Kinetic and isotherm studies for the biosorption of nickel ions by jute fabrics

ALEKSANDRA IVANOVSKA[1]*#, LEPOSAVA PAVUN[2]#, BILJANA DOJČINOVIĆ[3]
and MIRJANA KOSTIĆ[4]#

[1]University of Belgrade, Innovation Center of the Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia, [2]University of Belgrade, Faculty of Pharmacy, Vojvode Stepe 450, 11000 Belgrade, Serbia, [3]University of Belgrade, Institute of Chemistry, Technology and Metallurgy, Njegoševa 12, 11000 Belgrade, Serbia and [4]University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

Abstract: Raw jute fabric was chemically modified with 1 % NaOH for 30 min, 17.5 % NaOH for 5 min or with 0.7 % NaClO$_2$ for 30 and 60 min to study the influence of changes in the chemical composition of the fabrics and quantity of carboxyl groups on the biosorption capacity of the fabrics for nickel ions. The effects of contact time and initial concentration of nickel ions on the biosorption capacity of the fabrics were also investigated. The obtained results revealed that the biosorption of nickel ions can be explained by the pseudo-second-order kinetic model, while the experimental isotherm data fit better with the Langmuir model. The calculated ratios between maximal biosorption capacity (6.30–12.06 mg g^{-1}) of the jute fabrics and carboxyl group quantity indicated that approximately half of the carboxyl groups of the fabrics' would be involved in binding nickel ions during biosorption. Therefore, the quantity of carboxyl groups can be used to predict the maximal biosorption capacity of jute fabrics toward nickel ions. In the case of oxidized jute fabrics, the lignin removal, and consequently increased content of cellulose and hemicelluloses, also contributed to a higher biosorption capacity.

Keywords: chemical composition; carboxyl groups; hemicelluloses; lignin; pseudo-second-order; Langmuir model.

INTRODUCTION

Industrial expansion enables the detection of numerous environmental problems and contributes to improving the quality of the environment by applying new technologies to solve existing problems. In many instances, such problems are

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historic pollution and the presence of heavy metals in an environment posing a specific threat since they cannot be degraded. Among other heavy metals, considerable amounts of nickel ions are present in the wastewaters generated from mining, electroplating, pigments and ceramic industries. The presence of these ions in aqueous systems at trace levels has become a worldwide health and environmental concern due to the possibility of entering the food chain.

Various physicochemical methods have already been developed for the removal of heavy metal ions from wastewaters. Some of them (such as precipitation and coagulation) are less efficient and create sludge, while others (such as ion-exchange and reverse osmosis) are effective and efficient, but expensive. Additionally, these conventional methods are found to be inappropriate or ineffective for the removal of trace metals indicating the necessity for finding alternative wastewater treatments. Nowadays, biosorption using lignocellulosic waste has received remarkable attention since it is very efficient for absorbing heavy metals present in wastewaters at trace levels, *i.e.*, below 100 mg l^{-1}.[1] Besides the mentioned advantage, biosorption using lignocellulosic waste is economically feasible (use of a low-cost resource for applications in pollutant removal), technologically simple (simple equipment; can be applied to different flow regimes), and capable of treating large volumes.[2] On the other hand, lignocellulosic materials are renewable, biodegradable, and environmentally friendly resources. They are widely available in many countries, can be used in different forms (powders, fibres, felts, steams), can be easily modified by different chemical treatments and grafting reactions, and possess outstanding metal-binding capacities.[2] After use, these biosorbents can be burned and the metals recovered (*e.g.*, for catalysis), while the lignocellulosic material as abundant and low-cost waste can be used to prepare activated carbons.

It was shown earlier that alkali and oxidative chemical modifications significantly improved the sorption[3,4] and electrokinetic[5] properties of jute fabrics. Moreover, such prepared jute fabrics could be successfully used as protective clothes or textile of specific behaviour in environments sensitive to electrical discharges and home textiles due to their improved dielectric properties. Taking into account the wide variety of utilizations of the fabrics, the amount of jute waste having improved sorption properties has considerably increased. Therefore, the objective of the current study was to up value jute fabric waste by its utilization as a biosorbent for wastewater treatment. Recycling in the way of producing filters for wastewaters minimizes the disposal costs of such fabrics thus contributing to "closing the loop" of their lifecycle, which is in agreement with the circular economy concept.

