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REMOVAL OF CADMIUM (II) FROM AQUEOUS SOLUTION USING FERMENTED GRAPE MARC AS A NEW ADSORBENT

Article Highlights

- The sorption process is described by the Langmuir isotherm
- The grape marc represents an alternative to expensive sorbents
- FTIR and EDS analysis show that the sorption of Cd(II) is a complex process

Abstract

Biosorption of cadmium from aqueous solutions using fermented grape marc, as well as influence of the most important factors, such as: contact time, granulation of biosorbent and the initial concentration of metal ions, is investigated in this paper. The equilibrium sorption of cadmium ions is achieved after 15-20 min, depending on the initial concentration of metal ions. Such a short time needed to achieve the equilibrium indicates that mass sorption is the dominant process. Langmuir, Freundlich and Dubinin-Radushkevich isotherms were used to describe the equilibrium sorption process, and the Langmuir model was found to be the most convenient. Maximum of sorption capacity is 20 mg g⁻¹. EDS spectrum analysis showed that the process of ion exchange is one of the main sorption mechanisms. Minor changes observed in the FTIR spectrum of grape marc after the sorption of Cd(II) ions indicate the formation a bond between metal ions and partially ionized carboxyl and phenol groups from the biomass. The results of this study confirmed that fermented grape marc, due to its porous structure and characteristic chemical composition, is a good sorption material.

Keywords: biosorption, grape marc, isotherm, cadmium ions.

The presence of heavy metal ions in environment is a growing issue. Therefore, the treatment of wastewaters and the removal of heavy metals are very important from the aspect of protection of human health and of environmental protection. Among heavy metals, cadmium is one of the most toxic, carcinogenic and bioaccumulative [1].

Different methods have been used for removing heavy metals from wastewater: ion exchange, chemical precipitation, ultrafiltration, reverse osmosis, electrochemical processes, etc. [2,3]. The main disadvantages of these methods are low selectivity, high costs and other technical constraints, and it is, therefore, necessary to find an economical alternative.

Biosorption of heavy metal ions is a new method that proved to be very effective for their removal from aqueous solutions [4]. The main advantages of this method are: high selectivity, low cost (based on availability and price of natural biosorbents) and high efficiency at low concentrations of heavy metals [5].

Many biological materials are used as biosorbents for the removal of heavy metal ions: pomegranate peel [6], the bark of walnut, hazelnut and almond [7], the remains of sugarcane [8], olive leaf [9], lemon zest, bananas and oranges [10], etc.

Grape marc is a biological cellulosic material with a multilayer structure whose layers are connected systems of channels and pores that allow relatively high specific surface area necessary for sorption. Metal ions can easily penetrate through the pores and channels where they adsorb on the surface of a number of internal active centers. The chemical composition of grape marc, which prevails phenolic compounds, is of great importance for the sorption of

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heavy metals. The goal of this research was to investigate specific biosorbent properties of grape marc remains from wine production of the “Plantaže 13. Jul” company in Podgorica.

EXPERIMENTAL

Sorbent

The biosorbent was used in its original form or in the form of its various modifications in order to achieve better sorption effectiveness. The original form was modified by physical, chemical and thermal treatments. The grape marc remains from the wine production was subjected to leak through the press under a pressure of 2×10^5 Pa. The resulting grape marc was washed with distilled water, then rinsed with dilute hydrochloric acid, and again washed with distilled water to remove Cl^- . Biosorbent was then dried at ambient temperature and at 60°C to constant weight. The granulation was performed on a steel laboratory blender (ITNMS, Laboratory for preparation of raw materials, Belgrade) and the obtained biosorbent was sifted through a standard steel sieve to obtain fractions of appropriate particle size: <0.5 mm, 0.5 - 1 mm, 1 - 2 mm, >2 mm. Before doing any examination, biomass was again treated with dilute HCl, rinsed with distilled water to remove Cl^- ions and dried at 60°C to constant mass.

