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BIOSORPTION OF Cr(III)
FROM AQUEOUS SOLUTION BY ACTIVATED SLUDGE

BIOSORPCJA Cr(III) Z ROZTWORÓW WODNYCH
PRZEZ OSAD CZYNNY

Abstract: The biosorption of Cr(III) from aqueous solution using activated sludge (AS) microorganisms was investigated under various experimental conditions regarding pH and temperature. Biosorption equilibrium parameters were determined based on the Langmuir and Freundlich isotherm model. The kinetic parameters were established using the equations of pseudo-first order and pseudo-second order. Moreover thermo-dynamic parameters have been calculated. Langmuir isotherm provided a better fit to the equilibrium data. The maximum experimentally determined sorption capacity 26.95 mg $\cdot$ g^{-1} obtained at 25°C and pH 5.0. The kinetics model of pseudo-second order row has been better describing experimental data. The negative value of free Gibbs energy (ΔG^0) proves spontaneous Cr(III) biosorption by AS biomass and its decreasing along with temperature increase. The negative value of enthalpy (ΔH^0) and entropy (ΔS^0) indicates exothermic process and limitation of degree of freedom Cr(III) ions on the interphase surface solution/biosorbent. FT-IR spectroscopy analysis indicated the contribution of carboxylate groups towards the biosorption of Cr(III) by AS.

Keywords: activated sludge, biosorption, chromium(III)

Activated sludge is a commonly used biological treatment process to remove dissolved and suspended organic compounds present in wastewaters. Although the standard role of municipal treatment plants was to remove dissolved and suspended organic compounds, metals are also frequently present in the municipal sewage due to industrial wastewaters admitted to the sewerage [1]. Chromium compounds polluting the environment in the form of wastewater, dust and solid waste are generated by many industries, such as mining, metal surface machining, fertilizers manufacturing, tanneries. Aqueous solutions can be purified of chromium by the processes of reversed osmosis, filtration, ion exchange, electrochemical precipitation or extraction [2]. Practical application of these methods is frequently inefficient, due to the generation of large

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volumes of secondary waste, technical or economic limitation, in particular regarding the waste water in which the metal concentration is not higher than 100 mg · dm^{-3} [3].

Adsorption on low cost adsorbents decreases the cost significantly. Biomass from wastewater treatment plants offers the adsorbent at low cost. The excess sludge produced in the process includes both living and nonliving microbial fractions consisting of a variety of species among which bacteria and protozoa [4, 5]. Organic compounds such as carboxyl groups, acidic polysaccharides, lipids and amino acids existing in bacteria cell membrane and wall effectively take part in heavy metal biosorption [4]. Solution pH, temperature, type and quantity of chemical groups, surface area, type of impurities to be adsorbed, type and amount of biomass as well as the presence of other ions are the main factors influencing the metal sorption capacity.

The aim of the presented research were to study the adsorption of chromium(III) ions by activated sludge microorganisms from a wastewater treatment plant. During the experiments, the influence of pH and temperature on the biosorption process was assessed; statics, kinetics and thermodynamic parameters of the process were described.

Material and methods

Activated sludge (AS), a complex consortium of micro-organisms mainly containing bacteria obtained from wastewater treatment plant located in Opole (Poland). Dry biomass pellets were obtained after centrifugation in an MPW centrifuge model 215 at 4000 rpm and then dried at 50°C until constant weight. The stock solution of Cr(III) with 500 mg · dm^{-3} concentration was prepared from Cr(NO$_3$) · 9H$_2$O. The stock solution was reduced, if necessary. Deionized water was used from all solutions.

Chromium(III) concentration before and after biosorption were determined by spectrophotometric method, using the cuvette tests (Hach Lange) and the Photolab Spectral (WTW, Germany) spectrophotometer. Before the measurement, the sample was clarified by vacuum filtration on Whatman membrane filters with 0.45 μm pore size. Biosorption experiments were conducted in Erlenmeyer's flasks with 250 cm^3 capacity, containing 50 cm^3 of Cr(III) solution. The flasks were incubated at the temperature of 30°C and agitated at 120 rpm. The initial pH of the solutions was determined with the use of 0.1 M NaOH and 0.1 M HCl.