This study reports on the efficiency of chemically modified jute fabrics (derived as waste from other industries) for the removal of nickel ions under different experimental conditions, *i.e.*, initial nickel ion concentration and contact

time. Thereafter, the experimental data were correlated to different kinetic and adsorption isotherm models. Moreover, a correlation between the jute biosorption capacity and its molecular structure (*i.e.*, contents of cellulose and non-cellulosic components as well as quantity of COOH groups) was investigated in detail.

EXPERIMENTAL

Material

Raw jute fabric (sample denoted as RJ) was chemically modified with 1 % NaOH for 30 min (sample A1/30) or with 17.5 % NaOH for 5 min (sample A17.5/5) at room temperature. These modifications were followed by neutralization with 10 % CH_3COOH, washing with distilled water, rinsing with 0.5 % $NaHCO_3$, washing with distilled water, and drying at room temperature. On the other hand, oxidative modifications of raw jute fabric with 0.7 % $NaClO_2$ (pH 4–4.5) at boiling temperature for 30 or 60 min (samples marked as C30 and C60, respectively) were followed by washing with distilled water, rinsing with 2 % $NaHSO_3$, washing and drying at room temperature.

Characterization of obtained jute biosorbents

The chemical composition of raw and chemically modified jute fabrics was determined according to the modified procedure described by Soutar and Bryden.[6]

The carboxyl group quantity was determined using the calcium-acetate method.[7] The cellulose carboxyl groups react with calcium acetate (weak acid salt), forming a salt of the cellulose and releasing an equivalent amount of the weaker acid. The cellulose should be obtained in the acidic form by replacement of its cations with hydrogen ions by the treatment of jute sample (0.5 g) with 100 mL of 0.01 M HCl solution for 1 h following by washing with distilled water. In the next step, 50 mL of distilled water and 30 mL of 0.25 M calcium acetate were added and the mixture was stirred for 2 h. Thereafter, 30 mL portions of the liquid were titrated with 0.01 M NaOH solution, using phenolphthalein as an indicator. The quantity of carboxyl groups (Q_{COOH} / mmol g^{-1}) was calculated as follows:

$$Q_{COOH} = \frac{(80/30)0.01V_{NaOH}}{m} \qquad (1)$$

where: 0.01 is the NaOH concentration, mol L^{-1}, V_{NaOH} is the volume of NaOH solution used for titration mL, *m* is the weight of absolutely dry jute fabric, g.

Biosorption of nickel ions

Jute fabric (0.5 g) was immersed in 200 mL of $NiSO_4$ solution (distilled water was used as the aqueous matrix) (initial metal ion concentration: 10–30 mg L^{-1}, initial pH: 5.50 adjusted with 0.1 HCl) at room temperature and constantly shaken for 9 h. After defined times (5–540 min), the concentration of nickel ions in the aqueous solution was determined by inductively coupled plasma optical emission spectrometry, ICP-OES (iCAP 6500 Duo ICP, Thermo Fisher Scientific, Cambridge, UK). The measurements were performed at the Ni II 231.604 nm emission-line. Quality control was carried out using blank samples, matrix-matched calibration solutions, and triplicate analyses of each sample.

The biosorption of nickel ions (*q* / mg g^{-1}) was determined according to:

$$q = \frac{c_0 - c_t}{m}V \qquad (2)$$

where: c_0 and c_t in mg L^{-1} are the nickel ion concentrations in the solution before biosorption and after a defined period of biosorption, respectively, V / L is solution volume and m / g is the mass of dry jute fabric.

