



# Uranium(VI) biosorption by sugar beet pulp: equilibrium, kinetic and thermodynamic studies

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## Abstract

Native and chemically modified sugar beet pulp were used as a biosorbents for removal of uranium(VI) from the aqueous solution. The effects on the uranium(VI) uptake such as solution pH (2–9), adsorbent amount (50–500 mg), contact time (0–180 min), temperature (293–323 K) and initial concentration (20–60 mg U L<sup>-1</sup>) were studied. The obtained maximum adsorption capacities according to Langmuir isotherm model for native and modified sugar beet pulps were 20.45 and 19.80 mg g<sup>-1</sup>, respectively. Additionally, pseudo-second order kinetic model best fitted the experimental data.

**Keywords** Uranium(VI) · Sugar beet pulp · Biosorption · Kinetic

## Introduction

Due to its radioactivity and chemical toxicity at very low concentrations uranium is extremely dangerous for the environment and human health. Increasing amounts of wastewater containing uranium are produced annually from various activities such as exploration and processing of uranium, mining industry, nuclear industry associated activities, industries that utilize radioisotopes and geological disposal of radioactive waste [1, 2]. In the environment uranium is mainly present in two oxidation states U(IV) and U(VI). Among this two uranium species, U(IV) is less soluble and occurs under strongly reducing conditions and U(VI) appears as a mobile, hydrated uranyl UO<sub>2</sub><sup>2+</sup> ion [3, 4]. Some of conventional methods for removal of uranium from the wastewater are chemical precipitation, co-precipitation, electrochemical treatment, ion exchange, membrane processes, solvent extraction and adsorption [5–12]. All listed methods have a lot of limitations, such as incomplete metal removal, high energy depletion, limiting pH and concentrated liquid wastes [13, 14]. Among listed methods, the most promising method for uranium removal is adsorption due to its simplicity, applicability, high selectivity and capacity. Recently a

lot of research is focused on development of new innovative adsorbents with special emphasis on biosorbents. Based on the use of biomass as an adsorbent, biosorption of different types of metal ions including uranium from aqueous solutions is considered as a very promising procedure. Compared to other methods, biosorption is more economical, eco-friendly, energy saving, has a wide working range of pH and temperature, minimal volume of disposable sludge, high efficiency, selectivity, moreover, once used biomass can be reused in multiple cycles depending on its capacity [15–18].

A lot of different adsorbents, such as pummelo peel [19], *Eucalyptus citriodora* distillation sludge [20], *Citrobacter freundii*, a bacterium [1], *Catenella repens*, a red alga [21], pollen pini [22], *Bangia atropurpurea*, a red alga [23], alginate beads [24], wood powder and wheat straw [4], polyaniline coated magnetic carboxymethylcellulose [25], silica particles grafted with polyacrylonitrile beads [26] etc. were considered for the removal of uranium ions from the aqueous solution, but there are only a few studies in which removal of uranium and other radionuclides were explored by adsorption on agricultural waste [27–31]. Agricultural waste as a low-cost biosorbent is of great interest for treatment and removal of toxic pollutants due to its several advantages such as specific chemical composition (i.e. hemicellulose, cellulose, lipids, lignin, sugar, pectins etc.) containing a lot of acidic functional groups (i.e. carboxylic and hydroxy) which can bind pollutant ions very effectively. Additional advantages are availability, high efficiency as well as easy to modify with different chemical reagents [13, 32]. Moreover,

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a numerous byproduct from industrial operations can be used as a potential inexpensive biosorbents [32]. Sugar beet pulp (*Beta vulgaris*) is a low-cost byproduct of the sugar industry and it is mainly used as animal feed. It is very rich in pectic substances which are complex heteropoly-saccharides containing galacturonic acid, arabinose, galactose and rhamnose as the major sugar constituents. It is also rich in cellulose, which main component is glucose [33–36]. Functional groups found in sugar beet pulp are associated to very strong heavy metal binding, specially carboxyl groups with a great biosorption and heavy metal removal potential [37]. According to its properties, sugar beet pulp has been widely used in many studies for heavy metal removal [34–42].

In this study sugar beet pulp was used as biosorbent in native and modified form for the removal of uranium(VI) from aqueous solution. The influence of adsorption conditions, such as pH, biosorbent dosage, contact time, initial metal ions concentration, and temperature on the biosorption process was investigated.

## Experimental

### Reagents and solutions

All used chemical reagents were of analytical grades. Arsenazo(III),  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and 60%  $\text{HClO}_4$  were obtained from Merck (Darmstadt), 37% hydrochloric acid was purchased by Semikem (Sarajevo), 65% nitric was purchased by Carlo Erba (Milano) and sodium hydroxide by Alkaloid (Skopje). The stock solution of U(VI) (1000 mg/L) was prepared by weighting 2.1308 g (Metler Toledo balance,  $\pm 0.1$  mg) of uranyl nitrate hexahydrate salt (A.R. grade) and dissolving it in 3 mol  $\text{dm}^{-3}$  perchloric acid. Working solution were prepared by diluting the stock solution.

### Sugar beet pulp preparation and characterization

Sugar beet pulp was provided by Sunoko Sugar Factory (Novi Sad, Serbia) as a fraction which is remaining after the production of sugar. The sugar beet pulp was first washed by the tap water, then several times with distilled water, and then air-dried for 24 h. After 24 h it was dried in an oven at 100 °C to a constant weight and then divided into two equal parts. One part of it was blended and powder was sieved through a standard sieve ( $\varnothing$  0.25 mm) then stored in hermetically sealed container and it was marked as a native sugar beet pulp (NSBP).

Second part of the remaining sugar beet pulp was modified with 0.25 mol  $\text{L}^{-1}$  nitric acid and 0.1 mol  $\text{L}^{-1}$  sodium hydroxide in the way it was first immersed in 0.25 mol  $\text{L}^{-1}$   $\text{HNO}_3$  and occasionally stirred for 24 h. After 24 h it was rinsed with distilled water until pH of the filtrate wasn't

settled and then it was air-dried. The sugar beet pulp was then left 4 h in NaOH solution, washed several times with small quantities of distilled water until pH settled, air-dried, blended then sieved through a standard steel sieve ( $\varnothing$  0.25 mm) and stored in hermetically sealed container and marked as modified sugar beet pulp (MSBP).

The morphology and composition of the milled native (NSBP) and modified sugar beet pulp (MSBP) were characterized by SEM/EDS using a JEOL JSM-6610LV Scanning Electron Microscope, at an accelerating voltage of 20 keV. In order to determine functional groups of NSBP and MSBP, infrared spectra of the Fourier transform (FT-IR) were recorded on a Perkin Elmer BX FT-IR spectrometer using KBr pellet technique in a region from 4000 to 400  $\text{cm}^{-1}$ .

### Biosorption experiments

Batch techniques were conducted in closed polypropylene bottle at 95–100 movements per minute (mechanical shaker: Heidolph Duomax 2030) to study the biosorption of U(VI) onto NSBP and MSBP. Experiments were performed by adding (50–500 mg) of NBP and MSBP biosorbents into a 50 mL solution which was having a particular concentration of uranium(VI) (20–60  $\text{mg L}^{-1}$ ).