The effect of pH on adsorption of Cr(III) onto AS biomass was studied at pH values within the range from 2.0 to 6.0, at the biosorbent addition of 2.0 g · dm^{-3}. The quantity of Cr(III) ions adsorbed by the AS biomass was calculated from the relation:

$$q_e = V \cdot (C_0 - C_e) / M \qquad (1)$$

where: C_0 and C_e are the respective initial and equilibrium Cr(III) concentrations in the solution [mg · dm^{-3}], V the solution volume [dm^3], and M the adsorbent dry weight [g]. The metal sorption efficiency of the biomass was determined by the above-described procedure in every of the following experiments unless stated otherwise.

The impact of temperature at 20, 30 and 40°C on biosorption in equilibrium conditions was analysed for Cr(III) concentrations in the solution, within the range from

11.63 to 231.1 mg · dm^{-3}, adding 2.0 g · dm^{-3} of the biosorbent and setting the solution pH at 5.0. Langmuir [6] and Freundlich [7] isotherm models were applied to this study. Langmuir model is described by the equation:

$$q_e = q_{max} \cdot K_L \cdot C_e / (1 + K_L \cdot C_e) \tag{2}$$

where: K_L – Langmuir constant [dm^3 · mg^{-1}], q_{max} – maximum adsorption capacity [mg · g^{-1}]. K_L and q_{max} values were determined on the basis of linear relations $1/q_e$ from $1/C_e$.

In addition, the Freundlich isotherm was also applied:

$$q_e = K_F \cdot C_e^{1/n} \tag{3}$$

where: K_F – constant of Freudlich equation [mg · g^{-1}], n – exponent of Freundlich equation. Both K_F, and n were calculated on the basis of direction coefficients and shift coefficients of the lines determined for the relation of $\ln q_e$ from $\ln C_e$.

Analyses of temperature at 20, 30 and 40°C, on the kinetics of the biosorption process, were carried out in Erlenmeyer flasks of 1000 cm^3 capacity, containing 400 cm^3 of Cr(III) solution with concentration of 50 mg · dm^{-3} and pH 5.0.

The pseudo-first order rate expression of Lagergren [8] is:

$$(dq / dt) = k_1 \cdot (q_e - q_t) \tag{4}$$

where: q_e and q_t Cr(III) – ions biosorption volume, in equilibrium and after time t [mg · g^{-1}] respectively, k_1 – Lagergren model speed constant [min^{-1}]. The parameters q_e and k_1 were calculated by linear regression method, from the relation $\log (q_e - q_t)$ from t.

Pseudo-second order [9] can by described by:

$$(dq / dt) = k_2 \cdot (q_e - q_t)^2 \tag{5}$$

where: k_2 – speed constant of pseudo-second order model [g · mg^{-1} · min^{-1}]. The parameters q_e and k_2 were calculated on the basis of gradient and shift coefficients of the lines, from the relation t/q_t from t.

Thermodynamic parameters including the change in free energy (ΔG^o), enthalpy (ΔH^o) and entropy (ΔS^o) were calculated from the following equations:

$$\Delta G^0 = -RT \cdot \ln K_c^{\,0} \tag{6}$$

where: R is the universal gas constant [8.314 J · mol · K^{-1}] and T is temperature [K], $K_c^{\,0}$ is the equilibrium constant obtained from the Langmuir isotherm [10].

The relationship between the equilibrium constant, $K_c^{\,0}$ and the temperature is given by the Van't Hoff equation:

$$\ln K_c^{\,0} = (\Delta S^0 / R) - (\Delta H^0 / RT) \tag{7}$$

The plot $\ln K_c^0$ versus $1/T$ enables us to obtain the entropy (ΔS^0) and enthalpy (ΔH^0) values from the slope and the intercept, respectively.

The functional groups of AS microorganisms and possible chromium(III) binding sites were detected by FT-IR analysis (Fourier transform-infrared spectrometer, Nicolet Nexus, USA Nicolet Co.). The FT-IR spectra obtained at 400–4000 cm^{-1} were used to examine the activated sludge before and after metal-loaded (for 24 h in 50 mg $\cdot$ dm^{-3}) initial Cr(III) concentration.