Adsorption kinetics

The pseudo-first[8] (Eq. (3)) and pseudo-second-order[9] (Eq. (4)) kinetic models were used to investigate the biosorption mechanism of nickel ions onto jute fabrics:

$$\log (q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \tag{3}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right)t \tag{4}$$

where q_e and q_t in mg g^{-1} are the amounts of nickel ions adsorbed per gram adsorbent at equilibrium and at time t / min, k_1 / min^{-1} is the pseudo-first-order rate constant, while k_2 / g mg^{-1} min^{-1} is the pseudo-second-order rate constant.

Adsorption isotherms

Langmuir[10] (Eq. (5)) and Freundlich[11] (Eq. (6)) isotherm equations were applied for modelling the equilibrium adsorption data, and to assess the maximum biosorption capacities of the jute fabrics:

$$\frac{c_e}{q_e} = \left(\frac{1}{q_m K_L}\right) + \left(\frac{1}{q_m}\right)c_e \tag{5}$$

$$\ln q_e = \ln K_f + \frac{1}{n}\ln c_e \tag{6}$$

where q_e and q_m in mg g^{-1} are the equilibrium and maximal adsorbed nickel per g of adsorbent, K_L / L mg^{-1} is Langmuir constant, c_e / mg L^{-1} is the equilibrium nickel concentration in the solution, K_f is the Freundlich constant / (mg g^{-1}) (L mg^{-1})$^{-1/n}$, $1/n$ is a constant related to the surface heterogeneity of the fabric.

RESULTS AND DISCUSSION

Characterization of the jute biosorbents

Chemical modifications with NaOH and NaClO$_2$ were performed to obtain jute fabrics with different chemical compositions and quantity of carboxyl groups. From the results given in Table I, it is clear that alkali modifications contributed to the selective removal of hemicelluloses (their content decreased to 14 and 37 %), while oxidative modifications led to selective lignin removal (its content decreased to 39 and 63 %). After both types of chemical modifications, cellulose-rich fabrics were obtained (cellulose content increased up to 22 %). Bearing in mind the aim of this investigation, *i.e.*, utilization of waste jute fabrics as biosorbents and the fact that the carboxyl groups represent potential sites for binding metal ions through ion-exchange, their contents within jute fabrics should be taken into consideration, Table I. The increased quantity of carboxyl groups (up to 58 %) in the alkali modified jute fabrics is due to the rupture of lignin–hemicellulose ester linkages, while the sodium chlorite oxidation leads to

conversion of the aldehyde cellulose groups to carboxyl groups (carboxyl group quantity increased up to 81 %), Fig. S-1 of the Supplementary material to this paper.

TABLE I. Chemical composition of jute biosorbents and quantity of COOH groups

Jute fabric	Content ± SD, %			Q_{COOH} / μmol g^{-1}
	Hemicelluloses	Lignin	Cellulose	
RJ	21.76±0.38	13.48±0.56	60.1±1.3	207±1
A1/30	18.62±0.10	13.45±0.14	67.9±1.1	327±7
A17.5/5	13.79±0.30	12.91±0.25	73.3±1.0	284±7
C30	21.29±0.23	8.25±0.39	70.4±1.5	345±9
C60	24.93±0.19	4.96±0.08	70.1±1.4	375±3

Application of the Student's *t*-test confirmed a statistically significant difference between the hemicellulose (in the case of alkali modified jute fabrics) and lignin (in the case of oxidized jute fabrics) contents, as well as the quantity of COOH groups (Table S-I).

Biosorption of nickel ions

Based on the earlier published results,[3,4] *i.e.*, improved sorption properties (such as moisture sorption and water retention power) of chemically modified jute fabrics, it was expected that waste of those fabrics will possess satisfactory biosorption potential towards heavy metal ions. In this section, the influence of contact time, and initial concentration of nickel ions (selected as a model ion) on the biosorption capacity of jute fabrics was analysed in detail. Moreover, the links between the chemical composition of fabrics as well as the quantity of COOH groups and the biosorption capacity of jute fabrics towards nickel ions were established.