All experiments were performed in triplicate at 25 °C and the mean values have been used for data evaluation because the variation of the experimental data was within the measurement error ( $\pm 5\%$ ). The pH of each sample solution was adjusted with 3.0 mol  $\text{L}^{-1}$  NaOH and measured by digital pH meter (CG 841 Schott-GERÄTE GmbH). The concentration of uranium(VI) in solution was determined spectrophotometrically by Arsenazo-III method on a UV-Vis spectrophotometer (model: Varian Hary 50) [31, 43]. The maximum absorbance of the formed complex was on 650 nm, and the maximum absorbance of the Arsenazo(III) reagent was at 535 nm [31].

The amount of adsorbed uranium(VI) ions as adsorption capacity,  $q$  ( $\text{mg g}^{-1}$ ) onto NSBP and MSBP was calculated according to the equation:

$$q = (C_i - C_f)V/m \quad (1)$$

where  $C_i$  and  $C_f$  are initial and final concentrations of uranium(VI) ions ( $\text{mg L}^{-1}$ ),  $V$  is the volume of the solution and  $m$  is the mass of adsorbent (g).

Removal efficiency (%) as the amount of uranium(VI) hold on the biosorbents was calculated through the following equation:

$$\text{Removal efficiency (\%)} = (C_i - C_f)100/C_i \quad (2)$$

where Removal efficiency (%) is the amount of metal ion retained on the biosorbent.

Under the same experimental conditions but without biosorbent a control sample was subjected to all steps of the

experiment to find out was there a loss of analyte during the process, and the concentration of control samples was taken as initial concentration of uranium U(VI) ions ( $C_i$ ).

### Biosorption kinetics

For biosorption kinetic modeling of U(VI) onto native sugar beet pulp (NSBP) and modified (MSBP) were used Lagergren pseudo-first-order model [44] (Eq. 3) and applied as follows:

$$\ln(q_e - q_t) = \ln q_e - k_1 \cdot t \quad (3)$$

where  $q_e$  and  $q_t$  are the amounts adsorbed at equilibrium and at time,  $t$  ( $\text{mg g}^{-1}$ ), respectively and  $k_1$  is the rate constant of the pseudo-first order adsorption ( $\text{min}^{-1}$ ) which can be obtained from the slope of the linear plot of  $\ln(q_e - q_t)$  versus  $t$ .

The second used kinetic model was Lagergren pseudo-second-order model (Eq. 4) which assumes the limiting step is chemisorption [45–47] and for this study this model was applied in the form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \cdot t \quad (4)$$

where  $k_2$  ( $\text{g mg}^{-1} \text{min}^{-1}$ ) is the rate constant of pseudo-second order kinetic model. The plot of  $t/q_t$  versus  $t$  give a linear relation with a slope  $1/q_e$  and an intercept of  $1/(k_2 q_e^2)$  [48].

Additionally, intraparticle diffusion (Weber–Morris) model (Eq. 5) was used [49] in the form:

$$q_t = k_{in} \cdot t^{1/2} + C \quad (5)$$

where  $k_{in}$  is the intraparticle diffusion rate constant ( $\text{mg g}^{-1} \text{min}^{-1/2}$ ) and  $C$  is the constant of the model in the function of the boundary layer thickness ( $\text{mg g}^{-1}$ ).

The applicability of these kinetic models were based on the coefficient of correlation ( $r^2$ ), because it is a measure of experimental data, compatible with the suggested model.

### Equilibrium biosorption isotherms

The experimental data were analysed using Langmuir [50], Freundlich [51] and Temkin models [52].

The Langmuir model was applied in the linearized form:

$$\frac{1}{q_e} = \left( \frac{1}{K_L q_{\max}} \right) \frac{1}{c_e} + \frac{1}{q_{\max}} \quad (6)$$

where  $q_e$  ( $\text{mg g}^{-1}$ ) is the equilibrated amount of analyte adsorbed per unit weight of biosorbent,  $c_e$  ( $\text{mg dm}^{-3}$ ) is equilibrium concentration after biosorption,  $q_{\max}$  ( $\text{mg g}^{-1}$ ) is maximum biosorption capacity and  $K_L$  ( $\text{dm}^3 \text{g}^{-1}$ ) is the Langmuir isotherm constant that is associated with

biosorption free energy. A plot of  $1/q_e$  versus  $1/c_e$  gives the adsorption coefficients.

Affinity between the sorbate and sorbent using Langmuir isotherm is described by non-dimensional separating factor,  $R_L$  given by the equation:

$$R_L = \frac{1}{1 + K_L c_0} \quad (7)$$

The value of  $R_L$  indicates the nature of the biosorption mechanism [27]. The  $R_L$  values are in the range of  $0 < R_L < 1$ , and if the value is between 0 and 1, the biosorption process is favorable [50].

The Freundlich model was also applied in the linear form as:

$$\log q_e = \log K_F + \frac{1}{n} \log c_e \quad (8)$$

where  $K_F$  ( $\text{mg g}^{-1} (\text{dm}^3 \text{mg}^{-1})^{1/n}$ ) and  $n$  ( $\text{g dm}^{-3}$ ) are Freundlich's constants of adsorption isotherms constants indicating adsorption capacity and intensity, respectively. The values of Freundlich's constants ( $K_F$  and exponent  $n$ ) are obtained from a plot of  $\log q_e$  versus  $\log c_e$  [51].

The linear form of the applied Temkin's isotherm equation is given as:

$$q_e = \left( \frac{RT}{b} \right) \ln(A_T) + \left( \frac{RT}{b} \right) \ln(c_e) \quad (9)$$

where  $c_e$  is the concentration of adsorbate ( $\text{mg L}^{-1}$ ),  $q_e$  is the amount of adsorbate adsorbed per unit mass of biosorbent ( $\text{mg kg}^{-1}$ ),  $A_T$  ( $\text{L g}^{-1}$ ) is the equilibrium binding constant relating to the maximum binding energy and determines the Temkin adsorption potential,  $b$  ( $\text{J mol}^{-1}$ ) is indicator to the heat of biosorption, and  $T$  and  $R$  are the temperature (K) and gas constant ( $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$ ), A plot of  $q_e$  versus  $\ln c_e$  gives Temkin's constants  $A_T$  and  $b$  [52, 53].

### Thermodynamics of uranium biosorption

Thermodynamic parameters, such as enthalpy change ( $\Delta H$ ), entropy change ( $\Delta S$ ) and change in Gibbs free energy ( $\Delta G$ ), were obtained from the following equations:

$$\Delta G = -RT \ln K_c \quad (10)$$

$$K_c = \frac{C_{Ac}}{c_e} \quad (11)$$

where  $\Delta G$  ( $\text{kJ mol}^{-1}$ ) is change in Gibbs free energy,  $T$  is temperature in Kelvin (273 K),  $R$  is gas constant ( $8.314 \times 10^{-3} \text{ kJ mol}^{-1} \text{ K}^{-1}$ ),  $K_c$  is the equilibrium constant,  $C_{Ac}$  is the equilibrium concentration ( $\text{mg L}^{-1}$ ) of the metal ion on adsorbent and  $c_e$  is equilibrium concentration in the solution.