Materials

All chemicals used in this experiment were p.a. grade. The initial concentration of cadmium ions was 1000 mg dm^{-3} obtained by dissolving the appropriate amount of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in distilled water. The tests were performed in the concentration range of cadmium ions from 20 - 400 mg dm^{-3} , and solutions of specified concentrations were obtained by successive dilution of the initial solution.

Analysis

Method of flame atomic absorption spectrophotometry was performed using AA-6800 instrument (Shimadzu, Japan) at a wavelength of 228.8 nm . The pH value was measured using a pH-meter (WTW inoLab pH 720). Fourier transform infrared spectroscopy (FTIR) was used to identify different chemical functional groups present in the grape marc in the wavelength range 4000 - 400 cm^{-1} .

Sorption experiments

The sorption experiments were performed using a mixture of 0.1 g of grape marc and 100 cm^3 of solution that contains various concentrations of cadmium ions. In order to find the optimal conditions for

sorption, pH values (3 - 6), contact time (3 - 120 min), the concentration of cadmium ions (20 - 400 mg dm^{-3}) and sorbent particle size were varied. The mixture was shaken in a Heidolph reciprocal shaker 130 rpm . The filtration and stabilization of filtrate using 1 cm^3 of concentrated HNO_3 were done before spectrophotometric analysis.

The amount of metal ion adsorbed per unit mass of the biosorbent was calculated as [11]:

$$q = \frac{C_0 - C_e}{V} S \quad (1)$$

where C_0 is the initial metal ion concentration (mg dm^{-3}), C_e is the equilibrium metal ion concentration (mg dm^{-3}), V is the volume of metal ion solution (dm^3) and S is the mass of biosorbent (g).

Percent removal ($\% R$) of metal ions was calculated from the following equation [12,13]:

$$\% R = 100 \frac{C_0 - C_e}{C_0} \quad (2)$$

RESULTS AND DISCUSSION

The influence of initial pH

Increase in the percentage removal of cadmium ions with increasing pH (Table 1) indicates that the investigated sorption process largely performed through the ionic changes [13,14]. The efficiency of removal of cadmium ions increase with decreasing grain size of grape husk for constant pH value. At pH 3, 44% of total Cd in solution was removed by fine grain sorbent while only 40% was removed by larger grain of biosorbent. Percentages of removed Cd ions at pH 5 for mentioned grain size are 97.0 and 86.6%, respectively. At $\text{pH} > 5$, percentage of removal Cd ions is slightly decreased, because $\text{Cd}(\text{II})$ ions are transformed to $\text{Cd}(\text{OH})^+$ or $\text{Cd}(\text{OH})_2$. For these reasons, the concentration of Cd (II) ions in the solution and thus amount of adsorbed metal are reduced.

Table 1. Percentage removal of cadmium ions at different initial pH value

Initial pH value	Grain size	
	Fine	Large
3	44	40
4	85	75
5	97	86.6
6	96	85.6

The best results obtained at pH 5 can be explained by decreasing quantity of adsorbed H^+ ions which reject $\text{Cd}(\text{II})$ ions with increasing pH solution value. The influence of pH on the adsorption of metal

ions is significantly related to the presence of different functional groups (phenol, aldehyde, carboxylic) on the biosorbent surface [15]. These functional groups have a great affinity for metal ions, which are bounded to the surface in complex ion [16,18-20].

The influence of biosorbent average grain size on removal Cd (II) ions

The biosorbent was fragmented using a steel mill, and after screening four fractions (0.5 mm, 0.5-1 mm, 1-2 mm, >2 mm) were obtained. It is evident that the absorption of Cd(II) ions is enhanced with decreasing biosorbent average grain size (Table 2) due to contact surface enlargement [21,22]. The best results were obtained for granulation of 0.5 mm. However, the fraction with the largest grain size (unground grape marc) achieved the removal percentage of 86.6%, which is significant in terms of future applications from the perspective of technology simplification and cost reduction.