The effect of contact time on the biosorption capacity of jute fabric (in further text: *q*) toward nickel ions ($c_0 = 15$ mg L^{-1}) is depicted in Fig. 1. The *q* of raw jute (sample RJ) within the first hour of contact time was about 83 % of its equilibrium biosorption capacity, which is reached after a contact time of 4 h. On the other hand, chemically modified jute fabrics possess higher but slower biosorption of nickel ions. Namely, about 79–81 % of their q_e were observed within a contact time of 2 h, while the equilibriums were attained after a contact time of 7 h. More precisely, at the beginning of the biosorption process, the presence of a large number of free sites for nickel adsorption increased the q_t values (*i.e.*, a quite fast external surface adsorption occurred due to the well-shaken system).[12] However, with prolonged contact time, nickel ions travel within the jute pores, the number of free sites decreased, reducing the nickel adsorption and reaching a plateau. This behaviour could be also ascribed to nickel intra-particle diffusion inside the pores that slowed down the adsorption mainly at the end of the pro-

cess.[13] Additionally, repulsive forces between nickel ions in solution and those adsorbed, could hinder the diffusion of nickel into the adsorbent structure by extending the contact time.[12]
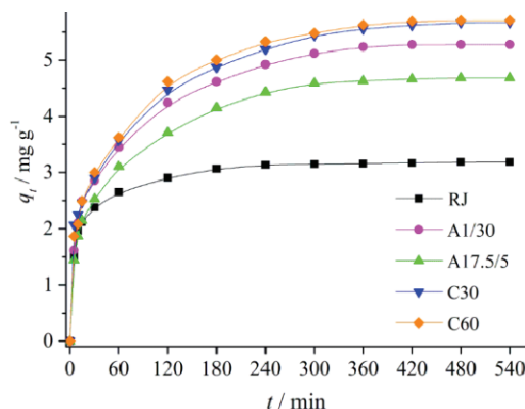

Fig. 1. Biosorption capacity (q_t) of nickel ions ($c_0 = 15$ mg L^{-1}) onto jute fabrics as a function of contact time.

The highest content of non-cellulosic components (Table I) is the possible reason for the lowest q values of the raw jute fabric (sample RJ) since they impeded the diffusion and biosorption of nickel ions, Fig. 1. Moreover, this sample has the lowest amount of active sites, *i.e.*, the lowest quantity of COOH groups (207 µmol g^{-1}), Fig. 2a (q_e is given in µmol g^{-1} to compare with the quantity of COOH groups). A good linear correlation ($r = 0.983$) between the quantity of COOH groups and the equilibrium biosorption capacity of the fabrics (in further text: q_e) was observed, Fig. 2b. Namely, with increasing the quantity of COOH groups in the range 37.2–81.2 %, the q_e of chemically modified jute fabrics was improved by 47.0–78.2 %, compared to the raw jute fabric. In the
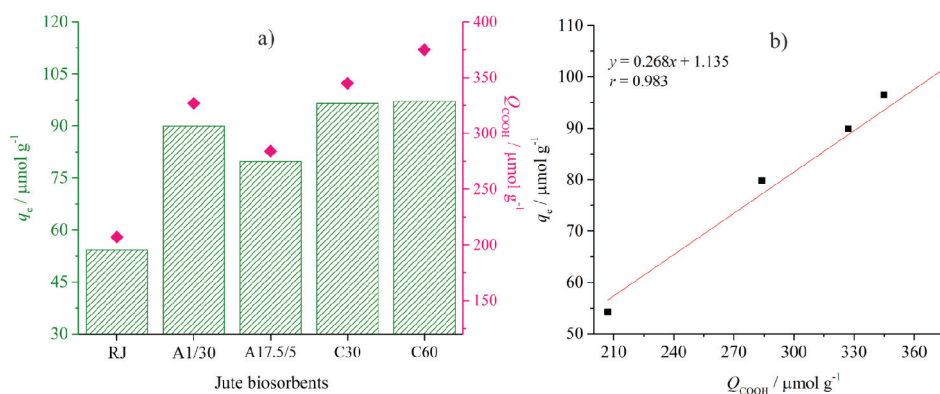

Fig. 2. a) Equilibrium biosorption capacities (q_e) of the fabrics *vs.* quantity of COOH groups (Q_{COOH}) and b) the relationship between the q_e of the fabrics and the quantity of COOH groups.

case of oxidized jute fabrics (samples C30 and C60), besides the quantity of COOH groups, the lignin removal, and consequently the increased content of available hydrophilic components (cellulose and hemicelluloses), also contributed to higher q_e, Fig. 2a.

