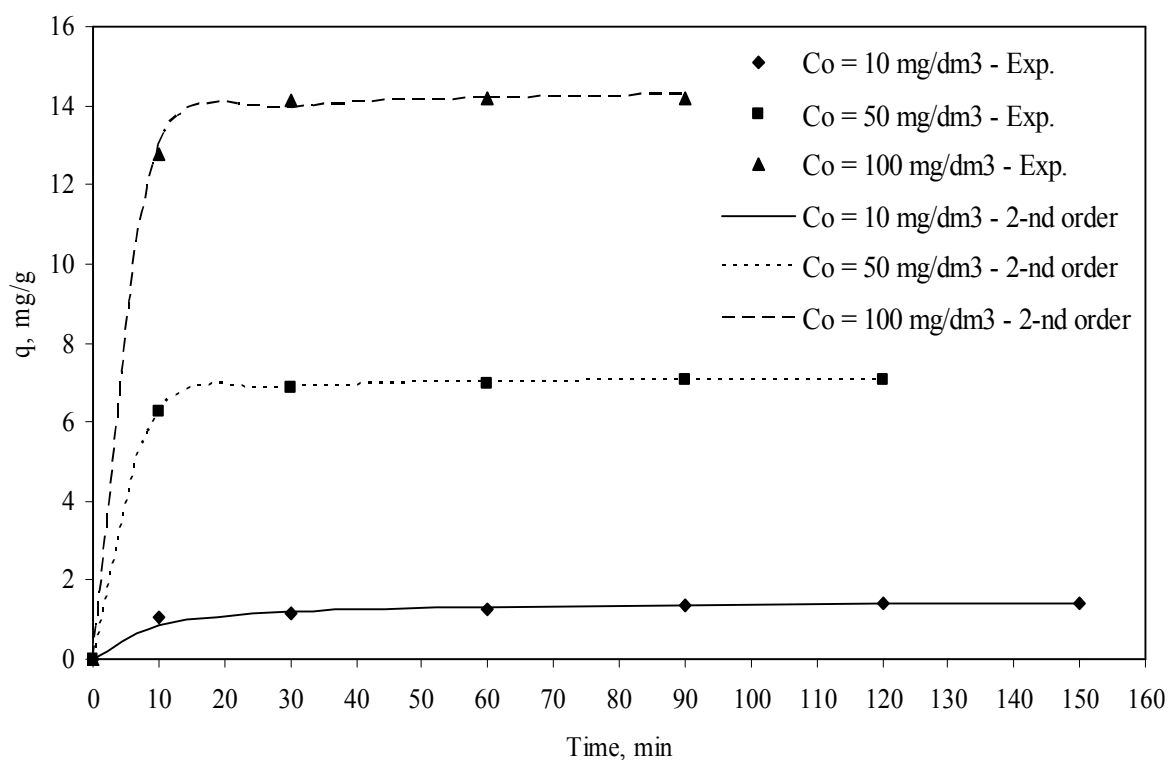


Fig. 8. Biosorption kinetics of Cu (II) ions on powder of Jerusalem artichoke stalks (biosorbent dosage  $5 \text{ g/dm}^3$ ,  $20^\circ\text{C}$ , pH 4.0) and model fitted using pseudo second-order model

By comparing the fitting results by pseudo first-order, second-order, Elovich's and intraparticle diffusion kinetic models, pseudo second-order model

seems to give better representation. Moreover, calculated  $q_e$  values from pseudo second-order model agree quite well with experimental data (Table 1).

Table 1. Kinetic parameters for the biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks

| $C_0$ , mg/dm <sup>3</sup> | $q_e^{exp}$ , mg/g | Pseudo first-order model |                                 |       | Pseudo second-order model |                        |       | Elovich's model |              |       | Intraparticle diffusion model  |       |
|----------------------------|--------------------|--------------------------|---------------------------------|-------|---------------------------|------------------------|-------|-----------------|--------------|-------|--------------------------------|-------|
|                            |                    | $q_e$ , mg/g             | $K_{1,ads}$ , min <sup>-1</sup> | $R^2$ | $q_e$ , mg/g              | $K_{2,ads}$ , g/mg.min | $R^2$ | $a$ , g/g.min   | $1/b$ , mg/g | $R^2$ | $K_d$ , mg/g.min <sup>-1</sup> | $R^2$ |
| 10                         | 1.41               | 0.56                     | 0.027                           | 0.930 | 1.47                      | 0.100                  | 0.997 | 0.02            | 0.149        | 0.968 | 0.048                          | 0.991 |
| 50                         | 7.06               | 1.02                     | 0.045                           | 0.919 | 7.17                      | 0.098                  | 1.000 | 875             | 0.372        | 0.916 | 0.123                          | 0.817 |
| 100                        | 14.2               | 2.03                     | 0.082                           | 0.905 | 14.47                     | 0.062                  | 0.999 | 548             | 0.821        | 0.879 | 0.306                          | 0.787 |