Enthalpy change ( $\Delta H$ , in  $\text{kJ mol}^{-1}$ ) and entropy change ( $\Delta S$ , in  $\text{J mol}^{-1} \text{K}^{-1}$ ) were obtained from the slope and intercept of Vant Hoff plots of  $\log K_c$  versus  $1/T$  using the following equation [54]:

$$\log K_c = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \quad (12)$$

The change in Gibbs free energy was calculated from the equation:

$$\Delta G = \Delta H - T\Delta S \quad (13)$$

## Results and discussion

### Biosorbent characterization

#### Fourier transform infra-red (FT-IR) spectroscopy

FTIR spectroscopy was used to determine which functional groups are present in the native and modified sugar beet pulp. Obtained spectra of native sugar beet pulp (NSBP) and modified sugar beet pulp (MSBP) are represented in Fig. 1.

The intense broad peaks at  $3415 \text{ cm}^{-1}$  for (NSBP) and (MSBP) are attributed to the O–H vibrations due to the stretching of the alcohols, phenols and carboxylic acids in cellulose, lignin and pectin. This is in accordance with the composition of the sugar beet pulp (lignin, cellulose, hemicellulose and pectin) and it indicates the presence of the “available” and associated O–H groups on which metal ions can bond [37, 40, 55–58]. In the spectra of (MSBP) appearance of low intensity peak at  $2927 \text{ cm}^{-1}$  is assigned to the symmetrical or asymmetrical C–H stretching vibrations of hydrocarbon chains [31, 40, 56–58]. This saturated C–H bond, probably originated from compounds formed by

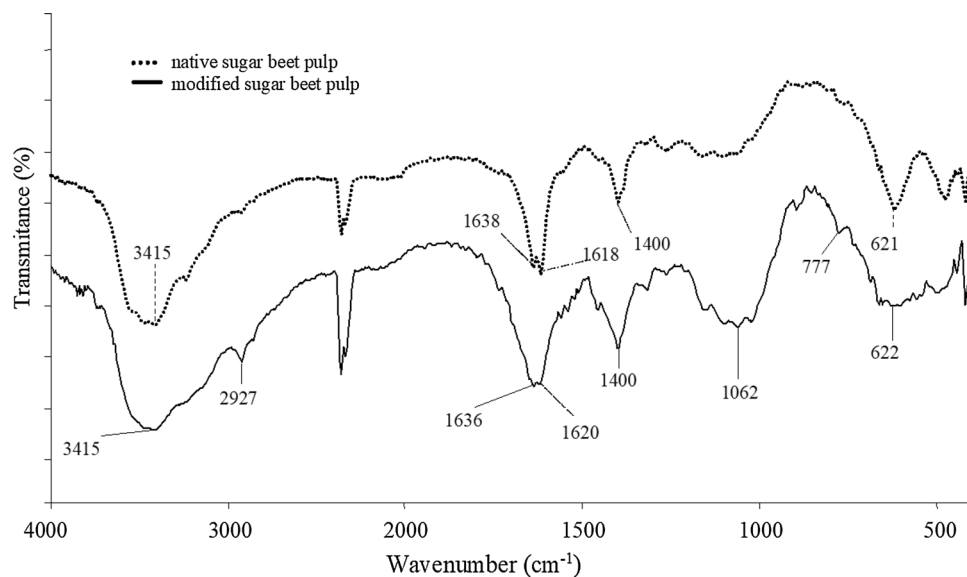
basic hydrolysis of ester group. The peak at  $\sim 2360 \text{ cm}^{-1}$  for (NSBP) and (MSBP) indicates the presence of a triple  $\text{C}\equiv\text{C}$  bond. The bands at  $1638 \text{ cm}^{-1}$  for (NSBP) and  $1636 \text{ cm}^{-1}$  for (MSBP) are assigned to stretching of the  $\text{C}=\text{O}$  in the in the carboxyl groups of pectin and hemicellulose [59–61]. In the spectra of (MSBP), there is an overlap of the band by an intense band at  $\sim 1620 \text{ cm}^{-1}$ , and this can be attributed to asymmetrical stretching of the dissociated carboxyl groups ( $\text{COO}^-$ ) [31, 62]. A peak at  $1618 \text{ cm}^{-1}$  in the spectra of (NSBP) is assigned to the stretching vibrations of the carbonyl group in ester, because of their present in pectins which are obtained from the pulp residue of the sugar industry, and they have a high amount of methylester [40]. In the spectra of both peaks at  $1400 \text{ cm}^{-1}$  can be assigned to symmetric stretching in carboxylate ions [61]. Additionally, in the spectra of (MSBP) a new peak at  $1061 \text{ cm}^{-1}$  can be observed, and it is attributed to the ether groups, which were formed in the presence of NaOH during the modification of the sugar beet pulp. The absorptions at  $622 \text{ cm}^{-1}$  in the spectra of (NSBP) and (MSBP) are assigned to the vibrations of the bonds between the carbon atoms and the halogens in the haloalkanes.

There can't be observed significant differences in the spectra of native and modified sugar beet pulp, changes are only related to the intensities of absorption which were increased in the spectra of modified sugar beet pulp (MSBP) indicating the modification increased a number of the existing functional groups.

#### Scanning electron microscopy (SEM) and electron dispersion spectroscopy (EDS)

Scanning electron microscopy is mainly a non-destructive method of analysis, which provides information about the

**Fig. 1** FT-IR spectra of native and modified sugar beet pulp





morphology, composition and other surface properties of the test sample in this study particularly, about biosorbents (NSBP and MSBP). Figure 2 shows scanning electron microscope picture of NSBP and MSBP. By comparing SEM micrographs (Fig. 2) differences in particle structure, shape and size between NBP and MBP are evident. As can be seen from SEM micrographs the heterogeneity of the surface of the particles on the native sugar beet pulp is noticeable. It has rough, irregular and non-uniform surface, particles are smaller and non-compact, while modified sugar beet pulp particles are larger and more compact.