All data shown are the mean values of three replicate experiments, and error bars are indicated wherever necessary.

Results and discussion

Solution pH in an important process parameter in sorption of pollutant from aqueous solution. Results are given in Fig. 1.
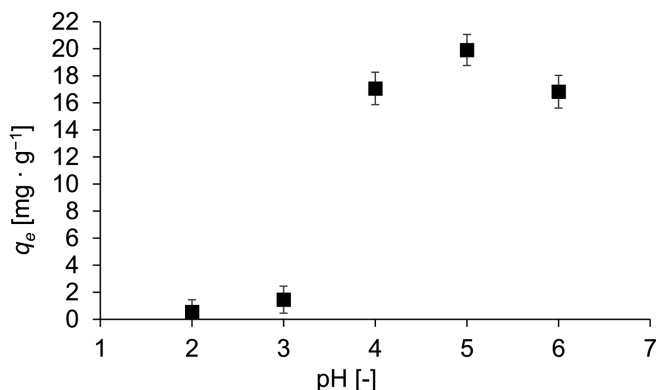

Fig. 1. The effect of pH on adsorption

The presented data show that the AS biomass sorption capacity was increasing in line with the increase of pH. The lowest value was recorded for pH 2.0, and the highest for pH 5.0, and they amounted to 0.54 and 19.91 mg $\cdot$ g^{-1}, respectively. At low pH, these groups are protonated and H$^+$ cations compete with chromium(III) ions for the active spots in AS biomass surface. The increase of pH causes deprotonation of functional groups, biomass surface generates negative load, which supports binding Cr(III) ions [3, 4, 11, 12]. In turn, the solution pH above 5.0 causes precipitation of chromium(III) hydroxide [2]. Other authors [13–15] have found the same trend for chromium(III) sorption by other sorbent materials.

The Langmuir and Freundlich adsorption isotherms were obtained at various temperatures and linearized Langmuir isotherm was given in Fig. 2. Table 1 presents the calculated isotherms coefficients.

The experiment data were better adjusted to Langmuir than Freundlich isotherm model. Linear regression coefficients of Langmuir model were > 0.989. The temperature increase from 20 to 40°C caused the decreased of AS biomass maximum
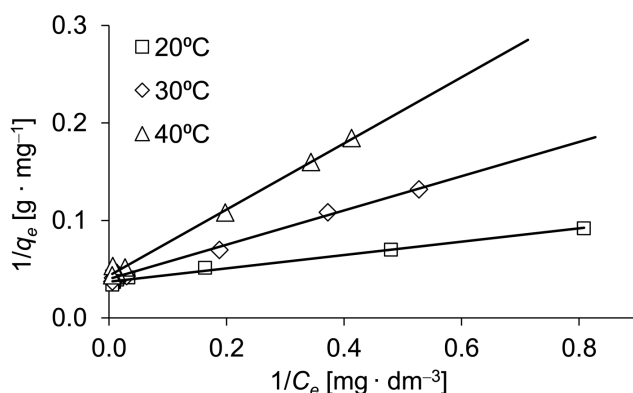
Fig. 2. Linear Langmuir isotherm plots at different temperature

Table 1

Langmuir and Freundlich isotherm constants at different temperature

Temperature [°C]	Langmuir			Freundlich		
	K_L [$dm^3 \cdot mg^{-1}$]	q_{max} [$mg \cdot g^{-1}$]	R^2	K_F [$mg \cdot g^{-1}$]	n	R^2
20	0.542	26.95	0.990	12.19	5.675	0.935
30	0.227	25.06	0.989	7.769	4.147	0.883
40	0.127	23.15	0.996	4.795	3.134	0.924

sorption capacity (q_{max}), the respective values were 26.95 and 23.15 mg · g^{-1}. The maximum monolayer capacity of some adsorbent materials by various researches were determined as 25.64 mg · g^{-1} by using waste-activated sludge [16], 120.0 mg · g^{-1} by freeze-dried activated sludge [17], 28.16 mg · g^{-1} by algal biomass [18] and 21.64 mg · g^{-1} by soybean meal waste [19].