Table 2. Percentage removal of cadmium ions for different biosorbent average grain size

Biosorbent average grain size, mm	0.5	1.0	2.0	2.5
Percentage removal of cadmium ions, %	97.0	96.6	96.0	86.6

Influence of initial concentration on the equilibrium of sorption processes

Investigation of adsorption at different initial sorbate concentrations (20-400 mg dm⁻³) enabled construction of sorption isotherms, based on which the maximum sorption capacity of grape marc was determined. Dependence of the sorption quantity on equilibrium concentration of metal ions in solution is shown in Figure 1.

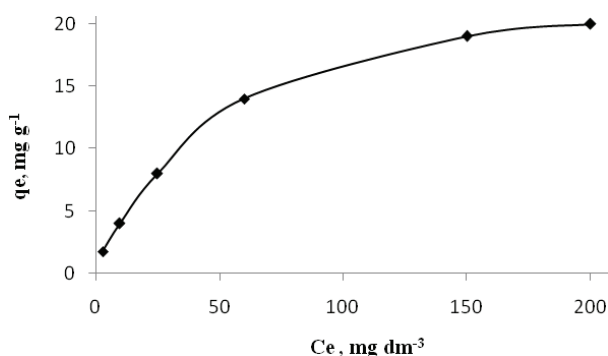


Figure 1. Adsorption isotherm of Cd(II) on grape marc at 25.0±0.5 °C and pH 5.0.

The obtained isotherm belongs to L-type isotherms, and is characterized by a much higher slope

in the range of lower metal concentrations, while at high concentrations the dependence slope decreases and tends to plateau. The maximum quantity of sorbed metal was about 20 mg g⁻¹. Three different isotherms were used in order to determine which one best matched the experimental results.

The Langmuir isotherm assumes monolayer adsorption on a uniform surface with a conclusive number of adsorption sites [22,23]. The linear form of the Langmuir isotherm model is described as:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (3)$$

where K_L is Langmuir equilibrium constant, related to the energy of sorption (dm³ mg⁻¹) and q_m (mg g⁻¹) is the maximum sorption capacity of the sorbent in a complete monolayer of sorbate (mg g⁻¹). The values of Langmuir parameters q_{max} and K_L were calculated from the slope and intercept of the linear plot of C_e/q_e vs. C_e as shown in Figure 2a. The values of q_{max} , K_L and regression coefficient R^2 are presented in Table 3.

Freundlich isotherm model applies to adsorption on heterogeneous surfaces with the interaction between adsorbed molecules. The linear form of the Freundlich isotherm model is described as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (4)$$

where K_F is the Freundlich constant related to the bonding energy and $1/n$ is the heterogeneity factor and n (g dm⁻³) is a measure of the deviation from linearity of adsorption.

Freundlich equilibrium constants were determined from the slope and intercept of the linear plot of $\ln q_e$ vs $\ln C_e$ as shown in Figure 2b. The values of the constants of the Freundlich isotherm are given in Table 3.

The obtained values of R^2 show that the Langmuir isotherm (with $R^2 > 0.99$) has the best agreement with experimental results. Additionally, the experimentally determined values of the sorption capacity of grape marc were close to its theoretical maximum. Such behavior was found for the sorption of heavy metals on the similar biosorbent [22,23]. The most important role in the sorption is assigned to oxygen functional groups, primarily the carbonyl and phenol, from the grape marc. In fact, oxygen electrons pair enables the sorption of positively charged Cd(II) ions. The sorption capacity values of grape marc are close to sorption capacity of other biosorbents (Table 4).

