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Original scientific paper

Studies on chromium(III) removal from aqueous solutions by sorption on *Sphagnum* moss peat

CATALIN BALAN, DOINA BILBA* and MATEI MACOVEANU

Department of Environmental Engineering and Management, Faculty of Chemical Engineering and Environmental Protection, Technical University of Iasi, D. Mangeron Blvd. 71A, 700050, Iasi, Romania

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Abstract: Batch sorption experiments were performed for the removal of chromium(III) ions from aqueous solutions using Romanian *Sphagnum* moss peat (untreated and treated with NaCl solution) as sorbent. In order to establish the best conditions for the sorption of chromium(III), the influence of initial pH, contact time, peat dose and metal ion concentration was investigated. The Freundlich, Langmuir and Dubinin–Radushkevich models were applied to describe the sorption isotherms and to calculate its constants. The experimental data fitted well to the Langmuir model with a maximum sorption capacity of 18.6 mg Cr(III)/g of peat. The mean free energy of sorption suggests that the binding of Cr(III) on peat occurred through an ion exchange mechanism. The kinetic data evaluated by pseudo-first order and pseudo-second order kinetic models showed that the sorption of chromium onto the peat followed a pseudo-second order rate equation. The chromium(III) could be easily eluted from the loaded peat using 0.10 M HCl and the peat may be reused in several sorption/desorption cycles. The experimental results indicated the potential of *Sphagnum* moss peat for removal of Cr(III) from wastewaters.

Keywords: *Sphagnum* moss peat; Cr(III), sorption; equilibrium studies; kinetics.

INTRODUCTION

Chromium is an important metal used in many industrial activities, including electroplating, leather tanning, electric-power production, pulp production, paint and pigment manufacture and ore and petroleum refining. These activities produce significant quantities of chromium wastes, which can generate a considerable pollution of water and soil.

The stable oxidation states of chromium in the environments are Cr(III) and Cr(VI), which are found in several different forms according to pH.¹ The toxicity of soluble chromium species and their mobility in aquatic and terrestrial envi-

* Corresponding author. E-mail: dbilba@ch.tuiasi.ro

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ronments depends on their oxidation state. Cr(III) is considered less toxic than Cr(VI) and even essential to human health in trace concentrations. However, at increasing concentrations, Cr(III) can cause adverse effects because of its high capability to coordinate various organic compounds, resulting in inhibition of some metallic-enzyme systems.^{1,2} Meanwhile, Cr(VI) is a confirmed human carcinogen and epithelial irritant.^{2,3} The tolerance limit for aqueous effluents discharge into inland waters is 1.0 mg/L for total chromium (Cr(III) and Cr(VI)) and 0.10 mg L⁻¹ for Cr(VI).⁴

Several technologies have been developed to remove heavy metals, particularly chromium, from industrial wastewater; these include chemical precipitation, coagulation/flocculation processes, membrane filtration, oxidation processes, activated carbon adsorption, reverse osmosis, ion exchange and solvent extraction.⁵ Many of these processes are often complicated and time consuming, generate slugs or other toxic wastes and may be ineffective or expensive. Adsorption is one of the important methods for the removal of heavy metals at medium and low concentrations (1–100 mg/L) from wastewaters. The high cost of commercial activated carbons has stimulated the search for cheaper alternatives and, recently, non-conventional and low cost agricultural by-products have been employed as adsorbents in the removal of heavy metals.^{6–9} Various materials of this type have been investigated for the removal of chromium species; these include agricultural or industrial wastes processed into activated carbons,^{10–13} peat,^{14,15} sawdust,¹⁶ and several biomasses.^{17–20}

Peat is an inexpensive and widely available natural material, consisting of organic matter at various stages of decomposition; the main components are lignin, cellulose, and humic and fulvic acids. These components contain various polar functional groups, especially weakly acidic groups such as carboxyl and phenolic hydroxide that can be involved in chemical bonding of transition metals and polar organics.²¹ The ability of peat to remove heavy metals, such as copper, cadmium, lead, nickel and chromium, has been reported by many authors.^{21–30}

In this study, the sorption capacity of Romanian *Sphagnum* moss peat for chromium(III) ions was investigated under batch conditions, whereby various process parameters, such as initial pH, adsorbent dose, chromium concentration and contact time, were considered. The sorption data were tested fitted to a number of isotherm and kinetic models, with the view of understanding the mechanism of Cr(III) sorption using moss peat as the adsorbent.