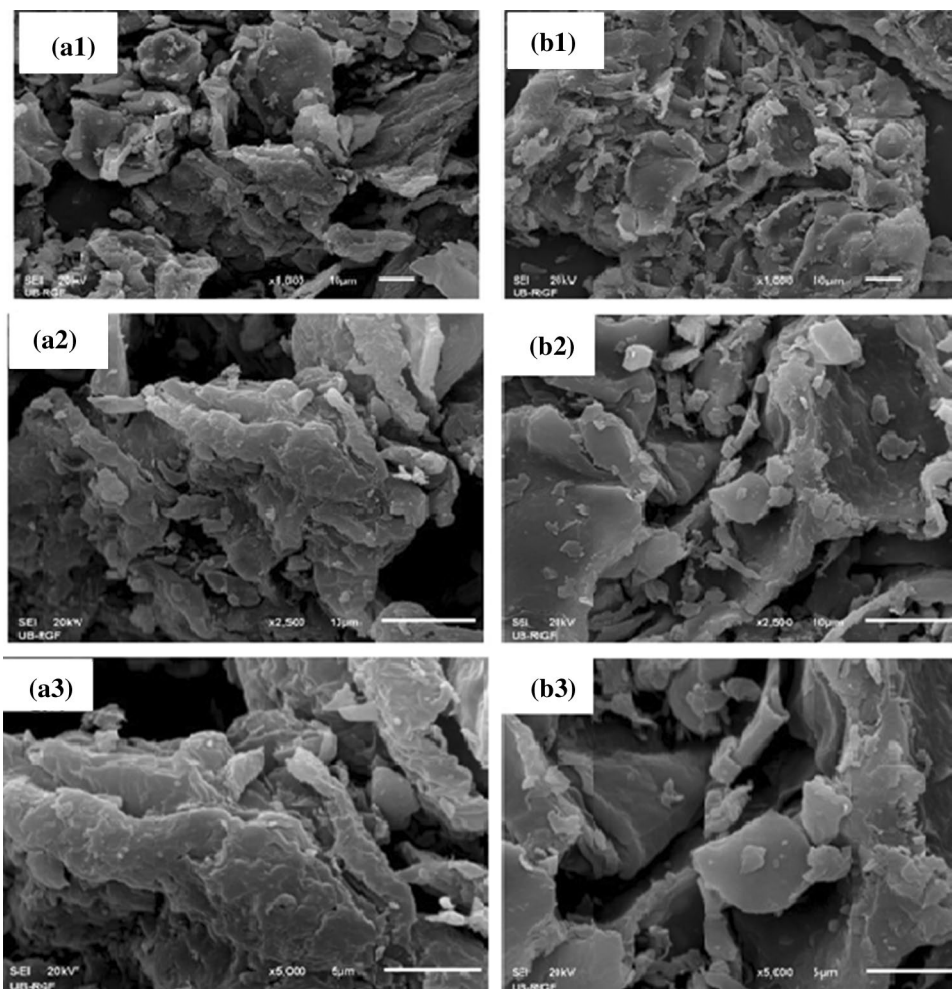
The reason for the increase in MSBP particles may be the agglomeration of the particles due to the increase in the number of functional groups that interact (the most dominant are the electrostatic interactions as well as the hydrogen bond) which brings them closer to each other.

It is evident that the modification resulted in the removal of smaller particles of the native material, and it can be assumed that the acid-alkaline treatment of the sugar beet pulp resulted in the reduction of the “inactive” parts of the biomass, thus making the “active” biosorbent

surface rich in functional groups more accessible for the binding of ions of interest. More precisely, after acid–base treatment, the structure of the MSBP is more homogeneous and smoother than the surface of NSBP particles indicating that the chemical treatment removed impurities from the surface (e.g. waxes and other fats) which were contributed to the roughness of the sugar beet pulp surface and was masking the functional groups available for biosorption [63]. The distribution of particles is less chaotic on the MSBP, and there is a noticeable decrease in the number of channels between the particles due to the agglomeration of particles. Based on the all mentioned above, modification of sugar beet pulp was successful and after it, functional groups are more accessible to water molecules and analyte ions. SEM analysis is an additional confirmation of the results obtained by FTIR spectroscopy, which means that the number of functional groups available for interaction with the analyte has increased.

In Table 1 are given results obtained by EDS analysis showing semi-quantitative composition of the utilized biosorbents surface.

**Fig. 2** SEM micrographs of prepared biomass: **a** native sugar beet pulp **b** modified sugar beet pulp at  $\times 1000$  (**a**<sub>1</sub>, **b**<sub>1</sub>),  $\times 2500$  (**a**<sub>2</sub>, **b**<sub>2</sub>) and  $\times 5000$  (**a**<sub>3</sub>, **b**<sub>3</sub>) magnification



**Table 1** Semi-quantitative surface composition of the used biosorbents obtained by EDS analysis

Material	Content of individual atoms (%)								
	C	Na	Cu	Mg	Si	S	O	K	Ca
NSBP	25.88	0.00	0.47	0.13	1.28	0.15	71.18	0.05	0.87
MSBP	25.22	1.50	0.57	0.14	0.80	0.22	69.98	0.07	0.78

The EDS analysis showed that the surface of NSBP and MSBP is mostly composed of carbon (C) and oxygen (O). Additionally, among alkali and alkaline earth metals magnesium ( $Mg^{2+}$ ), potassium ( $K^+$ ) and calcium ( $Ca^{2+}$ ) ions are also present on the surface. It is evident that the content of sulfur is higher compare to the content of potassium, which may indicate specific sulfur-containing amino acids in the protein structures of the biosorbent. Copper is also present, most probably as the part of metalloenzyme in the plant species. After modification, increased number of  $Na^+$  ions indicated successful modification. Additionally, a decrease in  $Ca^{2+}$  ions is evident, indicating that the mechanism of biosorption could take place via ion exchange. EDS analysis showed decreased number of silicon atoms. Namely, plants take silicon in the form of silica which is needed as a water resource in dry conditions. It is possible during the modification that the silica is converted into the form of sodium metasilicate or water glass, thus reducing the number of silicon atoms.

### Effect of pH on U(VI) biosorption

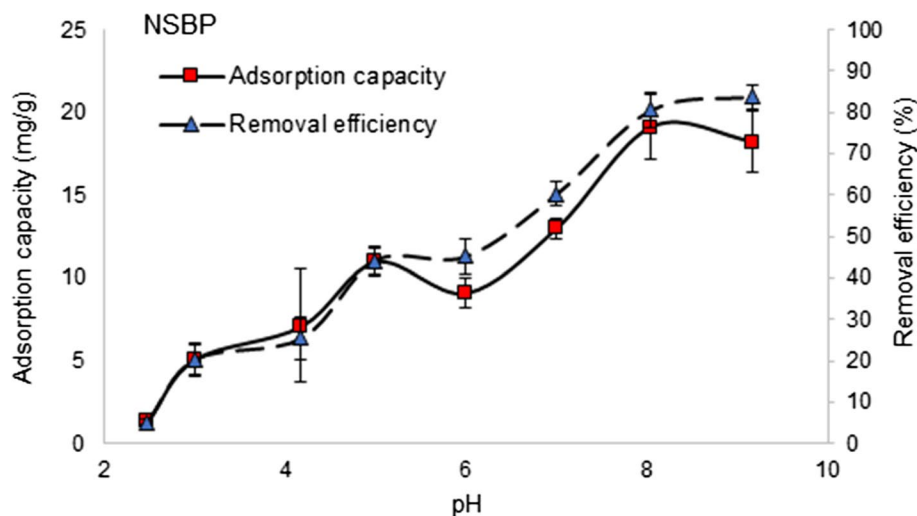
The initial pH of the solution is a very significant parameter for biosorption of metal ions from aqueous solutions on the biosorbents, because it influences the charge of the binding sites present on the adsorbent surface and the solubility and speciation of metal ions [64, 65]. Removal of uranium(VI) from aqueous solution onto NSBP and MSBP was determined in the pH region between 2.0 and 9.0 and

the results regarding adsorption capacity ( $mg\ g^{-1}$ ) and removal efficiency (%) are presented in Figs. 3 and 4. In order to investigate the pH at which the adsorbent surface has net electrical neutrality,  $pH_{pzc}$  was also determined (Fig. 5). Determination of zero point of charge ( $pH_{zpc}$ ) of native (NSBP) and (MSBP) was performed according to Zou an Zhao [66], with a difference that in this study was applied pH value from 1 to 12.