The correlation coefficient of linear Freundlich equation was > 0.883. The high value of K_F indicates a high adsorption. This is defined as the adsorbate adsorbed per unit weight of adsorbent. Higher the n ($n > 1$) value, higher is the intensity of adsorption [12].

The trends in Fig. 3 display that increases temperature from 20 to 40°C gave rise to a positive effect on biosorption. Many authors indicate that decreasing biosorption capacity at increased temperature indicate that the process is an exothermic [5, 12]. Therefore, the test results obtained at different operating temperatures in this study suggested that adsorption of Cr(III) on AS biomass is controlled by exothermic processes. The intensity of binding of Cr(III) by AS was highest during the first 5 minutes of biosorption, during which from 78.12 to 92.58% of chromium(III) were bound. The next stage, in which equilibrium was reached, lasted about 60 minutes. According to Fig. 3, equilibrium sorption capacity was 25.07, 20.06 and 15.95 mg · g^{-1} for 20, 30 and 40°C, respectively.
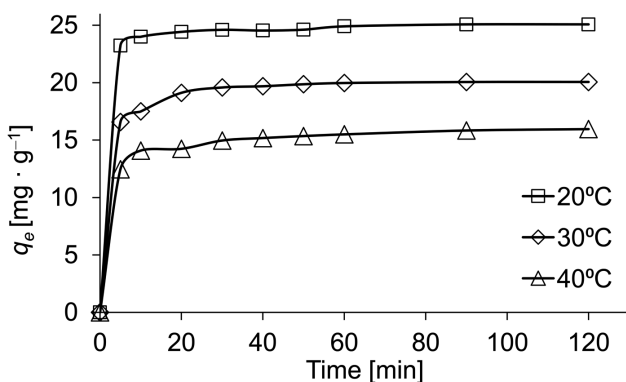
Fig. 3. The effect of temperature on sorption kinetics

The kinetic equations pseudo-first order and pseudo-second order have been applied to description of the research, in order to more fully interpret the obtained results. Both models assume reversibility and equilibrium of the adsorption process. Model para-meters (determined by linearization) for kinetic experiments preformed at different temperature (20–40°C) (Figs. 4 and 5) are presented in Table 2.
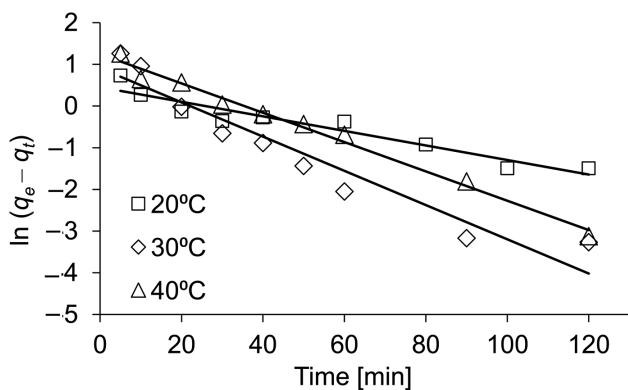

Fig. 4. Comparison of pseudo-first order sorption kinetics at different temperature

The results indicated that increase in temperature decreased both the pseudo-first and pseudo-second order rate constants for the adsorption process. The pseudo-second order kinetic model defines the measurements very well with the correlation coefficients very high (> 0.999). Also the q_e values fitted the experimental data (q_{exp}). The biosorption efficiency decreased, together with the increase of the temperature. This means that biosorption occurs is slower at higher temperatures. Also the amount of chromium(III) ions adsorbed by AS biomass decreased when the temperature of the process increased from 20 to 40°C, respectively, from 25.19 to 16.18 mg · g^{-1} (Table 2).
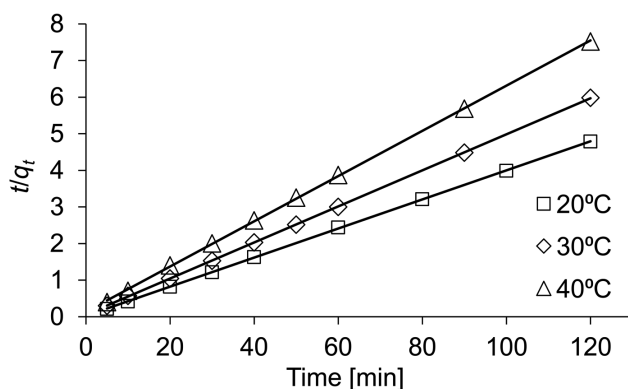
Fig. 5. Comparison of pseudo-second order sorption kinetics at different temperature