EXPERIMENTAL

Materials

In this study, a commercially available *Sphagnum* moss peat (Poiana Stampei, Romania) was used. The material was air-dried, ground and sieved to particles size 1–2 mm. Some of the main characteristics of the peat were determined according to standard methods; the

results (Table I) suggest a peat with a low degree of decomposition. This was designated P-H (peat in hydrogen form).

TABLE I. Characteristics of the *Sphagnum* moss peat

Property	Value
pH (1:50, w/v deionised water)	4.05±0.05
Ash content, mass % w/w	4.85
Organic mater, mass %	84–85
Moisture content, mass %	10.5–11
Cation exchange capacity, meq g ⁻¹ of peat ^a	0.575

^aDetermined by pH-metric titration in 0.1 M NaCl solution

In order to improve the dissociation of the weak carboxylic groups from the peat, an amount of sorbent was treated for 24 h with 1.0 M NaCl solution, washed with distilled water and dried. This sorbent was designated P-Na (peat in sodium form).

A stock solution of Cr(III) (520 mg/L) was prepared by dissolving analytical reagent grade chromium nitrate (Cr(NO₃)₃·9H₂O) (Sigma) in distilled water. All working solutions were prepared by adequate dilution of the stock solution with distilled water.

Sorption experiments

Batch sorption experiments were performed in 250 mL conical flasks by shaking the required amount of peat (P-H or P-Na) with 50 mL of aqueous solutions of Cr(III) of known concentration, at a constant temperature (20±1 °C). The pH of the solutions was adjusted to a constant value by the addition of dilute solutions of H₂SO₄ and NaOH or acetate buffer solution and measured with a pH-meter, Radelkis OP-271 pH/Ion analyzer. After a specified contact time, the peat was separated by filtration and the residual concentration of Cr(III) in the solution (previously oxidized with KMnO₄) was analyzed by a spectrophotometric method with diphenylcarbazide using a UV–Vis digital spectrophotometer, model S 104D/WPA.

The amount of Cr(III) adsorbed on the peat at equilibrium, q (mg g⁻¹), and the percentage of Cr(III) removed, R (%), were calculated using the following equations:

$$q = (c_0 - c)V / m \quad (1)$$

$$R = (c_0 - c)100 / c_0 \quad (2)$$

where c_0 and c are the Cr(III) concentrations (mg L⁻¹) in initial solution and after equilibrium, respectively, V is the volume of the solution (L) and m is the mass of *Sphagnum* moss peat (g).

The effect of operating process parameters on the removal of chromium(III) by peat was investigated according to Table II.

TABLE II. Experimental conditions used for the sorption of Cr(III) onto *Sphagnum* moss peat

Operating variable	Initial pH	Amount of peat g L ⁻¹	Cr(III) Concentration mg L ⁻¹	Contact time h
pH	1.0–5.5	4	26 and 52	24
Peat dose	4	2.0–12	83.2	24
Cr(III) concentration	4 and 5	4	10.4–104	24
Contact time	4	4	41.6 and 83.2	15 min–4 h

Adsorption isotherm models

The equilibrium data for Cr(III) removal using peat as the sorbent were analysed using different isotherm models available in the literature.^{24,31-33}

The Freundlich isotherm, which assumes a heterogeneous adsorption surface, was tested using the following linear equation:

$$\log q = \log K_F + 1/n \log c \quad (3)$$

where K_F and $1/n$ are constants related to the adsorption capacity and adsorption intensity (efficiency), respectively.

The Langmuir isotherm adsorption model is based on the assumption that maximum adsorption corresponds to a monolayer of solute species on the sorbent surface, containing a finite number of energetically equivalent sites. The Langmuir equation can be used in following linearized form:

$$1/q = (1/q_0 K_L c) + 1/q_0 \quad (4)$$

where q_0 is the maximum amount of solute adsorbed (mg g^{-1}) and K_L is a constant related to the binding energy of the solute (L mg^{-1}).