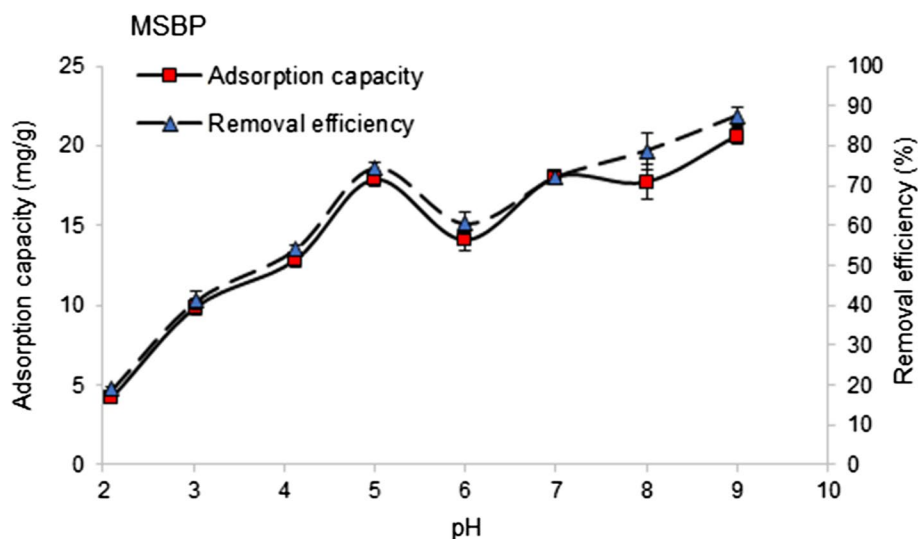
As can be seen in Figs. 3 and 4 the obtained values for the adsorption capacity and efficiency are higher using native sugar beet pulp (NSBP), the maximum adsorption capacity ( $19.10\ mg\ g^{-1}$ ) and removal efficiency (80.58%) are achieved at pH 8. Regarding the obtained value of  $pH_{pzc}$  (5.1) the selected pH value for NSBP was well-chosen because the selected pH was above  $pH_{zpc}$ , which means the surface is negatively charged, and more uranium(VI) ions should be removed from aqueous solution. As the pH increased, the biosorbent surface was in a less degree protonated and negatively charged groups appeared in a gradual increase in uranium biosorption.

The biosorption at pH 8 can be due to the surface complexation phenomenon, because of the dissociation of acidic functional groups ( $-COOH$ ) which were found on the sugar beet pulp. It might be also to the negatively charged surface which bind the positively charged U(VI) cations by electrostatic attraction [4]. The obtained results for NSBP are similar with results obtained for removal of U(VI) onto pollen pini and wood powder [4, 22].

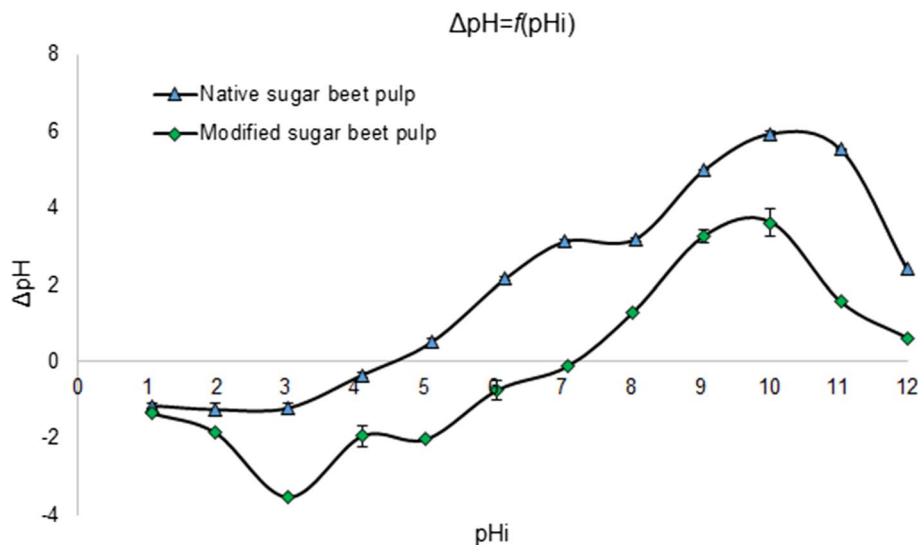
**Fig. 3** Effect of pH on U(VI) adsorption capacity and removal efficiency onto native sugar beet pulp (U(VI) initial concentration =  $50\ mg\ L^{-1}$ ; biosorbent dosage = 100 mg; solution volume = 50 mL; temperature  $\approx 25\ ^\circ C$ ; contact time = 60 min; shaking at 95–100 movements per minute)



**Fig. 4** Effect of pH on U(VI) adsorption capacity and removal efficiency onto modified sugar beet pulp (U(VI) initial concentration = 50 mg L<sup>-1</sup>; biosorbent dosage = 100 mg; solution volume = 50 mL; temperature ≈ 25 °C; contact time = 60 min; shaking at 95–100 movements per minute)



**Fig. 5** The zero point of charge (pH<sub>zpc</sub>) of native (NSBP) and modified (MSBP) sugar beet pulp



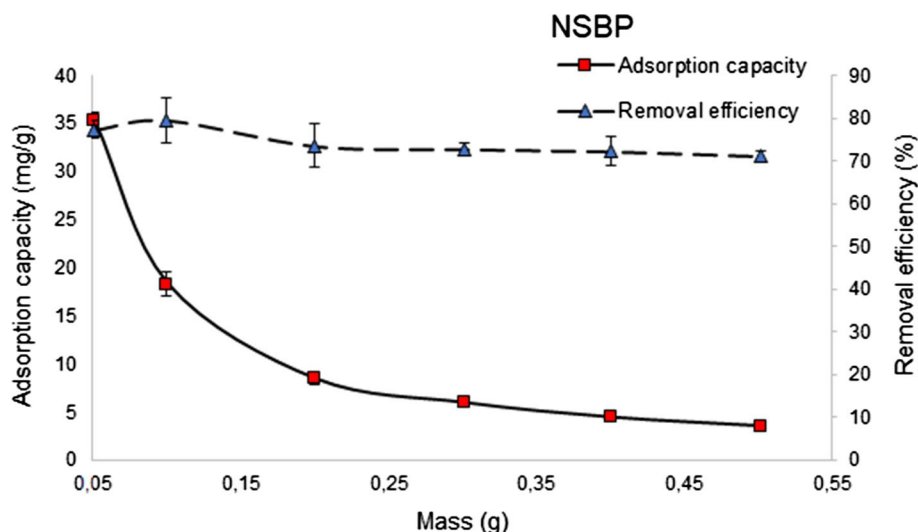
For MSBP highest adsorption capacity and removal efficiency was reached at pH 9 (20.62 mg g<sup>-1</sup> and 87.41%), but because it was notice that at this pH uranium is starting to precipitate, as working pH for MSBP was selected pH 5, because of the stability of uranium species and generally solution (17.88 mg g<sup>-1</sup> and 74.32%, respectively) [19, 47, 65]. The zero point of charge for MSBP (Fig. 5) was achieved at pH 7.1, which means at the pH < 7.1 surface of the biosorbent has more positively charged binding sites and because of that, the less amount of uranium(VI) will be adsorbed on the surface due to the repulsion effect between uranium(VI) ions and positive charge of the MSBP surface. However, in accordance with literature the chosen pH value can be lower or closer to pH<sub>pzc</sub> values [47, 66]. On the basis of all above, as optimal pH values for further experiments pH 8 was chosen for native sugar beet pulp, and pH 5 for modified sugar beet pulp, respectively.