The essential characteristics of the Langmuir isotherm parameters can be used to predict the affinity between the sorbate and sorbent using separ-

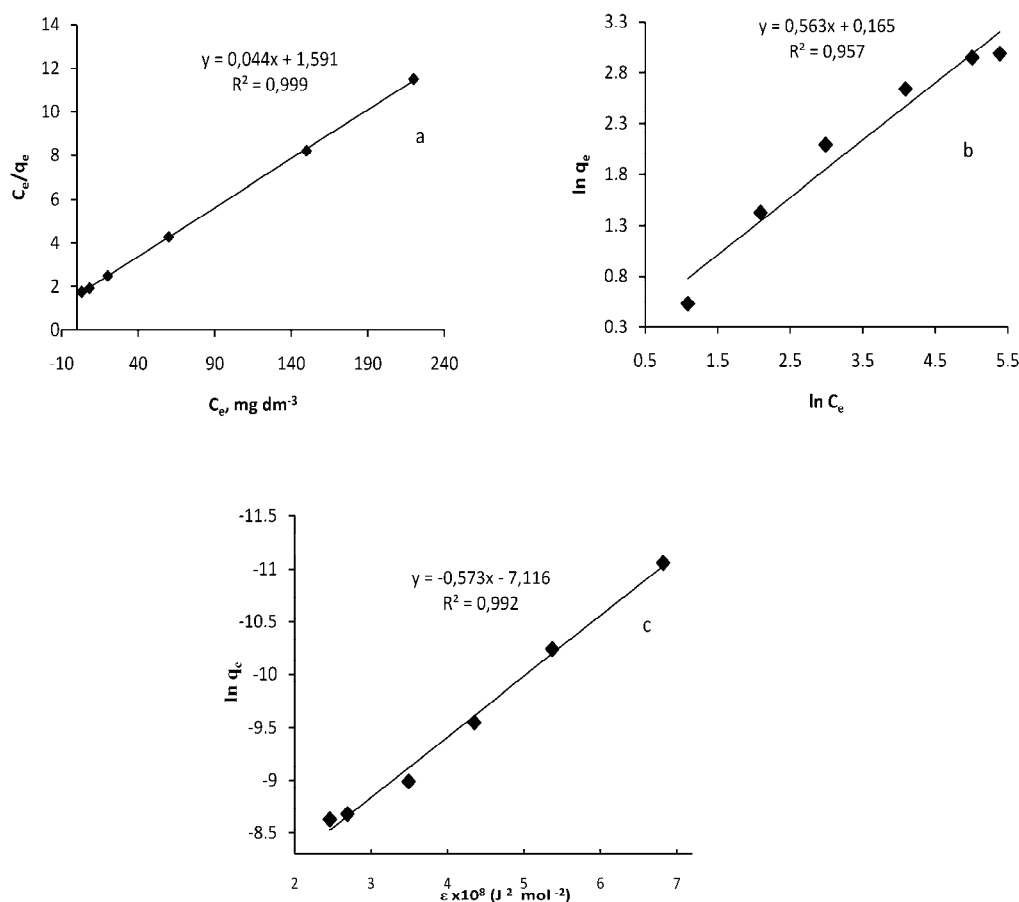


Figure 2. Isotherms for sorption of Cd(II) on grape marc: a) Langmuir isotherm; b) Freundlich isotherm; c) D-R isotherm at 25.0±0.5 °C and pH 5.0.

Table 3. Langmuir and Freundlich isotherm constants for bio-sorption of Cd(II) ions on grape marc

Langmuir constant			Freundlich constant		
$q_m / \text{mg g}^{-1}$	$K_L / \text{dm}^3 \text{mg}^{-1}$	R^2	K_F	n	R^2
22.7	0.028	0.999	1.179	1.776	0.957

Table 4. The sorption capacities of different sorbents for Cd (II)

Biosorbent	$q_m / \text{mg g}^{-1}$	Reference
Waste cork	2.4	25
Loquat leaves	48.78	26
Red alga	53.1	13
Ceratonia siliqua bark	14.27	27
Pomelo peel	21.83	28
Untreated coffee grou	15.65	29
Rice hus	8.58	30
Maize leaf	10.18	31
Lagenaria vulgaris	11.25	32
Grape marc	20	This work

ation factor or dimensionless equilibrium parameter, R_L expressed as in the following equation [32]:

$$R_L = \frac{1}{1 + K_L C_0} \tag{5}$$

The value of R_L indicated the type of Langmuir isotherm can be irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$) or unfavorable ($R_L > 1$).