In order to appreciate the nature – physical or chemical – of the sorption process, the isotherms data were analyzed by the Dubinin–Radushkevich (D–R) model, expressed by the following equation:

$$\ln q = \ln q_{\max} - \beta \varepsilon^2 \quad (5)$$

where q_{\max} is the maximum amount of solute adsorbed under optimized experimental conditions (mg g^{-1}), β is a constant related to the sorption energy ($\text{mol}^2 \text{kJ}^{-2}$) and ε (the Polanyi potential) is defined by the mathematical relation:

$$\varepsilon = RT \ln(1 + 1/c) \quad (6)$$

where R is the gas constant ($\text{kJ mol}^{-1} \text{K}^{-1}$), T is the absolute temperature and c is the equilibrium concentration in solution (mol L^{-1}).

The mean free energy of sorption, E (kJ mol^{-1}), can be calculated using the following equation:

$$E = 1/\sqrt{2\beta} \quad (7)$$

Values of the mean free energy less than 8 kJ mol^{-1} are characteristic of a physical sorption mechanism and values between 8 and 16 kJ mol^{-1} indicate an ion exchange mechanism.

Kinetic models

The kinetic adsorption data were evaluated using pseudo-first order and pseudo-second order kinetic models.^{32,34}

The pseudo first-order Lagergren model is usually expressed as:

$$\log(q_e - q) = \log q_e - \frac{k_1}{2.303} t \quad (8)$$

where q_e and q are the amounts of sorbed Cr(III) (mg g^{-1}) at equilibrium (24 h) and at any time t (min), respectively, and k_1 is the Lagergren rate constant of the first-order sorption (min^{-1}).

The pseudo-second order model (Ho model) assumes that the sorption follows a second order mechanism and that the rate limiting step may be chemical sorption involving valence

forces or covalent forces between the sorbent and the adsorbate. The rate of pseudo-second order reaction is expressed by the equation:

$$\frac{t}{q} = \frac{1}{q_e^2 k_2} + \frac{1}{q_e} t \quad (9)$$

where k_2 is the rate constant of second order sorption ($\text{g mg}^{-1} \text{min}^{-1}$) and $q_e^2 k_2 = h$ can be regarded as the initial sorption rate ($\text{mg g}^{-1} \text{min}^{-1}$) as t approaches zero.

RESULTS AND DISCUSSION

Effect of solution pH value

The solution pH value is one of the most important controlling parameters in the sorptive removal of heavy metals, influencing not only the dissociation of functional groups and surface charge of the sorbent, but also the solution chemistry of the heavy metals.^{7,35} According to the speciation diagram for Cr(III) complexes,^{36,37} in aqueous solutions at medium and low concentrations (below 10 mmol L^{-1}), about 98 % of the total amount of chromium is present as Cr^{3+} at pH 2, while this value decreases to 40 and the rest 60 % of chromium is in the hydroxylated CrOH^{2+} form at pH 4. The effect of the initial pH value (adjusted with sulphuric acid or sodium hydroxide) on the removal of Cr(III) ions on P-H and P-Na *Sphagnum* moss peat from two solutions of different initial concentration is shown in Fig. 1.

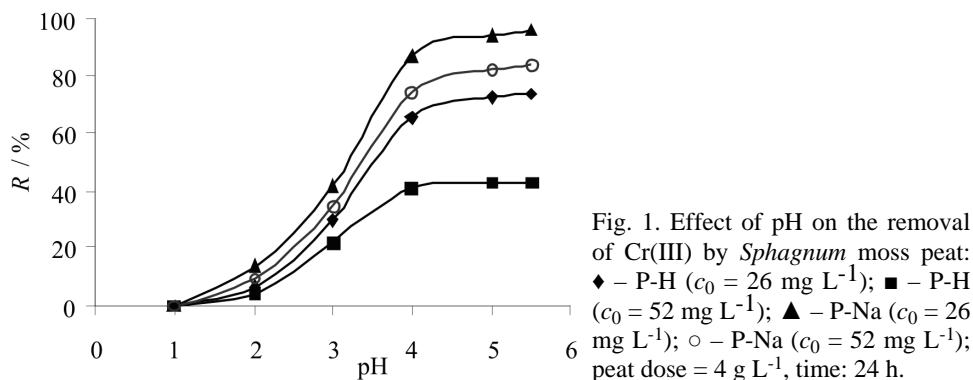


Fig. 1. Effect of pH on the removal of Cr(III) by *Sphagnum* moss peat: \blacklozenge – P-H ($c_0 = 26 \text{ mg L}^{-1}$); \blacksquare – P-H ($c_0 = 52 \text{ mg L}^{-1}$); \blacktriangle – P-Na ($c_0 = 26 \text{ mg L}^{-1}$); \circ – P-Na ($c_0 = 52 \text{ mg L}^{-1}$); peat dose = 4 g L^{-1} , time = 24 h.