Kinetic study

To describe the adsorption kinetic of nickel ions on the jute fabrics, the experimental data (Fig. 1) were fitted with pseudo-first and pseudo-second-order kinetic models. Their plots with the coefficients of determination (R^2) are shown in Fig. 3a and b, respectively.
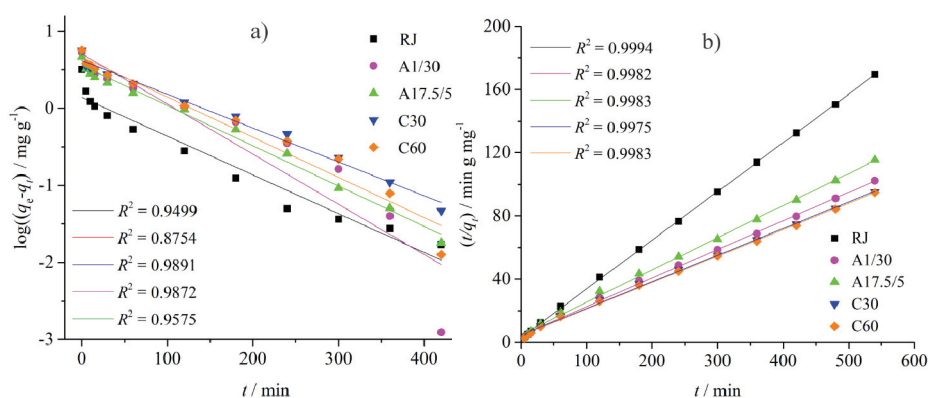

Fig 3. a) Pseudo-first and b) pseudo-second-order kinetics for the adsorption of nickel ions ($c_0 = 15$ mg L^{-1}).

According to the obtained R^2 values (Fig. 3) and model prediction values for the $q_{e,cal}$ (Table II), it is clear that the biosorption of nickel ions on all investigated samples was fitted better by the pseudo-second-order kinetic model, with relative errors of 1.7–4.9 %, indicating that the adsorption/binding was established through sharing or exchange of electrons between the adsorbate and the adsorbent, as ionic exchanges.[14] A significantly higher value of raw jute fabric pseudo-second rate constant (Table II, $k_2 = 0.034$ g mg^{-1} min^{-1}) than the values of chemically modified jute fabrics ($k_2 = 0.005$–0.008 g mg^{-1} min^{-1}) indicates that the rate of

TABLE II. Kinetic parameters obtained by the pseudo-first and pseudo-second-order kinetic models for the adsorption nickel ions ($c_0 = 15$ mg L^{-1}) on jute fabrics

Kinetic model	Kinetic parameters	Jute fabrics				
		RJ	A1/30	A17.5/5	C30	C60
Pseudo-first	$q_{e,cal}$ / mg g^{-1}	1.39	5.11	3.62	4.19	4.65
	k_1 / min^{-1}	0.012	0.015	0.012	0.010	0.012
Pseudo-second	$q_{e,cal}$ / mg g^{-1}	3.24	5.53	4.90	5.93	5.98
	k_2 / g mg^{-1} min^{-1}	0.034	0.008	0.008	0.007	0.005
	$q_{e,exp}$ / mg g^{-1}	3.187	5.278	4.682	5.666	5.700

nickel adsorption is higher in the case of raw fabric. On the other hand, the reported lower values of k_2 for the chemically modified fabrics are the result of a slower nickel adsorption,[15] as can be seen in Fig. 1.