The R_L was found to be 0.082-0.641 for concentration of 20-400 mg dm^{-3} of Cd(II) ions. They are in the range of 0-1, which indicates favorable bio-sorption. In support of this conclusion is the fact that the experimentally obtained isotherm is convex, which is characteristic of favored sorption.

The Dubinin-Radushkevich (D-R) model, which does not assume a homogenous surface or a constant bio-sorption potential as the Langmuir model, was also used to test the experimental data in order to determine the nature of the sorption process [33]. The Dubinin-Radushkevich isotherm is given by the equation:

$$\ln q_e = \ln q_m - \beta e^2 \tag{6}$$

where β is the coefficient associated with the free energy biosorption ($\text{mol}^2 \text{J}^{-2}$) and ε is Polanyi's potential, given by the equation:

$$\varepsilon = RT \left(1 + \frac{1}{C_0} \right) \quad (7)$$

The D-R isotherm parameters β and q_m were obtained from the slope and intercept of the plot of $\ln q_e$ vs. ε^2 (Figure 2c). The free energy of biosorption, E , was calculated from the β value using the following equation:

$$E = \frac{1}{\sqrt{2\beta}} \quad (8)$$

For Cd (II) ions biosorption on grape marc, the energy biosorption value was of 10.7 kJ mol^{-1} , indicating a chemisorption process and that one of the mechanisms of biosorption process is ion exchange [34].

The influence of contact time on removal of Cd (II) ions

The influence of contact time for different concentration sorption capacity was investigated. The results are shown in Figure 3.

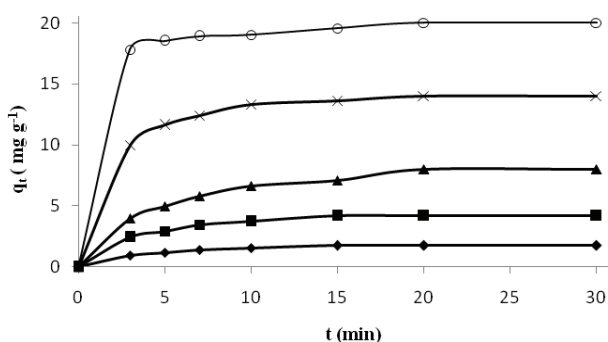


Figure 3. The effect of contact time on Cd(II) sorption on grape marc. The initial solution concentrations of Cd (II) ions (mg g^{-1}) were: 20 (♦), 50 (■): 100 (▲), 200 (X) 400 (○).

According to the results, the investigated sorption process consists of two phases. The first one is characterized by fast sorption and represents a consequence of significant difference in concentration of metals in solution and on the sorbent surface as well as of large number of available active sites. The second phase is slower and occurs until the equilibrium state. The time to achieve the equilibrium was 15 min for lower concentrations of 20 and 50 mg dm^{-3} and 20 min for higher concentrations of 100–400 mg dm^{-3} . The short time needed to achieve the equilibrium indicates that the dominant process is mass sorption.

The percentage of removal of cadmium ions largely depends on the concentration. With increasing the initial concentration, the percentage of removal of Cd(II) ions decreased from 85% at 20 mg dm^{-3} to 50% at a 400 mg dm^{-3} . This behavior is due to limited number of active sites on the surface for sorption process [35].

EDS Analysis

EDS spectrum analysis before the sorption process (Figure 4a) indicated that the carbon and oxygen are two main elements in the composition of biosorbent surface along with the other elements present in trace amounts. Registered peaks of alkali (K) and alkaline earth metals (Ca) are derived from the raw biosorbent which contains these metals. The minor amounts of copper are probably due to fertilization and after purification this metal is almost completely removed from biomass.