It is evident that at a pH lower than 2.0, the adsorption of Cr(III) was negligible but increased rapidly with increasing pH value, with a maximum in the pH range 4.0–5.5. This increase in the percent removal of chromium (from 6.8 to 74 % from solutions of 26 mg Cr L^{-1}) with increasing pH (from 2.0 to 5.5) is due to lower competition of protons for the binding sites and also to the increase in the concentration of CrOH^{2+} species, which exchange with H^+ from the carboxyl groups more readily than Cr^{3+} . For the sodium form of the peat (P-Na), the increase in the dissociation of the carboxyl groups generates a more negatively charged surface and electrostatic interactions with cationic species of Cr(III) is

increased, thus enhancing the sorption. The percentage of chromium removed is smaller when a higher initial concentration was used.

Effect of adsorbent dose

The effect of the concentration of *Sphagnum* moss peat (P-H and P-Na) on the sorption of chromium(III) from solutions of initial concentration 83.2 mg L^{-1} and pH 4.0 is shown in Fig. 2.

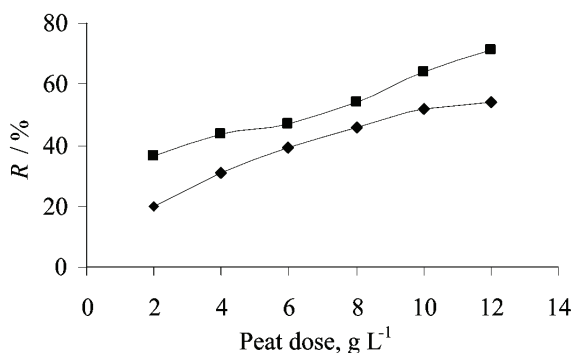


Fig. 2. Effect of *Sphagnum* moss peat dose on the sorption of Cr(III): \blacklozenge – P-H; \blacksquare – P-Na; pH 4, $c_0 = 83.2 \text{ mg L}^{-1}$, time: 24 h.

The sorption of chromium(III) increased with increasing peat dose from 2.0 to 12 g L^{-1} for both P-H and P-Na, probably due to the greater surface area and to more sorption sites on the sorbent. The chromium removal percent was higher for the sodium form of the peat.

Effect of chromium(III) concentration

The effect of the initial Cr(III) concentration on the sorption of chromium (III) onto *Sphagnum* moss peat untreated and treated with NaCl from solutions of various initial pH values is presented in Fig. 3.

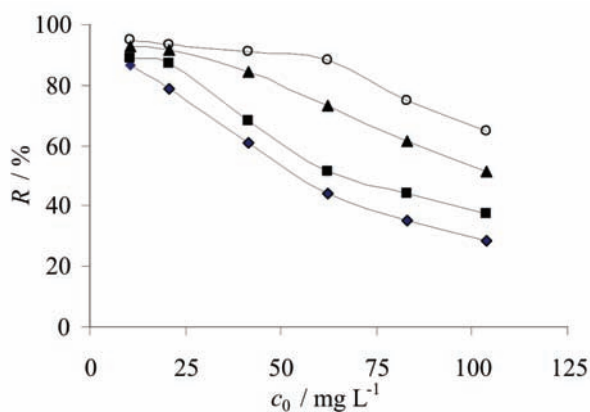


Fig. 3. Influence of initial Cr(III) concentration on the removal of chromium by *Sphagnum* moss peat: \blacklozenge – P-H (pH 4.0); \blacksquare – P-Na (pH 4.0); \blacktriangle – P-Na (pH 5.0, NaOH); \circ – P-Na (pH 5.0, acetate); peat dose: 4 g L^{-1} , time: 24 h.