### Effect of biosorbent mass

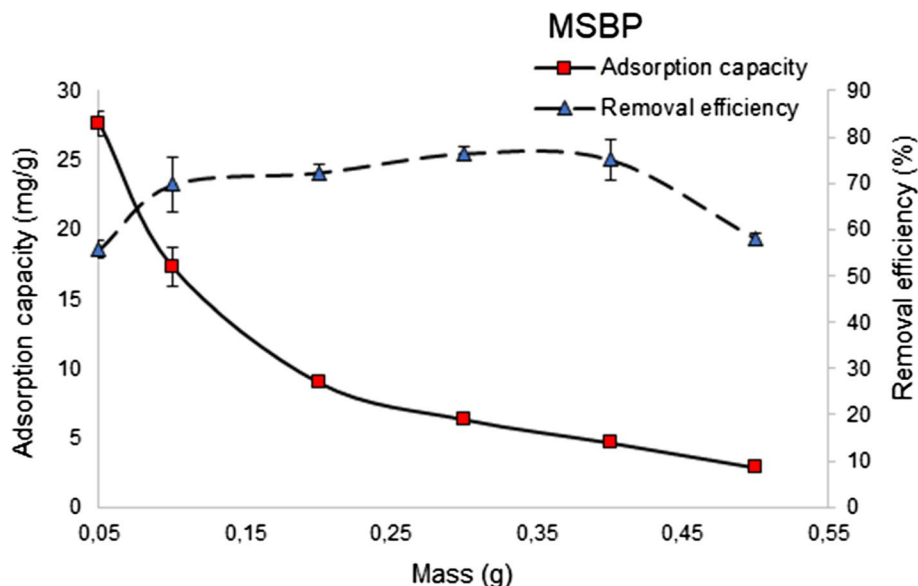
The biosorbent mass is an important parameter to be considered for effective metal ions biosorption. It influences the density of available functional groups for metal biosorption and it controls the sorbate-sorbent equilibrium of the sorption system [67, 68]. The influence of biosorbent mass on biosorption was determined in the range from 0.05 to 500 g at the initial uranium(VI) concentration of 50 mg L<sup>-1</sup> and the results are presented in Figs. 6 and 7.

It was found that the highest adsorption capacity of 18.32 mg g<sup>-1</sup> and 17.33 mg g<sup>-1</sup> was obtained for both used sugar beet pulps (NSBP and MSBP) with 0.1 g of added amount of biosorbent. As can be seen from Figs. 6 and 7 increase in biosorbent dosage decreased the adsorption capacity, for NSBP from 18.32 to 3.56 mg g<sup>-1</sup> and for

**Fig. 6** Effect of biosorbent amount on U(VI) adsorption capacity and removal efficiency onto native sugar beet pulp (U(VI) initial concentration = 50 mg L<sup>-1</sup>; solution pH = 8; solution volume = 50 mL; temperature ≈ 25 °C; contact time = 60 min; shaking at 95–100 movements per minute)



**Fig. 7** Effect of biosorbent amount on U(VI) adsorption capacity and removal efficiency onto modified sugar beet pulps, (U(VI) initial concentration = 50 mg L<sup>-1</sup>; solution pH = 5; solution volume = 50 mL; temperature ≈ 25 °C; contact time = 60 min; shaking at 95–100 movements per minute)



MSBP from 17.33 to 2.90 mg g<sup>-1</sup>. Obtained results are opposite due to the fact that the number and availability of the surface area of the biosorbent and biosorptive sites should be increased with an increase in the biosorbent dosage [63]. Decreased available surface area for biosorption of U(VI) ions can be due to partial aggregation and screening influence on the biosorbent surface which occurs at high biosorbent dosage [69]. Additionally, the mass exchange could be slow down, due to the increase of biosorbent amount mixing efficiency [31, 68]. Decreased biosorption capacity with increased amount of biosorbent dosage was also found in other similar studies in which removal of uranium(VI) from aqueous solution were performed using biomass [21, 27, 31, 70].

### Biosorption kinetic modelling

Kinetics of uranium(VI) biosorption onto MSBP and NSBP from aqueous solution was determined using the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models with the aim to determine the controlling mechanism of biosorption process. The applicability of three mentioned kinetic models was estimated through the kinetic constants ( $k$ ), equilibrium biosorption capacity ( $q_e$ ) and correlation coefficients ( $r^2$ ) which are shown in Table 2.

Obtained results indicate very well fitting to the pseudo second order kinetic model for the uranium(VI) biosorption process. According to the obtained values, the calculated  $q_e$  of the pseudo-second-order model for NSBP (20.37 mg g<sup>-1</sup>) and MSBP (19.16 mg g<sup>-1</sup>) were closest to



**Table 2** Kinetic parameters for the uranium(VI) biosorption onto native and modified sugar beet pulp for pseudo-first and pseudo-second order and intraparticle diffusion models at  $25.0 \pm 0.5$  °C and pH 8.0 (NSBP) and pH 5.0 (MSBP)

Sorbent	$q_{e,exp}$ (mg g <sup>-1</sup> )	Pseudo-first order			Pseudo-second order			Intraparticle diffusion					
		$q_{e,cal}$ (mg g <sup>-1</sup> )	$k_1$ (min <sup>-1</sup> )	$r^2$	$q_{e,cal}$ (mg g <sup>-1</sup> )	$k_2$ (g mg <sup>-1</sup> min <sup>-1</sup> )	$r^2$	$k_{int1}$	$r_1^2$	$k_{int2}$	$r_2^2$	$k_{int3}$	$r_3^2$
NSBP	19.78	8.49	0.016	0.988	20.37	0.0049	0.998	0.85	0.996	1.16	0.946	0.50	0.999
MSBP	18.63	7.97	0.026	0.933	19.16	0.0075	0.999	0.53	0.991	0.59	0.981	0.32	0.847