EDS spectrum analysis after the sorption process (Figure 4b) shows that the K peaks are almost absent, indicating that these ions were substituted by Cd(II) ions during the process of ion exchange, which is one of the main sorption mechanisms.

FTIR Analysis

The FTIR analysis enables identification of the type of functional groups on the surface of the sample that could participate in the binding of metal ions, as well as their features before and after the sorption process [19,36]. The FTIR spectra were obtained using Omic software and presented in Figures 5 and 6.

The broad intense band at 3324.4 cm^{-1} (which typically occurs in the range of 3200 to 3600 cm^{-1}) belongs to valence vibration of O-H group. The noticeable peak at 2922 cm^{-1} indicates the symmetric or asymmetric C-H valence vibration of aliphatic acids, while the peak registered at 1743.5 cm^{-1} further indicates the valence vibration of -C=O connection that comes from the unionized carboxyl group and may correspond to protonated form or the corresponding ester (-COOH , -COOCH_3) [37].

Also, the band at 1032.6 cm^{-1} can be connected with the existence of valence vibration of -C-O of alcohols or carboxylic acids [38]. FTIR spectrum of grape marc clearly indicates the presence of carbonyl and hydroxyl groups, which are the most important sites for the binding of cadmium ions.

The obtained results show that there are no significant changes in the spectra before and after the sorption process. The slightly decrease in intensity and shift of the peak at 3324.4 cm^{-1} indicates decreasing content of free hydroxyl groups of the sorbent.

Also, slightly decreasing in intensity and shift of the noticeable peaks at 1605.2 and 1032.6 cm^{-1} indicates binding heavy metal and $-\text{C}=\text{O}$ group (Table 5). The carbonyl groups have the highest probability for bind-

ing investigated metal ions. Since the partially ionized carboxyl and phenol groups are the most responsible for the sorption of metals, minor changes observed in the FTIR spectrum of grape marc after the sorption

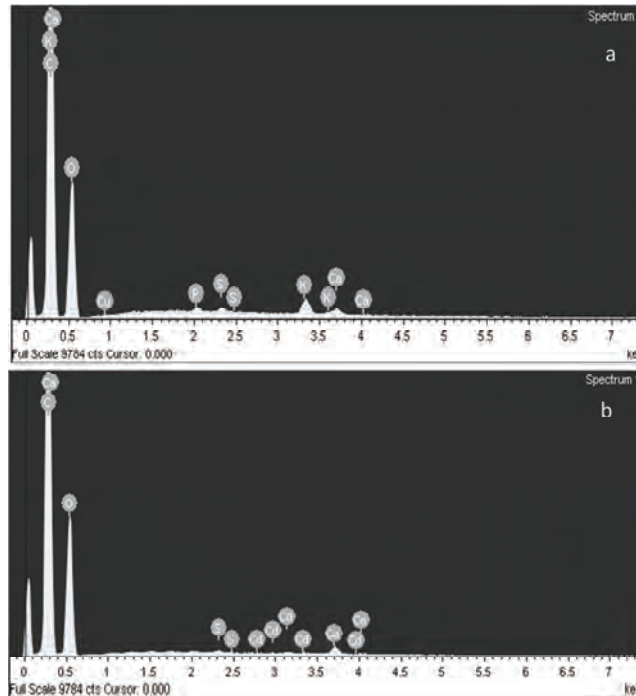


Figure 4. EDS Spectra of grape marc: a) before biosorption; b) after biosorption.