Increasing the initial Cr(III) concentration from 10.4 mg L^{-1} to 104 mg L^{-1} in solutions with an initial pH 4 decreased the percent removal of Cr(III) by P-H samples from about 86.5 to 28.5 %. This is explained by increasing ratios between the initial number of moles of Cr(III) and the limited number of available sorption sites on the peat. Chromium sorption onto P-Na samples slightly increased, showing that the replacement of protons with Na^+ enhanced the accessibility of the sorption sites as well as the swelling of the peat.³⁸ Also, increasing the initial pH of the solution had a favourable effect on chromium(III) removal; the best results were obtained from solutions of pH 5 adjusted with $\text{CH}_3\text{COOH}-\text{CH}_3\text{COONa}$ buffer.

Equilibrium modelling

Sorption isotherms describe the distribution of adsorbate species between the liquid phase and the solid phase when the sorption process reaches its equilibrium state. An analysis of equilibrium data by fitting them to different isotherm models is important for an estimation of the practical sorption capacity and optimization of the design of sorption systems.

The sorption isotherms of chromium(III) on *Sphagnum* moss peat (in the H- and Na-form) from solutions of various initial pH values are shown in Fig. 4.

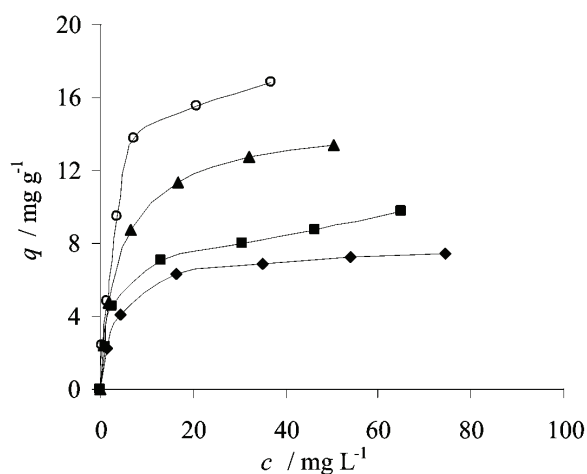


Fig. 4. Sorption isotherms of Cr(III) onto *Sphagnum* moss peat at various pH values: \blacklozenge – P-H (pH 4.0); \blacksquare – P-Na (pH 4.0); \blacktriangle – P-Na (pH 5.0, NaOH); \circ – P-Na (pH 5.0, acetate); peat dose: 4 g L^{-1} , time: 24 h.

The experimental equilibrium sorption data were compared with three adsorption isotherm models: the Freundlich, Langmuir and Dubinin–Radushkevich models, and the best-fit equilibrium model was established based on the linear regression correlation coefficients, R^2 .

The numerical values of the Freundlich, Langmuir and D–R isotherm parameters, evaluated from the slope and the intercept of the corresponding plots ($\log q$ vs. $\log c$; $1/q$ vs. $1/c$ and $\log q$ vs. ε^2 , respectively) are summarized in Table III.

TABLE III. Parameters for the sorption of Cr(III) on *Sphagnum* moss peat

Isotherm parameter		P-H (pH 4.0)	P-Na (pH 4.0)	P-Na (pH 5.0, NaOH)	P-Na (pH 5.0, acetate buffer)
Freundlich	$1/n$	0.299	0.315	0.396	0.454
	$K_F / (\text{mg g}^{-1})(\text{L mg}^{-1})^{1/n}$	2.367	2.762	3.372	4.158
	R^2	0.927	0.911	0.938	0.909
Langmuir	$q_0 / \text{mg g}^{-1}$	7.547	9.609	13.89	18.62
	$K_L / \text{L mg}^{-1}$	0.300	0.279	0.286	0.278
	R^2	0.998	0.991	0.999	0.998
Dubinin–Radushkevich	$q_{max} / \text{mg g}^{-1}$	13.67	17.80	34.74	56.60
	$\beta / \text{mol}^2 \text{kJ}^{-2}$	0.0030	0.0032	0.0038	0.0042
	$E / \text{kJ mol}^{-1}$	12.91	12.50	11.47	10.91
	R^2	0.955	0.949	0.964	0.943

The correlation coefficients higher than 0.9 show that the sorption of Cr(III) ions followed the Freundlich isotherm; the fractional values of $1/n$ ($0 < 1/n < 1$) suggest heterogeneity of the sorbent surface (the closer the $1/n$ value is to zero, the more heterogeneous is the surface³¹) and, simultaneously, indicates a beneficial adsorption of Cr(III) on the peat.