Adsorption isotherms

Initial ion concentration is another factor affecting the biosorption potential of jute fabrics towards nickel ions. The results shown in Fig. 4 reveal that an increase in the initial concentration of nickel ions leads to a remarkable increase in the q_e of jute fabrics. This phenomenon indicated that the driving force for the adsorption of nickel ions is the concentration gradient between the aqueous phase and the solid one, which overcomes the resistance to mass transfer between the two phases; the higher the gradient, the more favoured the process.[16] For example, increasing the initial ion concentration from 10 to 30 mg L^{-1} causes an increase in raw jute fabric q_e by about 92 %. In the case of the chemically modified fabrics, the most prominent increase was observed for the oxidized ones (C30 and C60) and accounts for 160 %, while the lowest increase in q_e was observed for fabric A17.5/5. Independently of initial nickel concentration, the same trend for q_e (RJ < A17.5/5 < A1/30 < C30 < C60) was noticed.
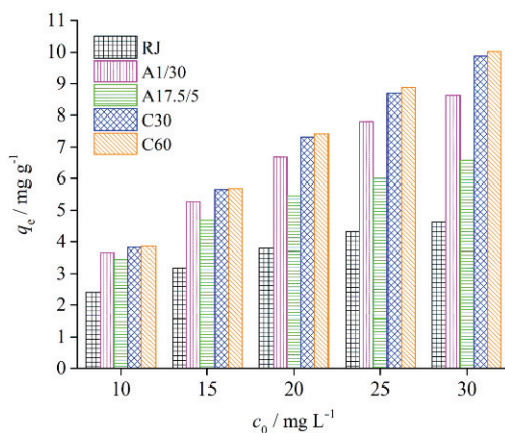

Fig. 4. Equilibrium biosorption (q_e) as a function of initial concentration nickel ions.

In order to determine the maximum biosorption capacities of the jute fabrics and to describe the adsorption behaviour of nickel ions at equilibrium, the Langmuir and Freundlich adsorption isotherms were used. The linear fit of the experimental data along with the coefficients of determination (R^2) are presented in Fig. 5. A comparison of the R^2 values reveals that the biosorption equilibrium data obtained were better fitted by the Langmuir ($R^2 > 0.9918$) than by the Freundlich ($R^2 < 0.9892$) model, implying that the adsorption process is monomolecular and occurs at homogenous active sites in the adsorbents.[17] The isotherm parameters determined by fitting the experimental data (Fig. 4) with the Langmuir and Freundlich adsorption equations are listed in Table III. The maxi-

mal biosorption capacity of the fabrics, *i.e.*, q_m values obtained from Langmuir isotherm ranged from 6.30 mg g^{-1} to 12.06 mg g^{-1}.
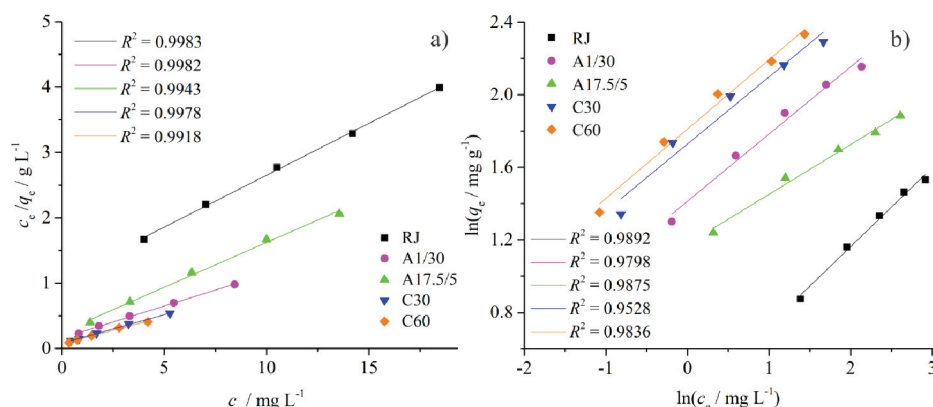

Fig. 5. a) Langmuir and b) Freundlich adsorption isotherm and the linear fit of the experimental adsorption data for nickel ions.

