

ADSORPTION OF CADMIUM(II) IONS FROM INDUSTRIAL WASTEWATER BY LOW MOOR PEAT OCCURRING IN THE OVERBURDEN OF BROWN COAL DEPOSITS

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Abstract: The adsorption of cadmium(II) ions by low moor Alder Peat occurring in the overburden of brown coal deposits in Belchatów Brown Coal Mine was investigated under dynamic conditions. Cadmium(II) ions were applied to the column in aqueous solutions containing either cadmium sulfate or cadmium chloride. Solutions were also prepared containing cadmium ions alone or in combination with zinc(II) and copper(II) ions. The peat used as the adsorbent in this study had a high capacity for adsorbing the ions tested. The cadmium adsorbing capacity of the peat was significantly affected by pH, the anions present in the solution, and other cations present in the solution. The cadmium adsorbing capacity of the peat was significantly lower in the presence of other metal cations such as zinc(II) and copper(II), because these cations effectively compete with cadmium ions for binding sites on the peat. Peat can be recommended for purification processes designed to remove cadmium ions. Because cadmium ions are predominantly loosely bound to the peat, they are easily extracted. This means that the cadmium adsorbing capacity of the peat is regenerated so that it can be used in further purification cycles.

INTRODUCTION

The discharge of industrial and municipal wastewater into rivers and streams is one of many factors which affect environmental water quality. In the province of Upper Silesia in southern Poland, 32.1 hm³ of the industrial and municipal water produced each year does not undergo purification. This represents 16% of the total wastewater output of the province. 22% of the untreated wastewater is discharged directly into municipal sewage systems [8].

Many small factories and workshops in which heavy metals are used do not have separate waste treatment facilities. They therefore discharge wastewater directly into municipal sewage systems, which greatly increases the heavy metal content of the sewage sludge so that it can not be used in agriculture. Literature data show that more than 50% of the sludges are inadequate for use in agricultural areas due to their metals content [7].

Cadmium is a toxic heavy metal that is widely used in industrial processes, including electroplating and the production of batteries and paints. Cadmium is also used in the processing of various metals, especially zinc, lead and copper. Another major source

of environmental cadmium contamination is the combustion of coal [13, 18]. Because cadmium is highly mobile, it can contaminate both soil and ground water. In aqueous solution, cadmium generally occurs as mono-atomic divalent ions. Depending on the ionic strength of the solution, the concentration of ligands in the solution, and the stability of the metal-ligand interactions, cadmium may also occur in various complex ions, including:

- hydrates, such as CdOH^+ , $\text{Cd}(\text{OH})_4^{2-}$ and $\text{Cd}(\text{OH})_3^-$;
- chlorides, such as CdCl^+ , CdCl_4^{2-} , and CdCl_3^- ; and
- organic chelates.

The removal of heavy metal ions from industrial wastewater by using various porous adsorbents has generated much interest [2, 6, 10, 12, 20]. Over the last twenty years, interest in using organic adsorbents that are rich in humic substances has particularly increased. Peat, compost and sewage sludge contain abundant exposed carboxyl and hydroxyl phenolic groups that can bind heavy metals [1–3, 11, 17, 19]. They are also low cost and efficient adsorbents. Proper implementation of an effective purification process based on adsorbents requires thorough knowledge of the metal binding properties of the adsorbent. These properties determine whether the adsorbent can be used over the long term in repeated sorption-desorption cycles. Organic adsorbents are especially promising because they have both a high adsorption capacity and a high capacity to form weak bonds. Among the factors affecting adsorption capacity of a particular adsorbent are the contact time, pH, temperature, amount of adsorbent, concentration of solute, properties of solute, and the mode of contact between the adsorbent and the liquid phase.

The aim of this study was to determine the capacity of Alder Peat occurring in the overburden of brown coal deposits in Bełchatów Brown Coal Mine (Central Poland) to adsorb cadmium(II) ions, and how this was affected by pH and cadmium ion concentration. The effect of other cations and anions was also examined. The susceptibility of adsorbed cadmium ions to extraction from the peat was also studied.