However, the R^2 values from Table III show that the isotherms data of chromium(III) ions sorption can be better described by the Langmuir model, indicating the formation of a monolayer coverage of the adsorbate at the outer surface of the peat. The obtained maximum sorption capacity was slightly higher on the Na-form of the peat than that on the untreated peat and increased with increasing solution pH. The highest capacity was obtained in acetate buffer solutions of pH 5.0. The high sorption equilibrium constant K_L suggests a strong interaction between chromium(III) cations and the binding sites of the peat.

The D–R isotherm parameter q_{max} (mg g^{-1}) indicates the porous structure of the peat sorbent (the maximum amounts of Cr(III) ions that could be sorbed under optimized conditions were much higher than the experimental values). The values of the mean free sorption energy were between 10.9 and 12.9 kJ mol^{-1} , which correspond to an ion exchange mechanism for the sorption of chromium(III) ions on the peat. A slight decrease of the energy sorption values with increasing pH was observed, probably due to an increase of the physical sorption of neutral chromium species.

Kinetic studies

Information on the sorption rate is required for the selection of the optimum conditions for operation in a full-scale batch process.

The results of Cr(III) sorption at pH 4.0 on the *Sphagnum* moss peat in the Na form as a function of contact time between the sorbent and aqueous solutions of adsorbate are presented in Fig. 5.

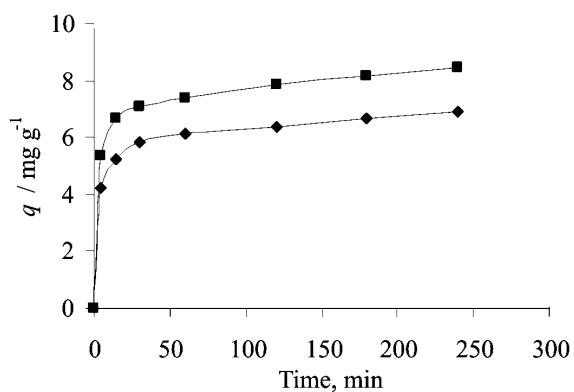


Fig. 5. Effect of contact time on the sorption of Cr(III) onto *Sphagnum* moss peat: \blacklozenge – $c_0 = 41.6 \text{ mg L}^{-1}$; \blacksquare – $c_0 = 83.2 \text{ mg L}^{-1}$; pH 4.0, peat dose (P-Na): 4 g L^{-1} .

As can be seen in Fig. 5, the sorption of Cr(III) ions was extremely rapid during the first 20 min and, thereafter, it decreased significantly near the equilibrium, which can be considered to have been obtained after 4 h (the values of the amount sorbed after 24 h were found to be 2.8 and 3.28 % higher, respectively, than those after 4 h contact). The fast sorption kinetics is typical for sorption of metal due to purely physico-chemical interactions between adsorbent and adsorbate.

The experimental data were interpreted by means of pseudo-first and pseudo-second order kinetic models. The constants of these kinetic models calculated from the linear plots ($\log(q_e - q)$ vs. t and t/q vs. t) and the corresponding correlation coefficients are presented in Table IV.

TABLE IV. Kinetic constants for Cr(III) sorption onto *Sphagnum* moss peat

Kinetic parameter		$c_0 = 41.6 \text{ mg Cr L}^{-1}$	$c_0 = 83.2 \text{ mg Cr L}^{-1}$
Pseudo-first order model	$q_0 / \text{mg g}^{-1}$	9.343	9.813
	k_1 / min^{-1}	1.396	1.282
	R^2	0.8868	0.8947
Pseudo-second order model	$q_0 / \text{mg g}^{-1}$	6.887	8.525
	$h / \text{mg g}^{-1} \text{ min}^{-1}$	1.148	1.404
	$k_2 / \text{g mg}^{-1} \text{ min}^{-1}$	0.0242	0.0193
	R^2	0.9976	0.9988

The R^2 values below 0.90 suggest that the Lagergren model is not a good model for the kinetic data; in addition, the estimated values of q_e do not agree very well with the experimental values.