TABLE III. Langmuir and Freundlich isotherm parameters for the adsorption of nickel ions onto jute fabrics

Adsorption isotherm	Isotherm parameters	Jute fabrics				
		RJ	A1/30	A17.5/5	C30	C60
Langmuir	K_L / L mg^{-1}	0.149	0.615	0.548	1.099	1.000
	q_m / mg g^{-1}	6.30	10.21	7.29	11.45	12.06
Freundlich	K_f / (mg g^{-1})(L mg^{-1})$^{-1/n}$	1.34	4.11	3.24	5.63	6.11
	$1/n$[a]	0.44	0.37	0.27	0.97	0.38

[a]Constant related to the surface heterogeneity of the fabric

Observing in parallel the results obtained for the q_m of the samples and quantity of COOH groups (Fig. 6), a good linear correlation ($r = 0.953$) between them was found. It has to be emphasized that the ratios between the q_m and
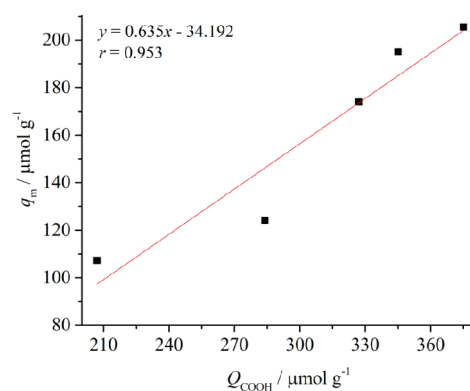

Fig. 6. Relationship between the maximal biosorption capacity of the fabrics (q_m) and the quantity of COOH groups (Q_{COOH}).

COOH group quantity are between 0.44 and 0.55, which means that approximately half of the COOH groups of the fabrics would be involved in binding nickel ions. According to the above mentioned, it can be concluded that the quantity of COOH groups can be used to predict the maximal biosorption potential of the jute fabrics towards nickel ions.

Comparison between other lignocellulosic fibres

Bearing in mind that the biosorption experimental conditions (*i.e.*, biosorbent dosage, solution pH, contact time, initial ion concentration and additional biosorbent treatments) used in the current study differ from those used in other studies (Table IV), it is very difficult to compare the results obtained for the biosorption capacities of jute fabrics with the results reported for other lignocellulosic fibres.

TABLE IV. Comparison between the biosorption potential of other lignocellulosic fibres and findings of the current study

Fibre type	Biosorbent dosage, g L^{-1}	pH	Contact time h	c_0 mg g^{-1}	q_{ex} mg g^{-1}	Reference
Raw hemp felt	0.010	5.00	1	25	≈ 2.4	18
Raw hemp	0.001	5.00	24	100	65	19
Hemp modified with NaOH and citric acid					69	
Raw kenaf	0.005	4.50	24	500	41.66	20
Raw coir	0.005	6.5	2	150	2.74	21
Alkali treated choir					8.84	
Raw coir	0.020	6.5	2	350	2.51	22
Oxidized coir					4.33	
Raw coir pith	0.002	5.3	2	50	15.72	23
	0.007	4.0	1	20	2.066	24
Raw coir	0.011	N/A	1/3	5.067	0.456	25
	0.002	5.95	1/3	20	9.44	26
Raw jute	0.002	5.50	2	500	~ 11	27
Alkali modified jute					~ 12.5	
Raw Jute	0.020	5.87	2	400	3.37	28
Oxidized jute				457	5.57	
Jute dyed with CI Reactive Orange 13				357	5.26	
Raw jute	0.002	5.50	7	25	3.19	Current study
Alkali modified jute					5.28	
Oxidized jute					5.70	