MATERIALS AND METHODS

The adsorption of cadmium ions by low moor peat was investigated under dynamic (flow-through) conditions. The liquid phase was brought into contact with the solid phase in a glass column. The column was 42 mm in diameter and 230 mm high. The column was filled with 100 g of peat. The upward flow rate was set at $0.1 \text{ cm}^3 \cdot \text{s}^{-1}$.

The adsorbent used was Alder Peat occurring in the overburden of brown coal deposit in Bełchatów Brown Coal Mine (Central of Poland). Material was air-dried, crushed in mortar, homogenized and sieved through a 1.00 mm mesh sieve. The physical, chemical and sorption properties of the peat are presented in Table 1. Porosity was determined by using mercury porosimeter (Carlo Erba model 2000), the total specific surface area was measured by using BET(H_2O) (Fisons, Sorptomatic 1990), pH was measured in 1:10 solid phase : H_2O or 1 M KCl ratio. Chemical composition and initial heavy metals present in native samples were determined by using the XRF method. Sorption properties were determined by standard soil analysis methods, which are described elsewhere [15]. Major cations occupying exchangeable positions were determined in 1 M NH_4OAc at pH 7, base cation exchange capacity (CEC_0) was obtained by calculation from the exchangeable cations contents. 0.1 M BaCl_2 was used to analyze total cation exchange capacity (CEC_t). Base saturation was calculated as a ratio of CEC_0 and CEC_t .

Table 1. Some physical, chemical and physicochemical properties of investigated peat

Physicochemical properties								
Decomposition rate [%]	Bulk density [g·cm ⁻³]	Specific gravity [g·cm ⁻³]	Porosity [%]	Specific surface area [m ² ·g ⁻¹]	pH (H ₂ O/KCl)	Ash content [%]		
70	0.395	1.493	51.8	218.98	5.62/5.11	20.88		
Mineral constituents [%]								
SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	Mn ₂ O ₄	CaO	MgO	Na ₂ O	K ₂ O
8.26	2.32	0.113	2.31	0.112	4.57	0.33	0.067	0.213
Trace elements content [mg·kg ⁻¹]								
Cr	Cu	Cd	Zn	Ni	Pb			
23.48	22.07	0.63	11.69	15.27	7.19			
Cation exchange capacity								
CEC ₀	CEC _t	Base saturation [%]	Exchangeable ions [cmol(+)·kg ⁻¹] (% of total content)					
[cmol(+)·kg ⁻¹]			Ca ²⁺	Mg ²⁺	Na ⁺	K ⁺	H ⁺	
102.61	118.11	86.88	95.18 (58.31)	7.14 (43.26)	0.20 (9.26)	0.09 (1.92)	15.5	

Cadmium(II) ions were applied to the column in aqueous solutions containing either cadmium sulfate 3CdSO₄·8H₂O or cadmium chloride CdCl₂·2.5H₂O. The concentration of cadmium(II) ions in the solutions was 500 mg·dm⁻³ or 250 mg·dm⁻³. The pH of both solutions was adjusted to 4.00 (with 0.1 M HCl or H₂SO₄ solution) before passing them through the column. Solutions were also prepared containing Cd(II) ions alone or in combination with Zn(II) or Cu(II) ions.

Acid wastewater from a galvanizing plant was also applied to the column. The wastewater had a pH of 1.47 and an Eh of 460 mV. The wastewater contained the following ions: Cd(II) – 171 mg·dm⁻³, Fe(II) – 14985 mg·dm⁻³, Zn(II) – 2870 mg·dm⁻³, Cr(III) – 235 mg·dm⁻³, Mn(II) – 122 mg·dm⁻³, and SO₄²⁻ – 12869 mg·dm⁻³.