The experimental data complied very well with the pseudo-second order kinetic model, with correlation coefficients higher than 0.99. It was observed that the initial metal ion concentration influenced the kinetic parameters. The values of the initial sorption rate, h , increased with increasing initial Cr(III) concentration, probably due to the greater concentration gradient between the sorbent and

solution. However, the values of rate constant, k_2 , decreased with increasing initial concentration. In addition, the good correspondence between the calculated and experimental values of the sorption capacity showed that the kinetic data are in agreement with the pseudo-second order rate equation.

Desorption studies

In order to check the reusability of the both the peat and Cr(III), several loading and elution experiments were performed. The desorption of Cr(III) ions from loaded peat was tested under batch conditions using different concentrations of aqueous solutions of mineral acids (as proton exchanging agents). The results indicate that the chromium(III) ions can be readily eluted with 0.10 M solutions of HCl or H₂SO₄ (about 96 % chromium recovery for a mass peat: acid volume ratio of 1:10). This fact is in agreement with the pH dependence of the chromium removal onto the peat and confirms the prevalence of the ion exchange mechanism of the sorption. The peat (rinsed with distilled water before reloading) was reused in fives sorption/desorption cycles without a significant loss in sorption capacity.

CONCLUSIONS

The removal of chromium(III) ions from aqueous solutions by sorption onto Romanian *Sphagnum* moss peat was studied as a function of solution pH value, metal ion concentration, peat dose and contact time. The results showed that the highest percentages of Cr(III) removal were attained using peat in the sodium form (4 g L⁻¹) from solutions of pH 5.0 (acetate buffer) with Cr(III) concentrations below 50 mg L⁻¹. The equilibrium data were analyzed against the Freundlich, Langmuir and Dubinin–Radushkevich models. The experimental data were best correlated by the Langmuir isotherm; under optimized conditions, the monolayer adsorption capacity was of 18.6 mg Cr(III) g⁻¹ of peat. Ion exchange was the major mechanism for the adsorption of Cr(III) on the peat particles with the sorption kinetics following a pseudo-second order rate equation. The Cr (III)-loaded peat could be regenerated by treatment with 0.10 M HCl and the sorbent may be reused in several sorption–desorption cycles. The results of this study showed that Romanian *Sphagnum* moss peat, a natural, cheap and available material, could be employed as a sorbent for the removal of chromium(III) from aqueous waste solutions.

ИЗВОД

ИСПИТИВАЊЕ УКЛАЊАЊА ХРОМА(III) ИЗ ВОДЕНИХ РАСТВОРА
СОРПЦИЈОМ НА ТРЕСЕТНОЈ МАХОВИНИ *Sphagnum*

CATALIN BALAN, DOINA BILBA и MATEI MACOVEANU

Department of Environmental Engineering and Management, Faculty of Chemical Engineering and Environmental Protection, Technical University of Iasi, D. Mangeron Blvd. 71A, 700050, Iasi, Romania

Изведена је група сорпционих експеримената за уклањање јона хрома (III) из водених раствора помоћу тресетне маховине *Sphagnum* са подручја Румуније (нетретиране и третиране раствором NaCl) као сорбентом. У циљу успостављања најпогоднијих услова за сорпцију хрома(III), испитивани су утицаји рН, времена контакта, количине маховине и концентрације јона. Примењени су модели Фројндлиха (Freundlich), Ленгмира (Langmuir) и Дубинин–Радушевича (Dubinin–Radushkevich) за опис сорпционих изотерми и за израчунавање одговарајућих константи. Експериментални резултати одговарају Ленгмировом моделу са максималним сорпционим капацитетом од 18,6 mg Cr(III) по граму маховине. Вредност средње енергије сорпције указује на то да се Cr(III) везује за маховину по механизму измене јона. Кинетички подаци, добијени на основу псеудо-првог и псеудо-другог кинетичког модела, показују да процес сорпције хрома на маховини следи закон брзине псеудо-другог реда. Сорбовани хром(III) лако може бити одвојен од маховине испирањем раствором 0,10 М HCl, док се иста маховина може користити у неколико циклуса сорпција/десорпције. Експериментални резултати указују на то да тресетна маховина *Sphagnum* има потенцијал за примену уклањања Cr(III) из отпадних вода.

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