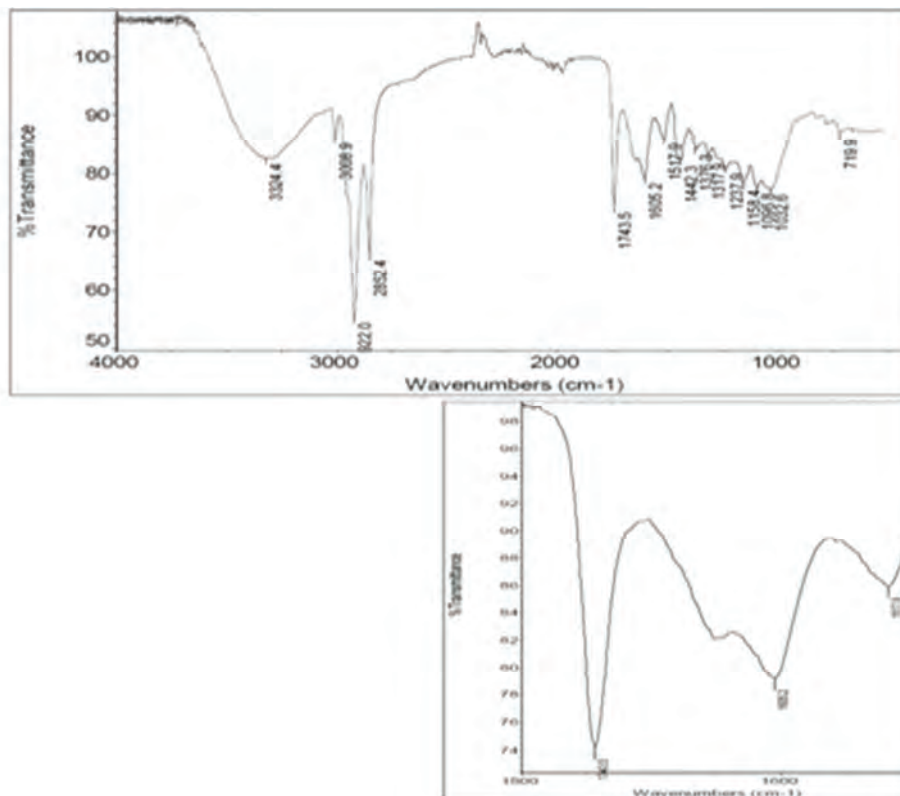


Figure 5. FTIR spectrum of biosorbent before the sorption process.