The total amount of cadmium adsorbed on the column was calculated according to the following formula:

$$S = \Sigma(C_{in} - C_{out}) \cdot V_o / M$$

where:

- S* – represents the amount of cadmium ions adsorbed [mg·kg⁻¹ of adsorbent],
- C_{in}* – represents the concentration of cadmium ions in the solution entering the column [mg·dm⁻³],
- C_{out}* – represents the concentration of cadmium ions in the solution leaving the column [mg·dm⁻³],
- V_o* – represents the water retention capacity of the column [dm³],
- M* – represents the mass of the adsorbent in the column [kg].

The concentrations of heavy metal ions in the solutions were measured both before and after perfusion through the column with help of an atomic absorption spectrometer (Thermo Jarrell Ash AA-Scan 1).

Sequential extraction was then carried out to determine the nature of the interactions between the cadmium ions and the adsorbent and to determine the susceptibility of the adsorbed cadmium ions to extraction. The extraction was carried out using the method described by Tessiera [21], as modified by Kersten and Forstner [14]. The capacity of the eluent for extracting metal ions was gradually increased during the procedure from step F0 to step F6. This extraction was designed mainly for identification of chemical forms of

metal binding onto heterogeneous mineral matrices with substantial admixture of organic matter. In the study of organic matter this scheme is used to define of metal mobility and potential for remobilization. These steps are:

- F0 – the most labile – metal ions retention in pore solution in organic matter;
- F1 – mobile – electrostatic binding due to an attraction between the negative charged surface of peat and the positively charged metal ions and replacing metal ions with exchangeable cations, mainly Ca^{2+} ;
- F2 – mobile – metal binding in reaction with Ca^{2+} no exchangeable ions;
- F3 – mobilizable – metal binding due to the reaction with manganese oxides;
- F4 – mobilizable – metal binding due to the reaction with iron oxides and hydroxy-oxides;
- F5 – a very strong stable binding of metal ions with humic acids, as a result of incorporation of metal ions into chelate rings formed with adjacent aromatic carboxylic COOH and phenolic hydroksy OH groups or two adjacent COOH groups;
- F6 – residual immobile fraction, metals are bound in the lithogenic crystallites of the mineral fraction; in studied organic matter this step does not occur.

RESULTS AND DISCUSSION

Physical and chemical properties of the adsorbent

The peat used as the adsorbent in this study was highly decomposed (70%). Low mineral fraction shows that peat can be treated as almost pure organic matter. Total ash content of this sample was 21% which was a higher value than in peat samples from undrained area, as well as bulk density and specific gravity. This peat was characterized by high porosity and specific surface area, 51.8% and $219 \text{ m}^2 \cdot \text{g}^{-1}$, respectively. The most abundant component of mineral fraction was SiO_2 (8.26%), CaO (4.57%), Al_2O_3 (2.32) and Fe_2O_3 (2.31%). The content of MgO and K_2O was minor (0.33%, and 0.213% respectively), while Na_2O was present in trace amounts. The total background metal concentrations reflected natural contents of these elements in unpolluted soils.

The total cation exchange capacity (CEC) of the air dried peat was $118.11 \text{ cmol} \cdot \text{kg}^{-1}$ (Tab. 1), which is comparable to values for clay minerals of the smectite group. The capacity of the peat for adsorbing metal ions was determined predominantly by the presence of organic colloids bearing negatively charged groups, especially the carboxyl groups of humic acids. Main exchangeable cations were Ca^{2+} , H^+ and Mg^{2+} , and they occupied 80.58%, 13.12% and 6.05% of exchangeable sites, respectively.

Adsorption of cadmium ions by the peat

The cadmium adsorbing capacity of the peat was determined using a dynamic (flow-through) method. Graphs were prepared depicting the amount of cadmium adsorbed. Graphs were also prepared depicting the ratio of C_{out} , the cadmium concentration of the eluate leaving the column, to C_{in} , the cadmium concentration of the solution entering the column (dimensionless liquid phase concentration). Both variables were plotted against x -axes representing the volume V of solution that had passed through the column.