Equilibrium adsorption isotherms are of fundamental importance in the design of adsorption systems since they indicate how metal ions are distributed between the adsorbent and liquid phases at equilibrium as a function of metal concentration. There are many equations for analyzing experimental adsorption equilibrium data. The equation parameters and the underlying thermodynamic assumption of these equilibrium models often provide some insight into both the adsorption mechanism and the surface properties and affinity of the sorbent. In this work, the following two of the most frequently used models were tested [7,17].

The Langmuir isotherm model describes quantitatively about the formation of a monolayer adsorbate on the outer surface of the adsorbent and after that no further adsorption takes place. The Langmuir represents the equilibrium distribution of adsorbate between the solid and liquid phases.

The Langmuir equation is formulated as:

$$q_e = q_{max} \frac{K_L \cdot C_e}{1 + K_L \cdot C_e} \quad (9)$$

where  $C_e$  (mg/dm<sup>3</sup>) and  $q_e$  (mg/g) are the equilibrium concentrations in the liquid and solid phase, respectively,  $q_{max}$  is a Langmuir constant that express the maximum metal uptake (mg/g) and  $K_L$  (dm<sup>3</sup>/mg) is also a Langmuir constant related to the energy of adsorption and affinity of the sorbent.

The following linearized form of Langmuir equation was applied in this study:

$$\frac{1}{q_e} = \left( \frac{1}{K_L q_{max}} \right) \cdot \frac{1}{C_e} + \frac{1}{q_{max}} \quad (10)$$

The plot of  $1/q_e$  versus  $1/C_e$  has a slope with the value of  $1/(K_L q_{max})$  and an intercept magnitude of  $1/q_{max}$ .

The Freundlich equilibrium isotherm equation is an empirical equation used for the description of

multilayer adsorption with interaction between adsorbed molecules. This equation is widely applied in heterogeneous systems:

$$q_e = K_F \cdot C_e^{1/n} \quad (11)$$

where  $K_F$  (mg/g) and  $n$  are Freundlich constants indicating the adsorption capacity and adsorption intensity, respectively.

Linearized form of Freundlich equation was applied to the biosorption equilibrium data:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (12)$$

The plot of  $\log q_e$  versus  $\log C_e$  has a slope with the value of  $1/n$  and an intercept magnitude of  $\log K_F$ .

Experimental adsorption equilibrium data of copper biosorption onto powder of Jerusalem artichoke stalks is depicted in Figure 9. The obtained data were applied to the Langmuir and Freundlich isotherms using linear expression of these models (Figs. 10 and 11).

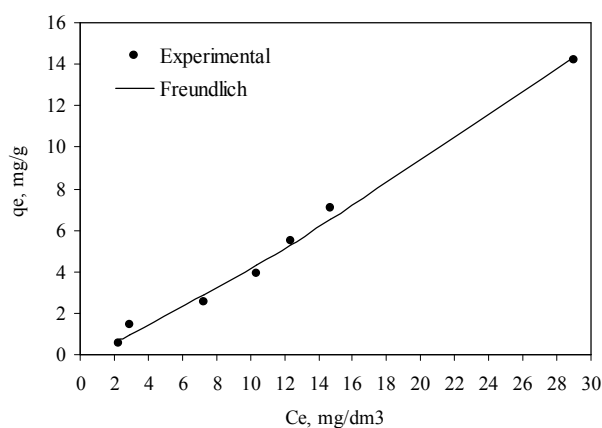


Fig. 9. Biosorption isotherms of Cu (II) ions onto powder of Jerusalem artichoke stalks (biosorbent dosage 5 g/dm<sup>3</sup>, 20°C, pH 4.0 and 100 rpm) and fitted Freundlich model

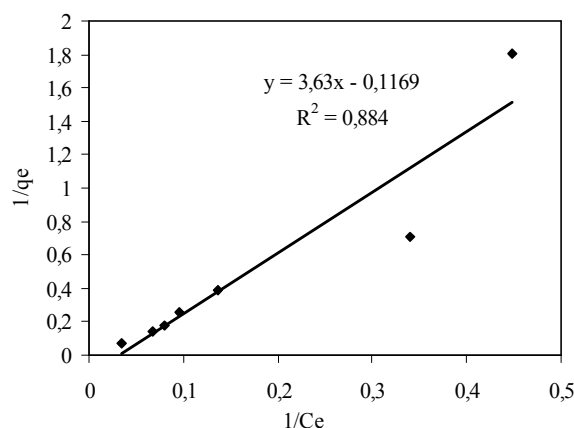


Fig. 10. Linearized Langmuir isotherm for biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks

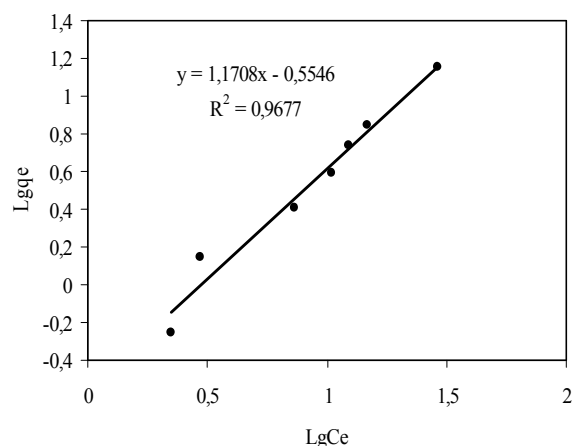


Fig. 11. Linearized Freundlich isotherm for biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks

Model parameters were determined using linear regression toolbox in Microsoft Excel software.

Table 2 summarized the results.

Table 2. Isotherm constants of Langmuir and Freundlich model for biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks

| Langmuir isotherm   |                 |       | Freundlich isotherm |                 |       |
|---------------------|-----------------|-------|---------------------|-----------------|-------|
| $q_{max}$ ,<br>mg/g | $K_L$ ,<br>L/mg | $R^2$ | $1/n$               | $K_F$ ,<br>mg/g | $R^2$ |
| 8.55                | 0.032           | 0.884 | 1.17                | 0.279           | 0.968 |

The regression parameters and coefficients of determination ( $R^2$ ) presented in Table 2, Figures 10 and 11, indicate that the adsorption data best fitted the Freundlich adsorption isotherm. At present, Freundlich isotherm is widely applied in heterogeneous systems. The slope ranges between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. Whereas, a value below unity implies chemisorptions process where  $1/n$  above one is an indicative of cooperative adsorption [9]. The obtained value of  $1/n$  was 1.17 (Table 2) which indicates that biosorption of Cu (II) ions onto powder of Jerusalem artichoke stalks is cooperative.

## CONCLUSION

This research showed that powder of Jerusalem artichoke (*Helianthus tuberosus* L.) stalks is a potential biosorbent for removal of Cu (II) from aqueous solution. The batch study parameters, such as pH of solution, biosorbent dosage, contact

time and initial copper concentration were found to be important parameters on the biosorption process. Finally, the experimental results of present study indicated maximum removal efficiency of copper (II) ions was 71 % at pH 4.0, biosorbent dosage 5 g/dm<sup>3</sup>, temperature 20°C and agitation speed 100 rpm. The kinetic study indicated that the biosorption data fit well to the pseudo second-order kinetic model ( $R^2 > 0.99$ ). The adsorption isotherm followed Freundlich model ( $R^2 = 0.968$ ). However, more detailed studies are needed to clarify the Cu (II) biosorption mechanism by powder of Jerusalem artichoke.

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## ОТСТРАНЯВАНЕ НА $\text{Cu(II)}$ ЙОНИ ОТ ВОДНИ РАЗТВОРИ ЧРЕЗ БИОСОРБЦИЯ ВЪРХУ БРАШНО ОТ ТОПИНАМБУР

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**Резюме.** Брашно от стебла на топинамбур (*Helianthus tuberosus L.*) е тествано за отстраняване на  $\text{Cu (II)}$  йони от водни разтвори, в качеството си на отпадъчен биосорбент. Проведени са експерименти за установяване влиянието на pH, количеството биосорбент, началната концентрация на метала и времето за контакт върху процеса на биосорбция. Максимална ефективност на отстраняване на  $\text{Cu (II)}$  от около 71 % е постигната при pH 4,0, 5 g/dm<sup>3</sup> биосорбент и време за контакт 120, 90 и 60 min, съответно при 10, 50 и 100 mg/dm<sup>3</sup> начална концентрация на медните йони в разтвора. За описание кинетиката на биосорбция са приложени четири кинетични модела. Установено е, че моделът от псевдо-втори ред най-добре описва получените кинетични данни, с коефициент на детерминация  $R^2 > 0,99$ . Получената равновесна адсорбционна изотерма се описва по-добре с модела на Фройндлих ( $R^2 = 0,968$ ), в сравнение с този на Лангмюир.

**Ключови думи:** биосорбент, тежки метали, медни йони, кинетика, изотерми.

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