Table 2

Comparison of the pseudo-first and pseudo-second rate constants at different temperature

Temperature [°C]	q_{exp} [mg · g^{-1}]	Pseudo-first order			Pseudo-second order		
		k_1 [min^{-1}]	q_e [mg · g^{-1}]	R^2	k_2 [g · mg^{-1} · min^{-1}]	q_e [mg · g^{-1}]	R^2
20	25.3	0.04	1.571	0.914	0.056	25.19	0.999
30	20.1	0.095	2.484	0.919	0.040	20.33	1.000
40	16	0.081	3.479	0.986	0.028	16.18	0.999

The pseudo-first order kinetic model of Lagergren was characterized by lower values of determination coefficient ($0.914 < R^2 < 0.986$) and not fit well to the whole range of contact time. It was generally applicable over the initial stage of the adsorption processes [20]. The calculated q_e values did not give reasonable value, which were too low to compare with experimental data (q_{exp}). This fact may suggest the chemisorption nature of binding of chromium(III) ions by AS biomass [12, 20, 22].

Gibbs free energy change (ΔG^0), enthalpy (ΔH^0) and entropy change (ΔS^0) are three important parameters used to describe the thermodynamics of a process. Values of thermodynamic parameters are grouped in Table 3.

Table 3

Thermodynamic parameters

Temperature [°C]	ΔG^0 [kJ · mol^{-1}]	ΔS^0 [kJ · mol^{-1}]	ΔH^0 [kJ · mol^{-1}]
20	−24.96		
30	−23.62	−0.104	−55.42
40	−22.89		

All negative values of ΔG^0 indicate that Cr(III) biosorption by AS is a spontaneous process. Similar conclusions have been drawn by Iddou et al. [16] and Yao et al. [17] who studied biosorption of Cr(III) by waste-activated sludge and by freeze-dried activated sludge, respectively. As reported by Horsfall and Spiff [22], values of ΔG^0 below -20 kJ · mol^{-1} are characteristic for electrostatic reactions between adsorbent and adsorbate (physical adsorption), whereas those above -40 kJ · mol^{-1} for covalent bonds (chemisorption). The ΔG^0 values obtained during own research, within the range from -22.89 to -24.96 kJ · mol^{-1} do not explicitly suggest the biosorption type, but they may indicate the advantage of chemical sorption. The negative value of the change of enthalpy of the system (ΔH^0) indicates exothermic character of the biosorption process. In an exothermic process, the total energy absorbed in bond breaking is less than the total energy released in bond making between metal and biomass, resulting in the release of extra energy in the form of heat [23]. Negative entropy value (ΔS^0) indicates an affinity of chromium(III) from the AS surface.

FT-IR analysis of AS microorganisms was given in Fig. 6. The position of the absorption bands in the FT-R spectra is related to the change in energy of particles resulting from the stretching and bending vibration of the atoms. The FT-IR spectroscopic analysis showed strong peaks at 3401, 2926, 1653, 1538, 1413, 1244 and 1079 cm^{-1} in the absence of Cr(III). These peaks were characteristic for bonded hydroxyl group (–OH), asymmetric stretch of CH_2, stretching vibration of COO, C=O and C–N (amide I), protein N–H bend, C–N stretch (amide II), protein C–N stretch (amide III), secondary amide and Pyridine(I), respectively [23]. The AS biomass is known as a rich organic (bacteria, algae and protozoa) and inorganic mass. The biochemical composition of organic biomass are protein, lipid, extra cellular polysaccharides and nucleic acids [12].
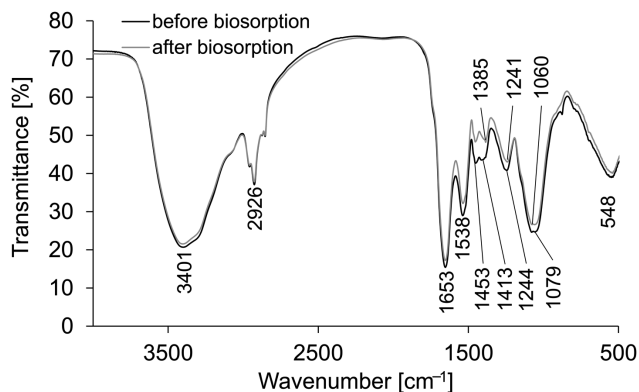