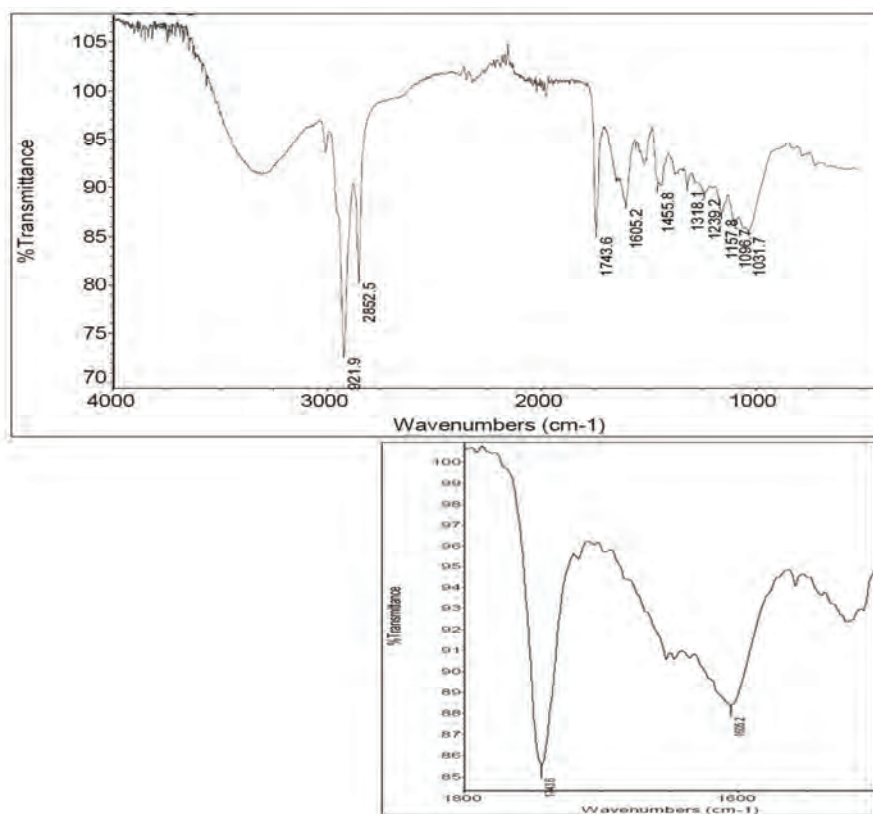


Figure 6. FTIR spectrum of biosorbent after the sorption process.

Table 5. Wavenumbers (cm^{-1}) and functional groups from FTIR spectra before and after sorption

Grape marc		Functional group
Native fermented	Fermented, after sorption	
3324.4	3314.4	valence vibration of O-H group
2922	2922	symmetric and asymmetric C-H valence vibration of aliphatic acids
1743.5	1743.6	-C=O valence vibration of carboxyl acids and corresponding esters
1605.2	1605.0	-C=O valence vibration (acids, esters)
1032.6	1031.7	-C-O valence vibration

process can be explained by the formation a bond between the metal ions and these groups [39]. It is assumed that the binding of metal cations to the aforementioned functional groups leads to the release of protons, because of their high affinity for Cd (II) ions. The release of protons is evident by decreasing pH of the solution during the sorption.

CONCLUSION

Effective removal of Cd (II) ions from aqueous solutions using grape marc largely depends on the experimental conditions such as pH value, initial concentration, contact time and biosorbent grain size. The optimal parameters of the metal ions sorption are: pH 5.0 and biosorbent grain size < 0.5 mm. The equilibrium of the investigated process is best des-

cribed by the Langmuir isotherm. The sorption process is fast and the maximum capacity of 20 mg g^{-1} is reached after 20 min.

Based on the results of FTIR and EDS analysis, it was found that sorption of Cd (II) in the grape marc complex process is followed by electrostatic attraction, ion exchange and formation of complex compounds [18,19].

The results of this study show that the biosorbent based on grape marc can be recommended as a very efficient and cost-effective mean to remove heavy metals from both natural and waste waters. The biosorbents represent an alternative to expensive sorbents and technologies used in the purpose of purification of aqueous solutions from heavy metals.

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NAUČNI RAD

UKLANJANJE KADMIJUM (II) JONA IZ VODENOG RASTVORA POMOĆU FERMENTISANE KOMINE GROŽĐA KAO NOVOG SORBENTA

U ovom radu je ispitivana biosorpcija kadmijuma iz vodenih rastvora korišćenjem fermentisane komine grožđa i uticaj najvažnijih faktora, kao što su: kontaktno vrijeme, granulacija biosorbenta i početna koncentracija metalnih jona. Ravnoteža procesa sorpcije jona kadmijuma se postiže nakon 15-20 min u zavisnosti od početne koncentracije metalnih jona. Tako kratko vrijeme potrebno da se postigne ravnoteža ukazuje na to da je transport mase dominantan proces. Za opisivanje ravnoteže sorpcionog procesa korišćene su Langmuir, Freundlich i Dubinin-Radushkevich izoterme, pri čemu je Langmuir model najpogodniji. Maksimalni sorpcioni kapacitet je 20 mg g^{-1} . Analiza EDS spektra je pokazala da je proces jonske razmjene jedan od glavnih mehanizama sorpcije. Manje promjene uočene u FTIR spektru komine grožđa nakon sorpcije Cd (II) jona ukazuju na formiranje veza između jona metala i djelimično jonizovane karboksilne i fenolne grupe iz biomase. Rezultati istraživanja pokazuju da je fermentisana komina groždja, zahvaljujući svojoj poroznoj strukturi i karakterističnom hemijskom sastavu, dobar sorbent.

Ključne reči: biosorpcija, komina groždja, izoterma, joni kadmijuma.