the obtained experimental data for NSBP (19.78 mg g<sup>-1</sup>) and (MSBP (18.63 mg g<sup>-1</sup>) with correlation coefficients (0.998 and 0.999), respectively. The difference between calculated and experimental  $q_e$  for the NSBP was 0.59 mg g<sup>-1</sup> and for MSBP was 0.53 mg g<sup>-1</sup>. The very good agreement of the experimental and theoretical data for the pseudo-second-order indicated that the rate-limiting step of the reaction is controlled by the chemisorption mechanism. A lot of other studies reported that the pseudo-second-order kinetic model describes better uranium sorption data than the pseudo-first-order model [20, 22, 27, 47, 71]. Additionally, intraparticle diffusion model also indicated significant importance for the uranium(VI) biosorption process onto sugar beet pulps (NSBP and MSBP). The plots showed multilinearity in the biosorption which indicates that three steps are operational. The correlation coefficients ( $r^2$ ) for MSBP for the three steps were in the order: 0.991; 0.981; 0.847 and for NSBP were: 0.996; 0.946; 0.999. The first phase is assigned to the diffusion of U(VI) ions toward the outer surface of the native and modified sugar beet pulp or the boundary diffusion layer of the soluble molecules. The second phase is attributed to the sequential biosorption of the U(VI) ions for both used biosorbents, and the third phase is assigned to the final equilibrium stage due to the lower concentration of the uranium ions left in the solution [72–74]. As it is shown in the Table 2, according to the obtained data the intraparticle model fitted well for the native sugar beet pulp. For the modified sugar beet pulp first two stages fitted well indicating that the biosorption process occurred mostly on the biosorbent surface due to the better attainability of the active sites.

The results for biosorption kinetic modeling showed the mechanism of the uranium(VI) biosorption onto NSBP and MSBP is a complex process and the contributors to the process are surface biosorption, intraparticle diffusion and chemisorption.

## Biosorption isotherms

Adsorption isotherm studies are very significant in the determination of the adsorption efficiency. Adsorption isotherms indicate the distribution of metal ion between the solid phase and liquid phase at equilibrium [75, 76]. In this study Langmuir, Freundlich and Temkin isotherm models were applied to experimental data in order to investigate the mechanism of uranium U(VI) adsorption onto modified and native sugar beet pulp. Models were used to calculate isotherm parameters (Table 3) and to estimate the experimental data.

Based on the obtained results the biosorption data for MSBP and NSBP as biosorbents for the uptake of U(VI) followed the Langmuir isotherm model. The high linear correlation coefficient values, with  $r^2=0.966$  (MSBP) and  $r^2=0.981$  (NSBP), indicate the monolayer biosorption process and that the sorption process occurs on homogenous surface of used biosorbents [77]. The maximum biosorption capacities, with  $q_{e,max}=19.80$  mg g<sup>-1</sup> (MSBP) and  $q_{e,max}=20.45$  mg g<sup>-1</sup> (NSBP) suggest very good agreement to the experimental data,  $q_{e,exp}=18.63$  (MSBP) and  $q_{e,exp}=19.78$  mg g<sup>-1</sup>, respectively. Additionally, separation coefficients,  $R_L=0.233$  for modified sugar beet pulp and  $R_L=0.648$  for native sugar beet pulp indicate favorable biosorption and applicability of the Langmuir isotherm model. Moreover,  $K_L$  values for both biosorbents are relatively low which again implies the high affinity of MSBP and NSBP for the uranium(VI). There is not significant differences for MSBP and NSBP for fitting in this model, but as can be seen from the results, NSBP showed better fitting. In most of the studies, the biosorption process of uranium was found to be in the best agreement with the Langmuir isotherm model [27, 31, 47, 78, 79].

Freundlich isotherm model didn't show the agreement with data obtained for NSBP, with  $r^2=0.727$ ;  $1/n=0.444$ ). Opposite, calculated data for MSBP showed a good agreement with this model. Value of  $n$ , obtained for MSBP, which indicates

**Table 3** Isotherm parameters obtained from Langmuir, Freundlich and Temkin models

Biosorbent	Langmuir				Freundlich			Temkin			$q_{e,exp}$
	$q_{e,max}$	$K_L$	$R_L$	$r^2$	$K_F$	$1/n$	$r^2$	RT/b	$A_T$	$r^2$	
NSBP	20.45	0.027	0.648	0.981	0.027	0.444	0.727	95.29	0.114	0.847	19.78
MSBP	19.80	0.165	0.233	0.966	5.04	2.719	0.906	581	1.85	0.884	18.63

whether the biosorption process is favorable or not, suggests the biosorption of U(VI) onto MSBP is highly favorable.

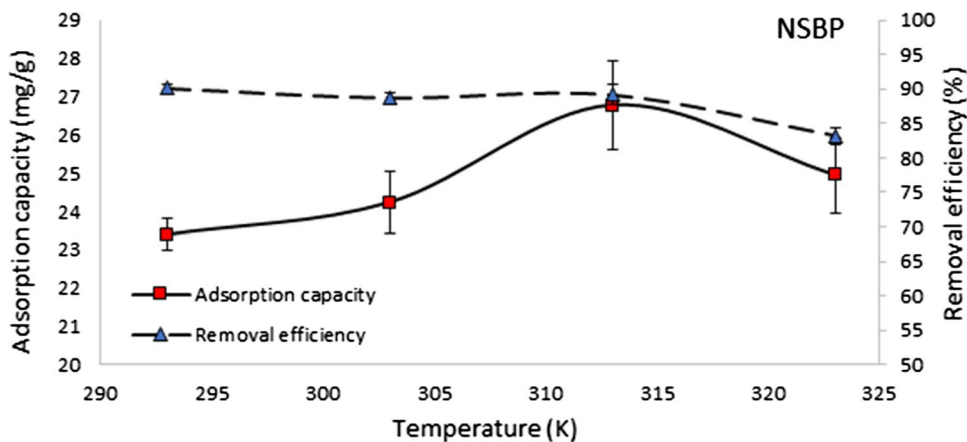
### Biosorption thermodynamics and temperature studies

Effect of temperature on the biosorption of uranium ions onto NSBP and MSBP from aqueous solution was estimated by variation of the temperature in the range from 293 to 323 K, while the other parameters (pH, biosorbent dosage, contact time, initial concentration, volume) were kept constant. As can be seen in Fig. 8 removal of uranium onto NSBP was slightly increased with increasing temperature, and then it was slightly decreased. On other side, for the uranium(VI) biosorption onto MSBP, which is shown in Fig. 9, decrease was even more obvious. This could be explained by the decreased surface activity, and moreover, due to the adsorption theory, molecules adsorbed on the surface of the biosorbents tend to desorb from the surface at higher temperatures [14, 20, 80]. Increased adsorption