Fig. 6. FTIR spectra of AS biomass before and after biosorption

After the chromium(III) sorption experiment, using AS biomass, Cr(III) loaded AS was dried in an oven at 60°C for removal of water. The only observable change in the spectrum of the Cr(III) exposed AS was in the symmetrical stretch of the carboxyl group, which showed higher intensity. Similar change which shifted from 1413 to

1385 cm^{-1} was observed earlier by Ashkenazy et al. [25]. This shift peaks can be attributed to a change in the counter ion associated with the carboxylate anion. These data suggest that acidic groups, particularly carboxylate, contribute to the chromium(III) ion uptake [24].

Conclusions

The obtained results indicate that the optimum parameters of Cr(III) adsorption by the AS biomass were as follows: pH 5.0 and temperature 20°C. The maximum Cr(III) biosorption capacity was determined as 26.95 mg · g^{-1} at 20°C and pH 5.0. The batch experimental results fitted well to the Langmuir isotherms model. The biosorption capacity of AS was decreased with increasing solution temperature. The kinetic study indicates that the rate of biosorption conforms to the pseudo-second kinetic rate equation. The negative value of free Gibbs energy (ΔG^0) proves spontaneous Cr(III) biosorption by AS. The negative value of enthalpy (ΔH^0) and entropy (ΔS^0) indicates exothermic of process and limitation of degree of freedom Cr(III) ions on the interphase surface solution/biosorbent. FT-IR showed the presence of different groups on the surface of the biosorbent. Because marked change observed at carboxyl group peak (1385 cm^{-1}) in chromium(III) loaded AS, carboxyl groups were the dominant species in the Cr(III) biosorption mechanism. The obtained results confirm the usefulness of the AS biomass in the process of Cr(III) biosorption from the wastewaters.

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BIOSORPCJA Cr(III) Z ROZTWORÓW WODNYCH PRZEZ OSAD CZYNNY

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Abstrakt: W pracy badano proces biosorpcji jonów chromu(III) z roztworów wodnych przez mikroorganizmy osadu czynnego (OC), w różnych warunkach pH i temperatury. Parametry równowagowe biosorpcji wyznaczono w oparciu o modele izoterm Langmuira i Freundlicha. Parametry kinetyczne określono za pomocą równań pseudopierwszego i pseudodrugiego rzędu. Obliczono również parametry termodynamiczne procesu. Izoterma Langmuira lepiej opisywała dane równowagowe. Maksymalna pojemność sorpcyjna wyznaczona eksperymentalnie wynosiła 26,95 mg · g^{-1} przy 25°C i pH 5,0. Model kinetyki pseudodrugiego rzędu lepiej opisywał dane doświadczalne. Ujemna wartość energii swobodnej Gibbsa (ΔG^0) świadczyła o spontaniczności biosorpcji Cr(III) przez biomasę OC i jej spadku wraz ze wzrostem temperatury. Ujemna wartość entalpii (ΔH^0) i entropii (ΔS^0) wskazywała na egzotermiczność procesu i ograniczenie stopni swobody jonów Cr(III) na powierzchni międzyfazowej roztwór/biosorbent. Analiza FT-IR wykazała udział grup karboksylanowych w biosorpcji Cr(III) przez OC.

Słowa kluczowe: osad czynny, biosorpcja, chrom(III)