In the study conducted by Shukla and Pai,[28] biosorption capacities of raw and oxidized jute fibres of 3.37 and 5.57 mg g^{-1} were reported, which is very similar to those obtained in the current study (3.19 and 5.70 mg g^{-1}, respectively). However, the researchers used significantly higher initial ion concentrations, a twice higher biosorbent dosage, and a ¼ lower solution volume. Twenty times higher initial nickel concentration and 20 % higher solution volume/biosorbent dosage ratio contributed to higher biosorption capacities of raw and alkali

treated jute fabrics[27] than that presented in the current study. In most of the presented papers (Table IV, initial nickel concentration above 100 mg L^{-1}), the adsorption is not necessary since higher nickel concentrations can be removed by using conventional methods for wastewater treatment. On the other hand, chemically modified jute fabrics used in this investigation are efficient biosorbents for nickel ions present at trace levels in wastewaters.

CONCLUSIONS

Different alkali and oxidative modifications were applied to raw jute fabric in order to obtain jute fabrics with different chemical compositions and quantity of carboxyl groups. Such prepared fabrics were used as biosorbents for nickel ions from an aqueous solution. According to the obtained results, the biosorption of nickel ions obeys the pseudo-second-order kinetic model, while the experimental isotherm data fit better with the Langmuir model. The calculated ratios between maximal biosorption capacities of the jute fabrics (6.30–12.06 mg g^{-1}) and quantity of carboxyl groups indicated that approximately half of the fabrics' carboxyl groups would be involved in binding nickel ions during biosorption. Therefore, the carboxyl group quantity can be used to predict the maximal biosorption capacity of jute fabrics toward nickel ions. Permanent collection and reuse of jute fabric waste have promising multi positive effects on the economy as well environment, including reducing its quantity, saving energy, and its utilization as raw material for profitable innovation, which is in agreement with the circular economy concept.

SUPPLEMENTARY MATERIAL

The jute fabric chemical composition and statistical analysis are available electronically at the pages of the journal website: https://www.shd-pub.org.rs/index.php/JSCS, or from the corresponding author on request.

И З В О Д
БИОСОРПЦИЈА ЈОНА НИКЛА НА ТКАНИНАМА ОД ЈУТЕ: КИНЕТИЧКА И ИЗОТЕРМСКА ИСПИТИВАЊА

АЛЕКСАНДРА ИВАНОВСКА[1], ЛЕПОСАВА ПАВУН[2], БИЉАНА ДОЈЧИНОВИЋ[3] и МИРЈАНА М. КОСТИЋ[4]

[1]Универзитет у Београду, Иновациони центар Технолошко–металуршког факултета, Карнегијева 4, 11000 Београд, [2]Универзитет у Београду, Фармацеутски факултет, Војводе Степе 450, 11000 Београд, [3]Универзитет у Београду, Институт за хемију, технологију и металургију, Његошева 12, 11000 Београд и [4]Универзитет у Београду, Технолошко–металурски факултет, Карнегијева 4, 11000 Београд

Сирова тканина од јуте је хемијски модификована 1 % NaOH у току 30 min, 17,5 % NaOH у току 5 min или 0,7 % NaClO₂ у току 30 и 60 min у циљу испитивања утицаја промена хемијског састава и садржаја карбоксилних група на биосорпциони капацитет тка-

нина према јонима никла. Такође, испитиван је и утицај времена контакта тканина са
раствором NiSO₄ и почетне концентрације раствора никла на биосорпциони капацитет
тканина од јуте. Добијени резултати указују на то да се процес биосорпције никла може
описати кинетичким моделом псеудо-другог реда, а равнотежне вредности добијене за
сорпцију никла се боље слажу са Langmuir адсорпционом изотермом. Израчунати однос
између максималних биосорпционих капацитета тканина од јуте (6,30–12,06 mg g^{-1}) и
садржаја карбоксилних група указују на то да ће приближно половина карбоксилних
група учествовати у везивању јона никла током биосорпције. Према томе, садржај кар-
боксилних група се може користити као индикатор максималног биосорпционог капа-
цитета тканина од јуте према јонима никла. Код оксидисане тканине, уклањање лигнина
које је праћено са повећањем садржаја целулозе и хемицелулоза је такође допринело
већем биосорпционом капацитету.

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