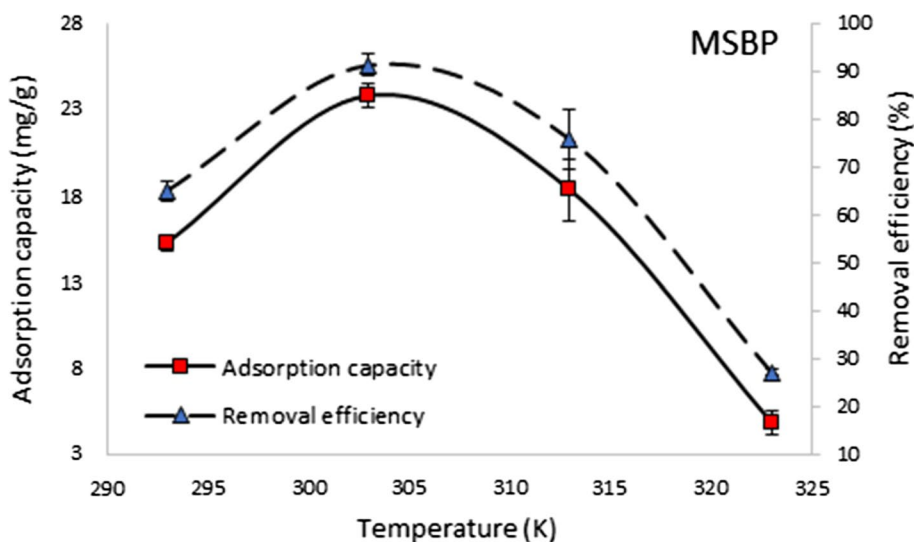
capacity at the beginning for both biosorbents could be due to increased energy of the system. From the obtained results it was established that biosorption of U(VI) onto NSBP and MSBP was temperature dependent process and the optimal temperature was 303 K for both biosorbents, with maximum adsorption capacities  $24.23 \text{ mg g}^{-1}$  and  $23.84 \text{ mg g}^{-1}$ , respectively.

Thermodynamic parameters for biosorption of U(VI) onto NSBP and MSBP ECDS are shown in Table 4. The positive values of  $\Delta H$  suggests that the process is endothermic, meaning the dehydration energy was partly compensated in the releasing of biosorption energy. Also, the magnitude of obtained  $\Delta H$  indicates the physical nature of the biosorption process which is related to hydrophobic and/or electrostatic interactions [78, 81, 82]. The negative values for biosorption entropy ( $\Delta S$ ) indicate decrease randomness at the solid-solution interface during biosorption. Moreover, negative values of obtained entropies are suitable results with the general information of decreasing entropy when U(VI) ions pass the solid phase from the solution. Fixation

**Fig. 8** Effect of temperature on uranium adsorption capacity and removal efficiency onto NSBP (initial U(VI) concentration =  $60 \text{ mg L}^{-1}$ , contact time = 60 min, pH = 8, biosorbent dosage = 0.1 g, solution volume = 50 mL, shaking at 95–100 movements per minute)



**Fig. 9** Effect of temperature on uranium adsorption capacity and removal efficiency onto MSBP (initial U(VI) concentration =  $50 \text{ mg L}^{-1}$ , contact time = 120 min, pH = 5, biosorbent dosage = 0.1 g, solution volume = 50 mL, shaking at 95–100 movements per minute)



**Table 4** Thermodynamics parameters ( $\Delta H$ ,  $\Delta S$ ,  $\Delta G$ ) for biosorption of uranium(VI)

Biosorbents	$\Delta H$ (kJ mol <sup>-1</sup> )	$\Delta S$ (J mol <sup>-1</sup> K <sup>-1</sup> )	$\Delta G$ (kJ mol <sup>-1</sup> )			
			293 K	303 K	313 K	323 K
NSBP	14.06	-0.029	22.58	22.87	23.16	23.45
MSBP	48.28	-0.15	92.42	93.93	95.44	96.94

of uranium(VI) ions as a result of biosorption was assigned to a decrease in the degree of freedom of biosorbate ions which lead to a negative entropy change [83–85]. The positive  $\Delta G$  values indicates non-spontaneous biosorption of uranium ions onto NSBP and MSBP. The confirmation of the non-spontaneous biosorption was further proven through the rise of  $\Delta G^\circ$  values with increase in temperature from 293 to 323 K (Table 4) The similar results for the thermodynamic parameters were obtained in the studies of Prashant et al. [86] and Nazal et al. [87].

### Comparison of adsorption capacities

The comparison of maximum adsorption capacities ( $q_{\max}$ ) of native and modified sugar beet pulp with previous studies are shown in Table 5 [20, 27, 30, 31, 47, 64–66, 85–97]. After comparison with other adsorbents and biosorbents, sugar beet pulp in native and modified form can be considered as a potential biosorbent for removal of uranium(VI) from wastewaters. Additionally, sugar beet pulp represents a low cost, biodegradable and biosorbent with high biosorption effectiveness and it doesn't need high consumption of chemicals.

### Conclusions

Results obtained by the present study confirmed that sugar beet pulp in native/modified form can be used as a low cost industrial waste for the removal of uranium(VI) from the aqueous solution under the optimum conditions pH 5/8, sorbent dosage 0.1/0.1 g, initial uranium concentration 60/50 mg L<sup>-1</sup>, contact time 60/120 min, temperature 303/303 K. Equilibrium data for both biosorbents fitted best to Lagmuir isotherm model, it followed pseudo-second-order model and it is also related to intraparticle diffusion. The positive  $\Delta G$  values at optimal temperature (22.87 kJ mol<sup>-1</sup> and 93.93 kJ mol<sup>-1</sup>) indicated non-spontaneous biosorption of uranium(VI) ions onto NSBP and MSBP. From the obtained results it was established that biosorption of U(VI) onto NSBP and MSBP was temperature dependent process and the optimal temperature was 303 K for both biosorbents. Native sugar beet pulp showed slightly higher adsorption capacity (20.45 mg g<sup>-1</sup>) compared to modified sugar beet pulp (19.80 mg g<sup>-1</sup>). Considering obtained capacity values, biomass preparation of native sugar beet pulp is very fast

**Table 5** Comparison of adsorption capacity of NSBP and MSBP to other adsorbents

Adsorbents	Adsorption capacity (mg g <sup>-1</sup> )	References
SDS-treated rice husk	42.4	[27]
Lemon peel	24.39	[31]
Tea waste	29.41	[88]
Citric acid modified pine sawdust	71.6	[66]
Citrus waste ( <i>Citrus grandis</i> , <i>Citrus aurantifolia</i> , <i>Citrus sinensis</i> )	38.97	[30]
Fresh Spirodela punctata	18.52	[89]
Calcium alginate beads	237.15	[90]
Apricot shell activated carbon	59.17	[65]
Coffee waste	34.8	[91]
Dried tea	59.5	[91]
Impregnated cellulose beads	7.62	[86]
Pine wood sawdust	44.8	[47]
Ceratophyllum demersum	140.45	[64]
Magnetite nanoparticles	8.07	[92]
Hematite	3.36	[93]
Calcined and acid-activated kaolin	4.52	[94]
Multiwalled carbon nanotubes	24.9	[95]
TiO <sub>2-x</sub>	65.41	[96]
Modified silica gel (SiAPMS-HL)	8.46	[97]
Natural sepiolite	34.61	[85]
<i>Eucalyptus citriodora</i> distillation sludge	57.75	[20]
Native sugar beet pulp	20.45	Present study
Modified sugar beet pulp	19.80	

and economic because it doesn't require consumption of any toxic and expensive chemicals.

### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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