The graphs depicting the amount of cadmium adsorbed were used to determine the cadmium adsorbing capacity of the peat. The graphs depicting dimensionless liquid phase

concentration were used to delineate the phases of the adsorption process, which are important for the practical application of a particular adsorbent in wastewater treatment. There are two phases in total sorption cycle of heavy metals sorption: I – full sorption, II – breakthrough phase.

In the full adsorption phase, all of the cadmium ions in the solution entering the column were adsorbed, and the concentration of the cadmium ions in the eluate leaving the column was below determination limit of spectrometer.

In the breakthrough phase, only some of the cadmium ions in the solution entering the column were adsorbed, and the concentration of cadmium ions in the eluate leaving the column increased until it approached the concentration in the solution entering the column.

In the equilibrium phase, there was no net adsorption of cadmium ions by the adsorbent, and the concentration of cadmium ions in the eluate leaving the column was equal to the concentration in the solution entering the column.

The water retention capacity (V_0) of the peat used in this study was 180 cm³ for the 100 grams peat in the column. The ratio of the liquid phase to the solid phase was therefore 1.8:1. This is significantly lower than the ratios used in the static batch method, which is commonly used in adsorption studies. However, the total amount of solution which passed through the column before the equilibrium phase was reached and much higher than the water volume in batch method. The peat used in this study had a high capacity to adsorb metal cations. This was greatly affected by what anions were present in the solution (Fig. 1).

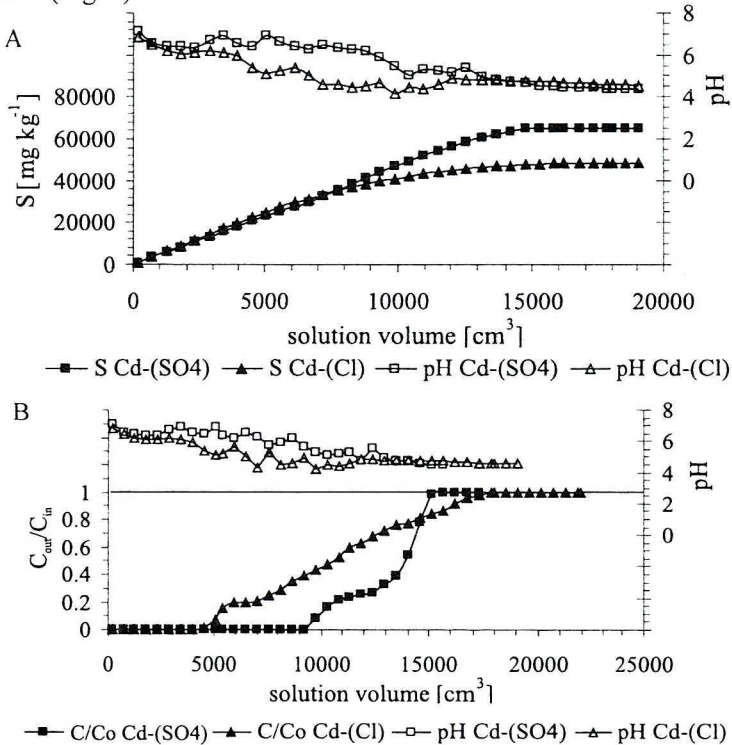


Fig. 1. Effect of sulfate and chloride ions on cadmium adsorption capacity in Alder Peat under dynamic flow conditions: A – total amount of cadmium ions adsorbed, B – dimensionless liquid phase concentration

In the solutions prepared using sulfate salts, the amount of Cd(II) ions retained on the column was $65260 \text{ mg}\cdot\text{kg}^{-1}$ for the solution containing only cadmium ions. And in the solutions prepared using chloride salts, the amount of Cd(II) ions retained on the column was $47300 \text{ mg}\cdot\text{kg}^{-1}$ for the solution containing only cadmium ions.

The amount of cadmium ions adsorbed during the full adsorption phase was about half as much with the chloride solutions as with the sulfate solutions. This was because cadmium has a high capacity for forming stable complexes with chloride ions ($\log K = 1.35$). When cadmium ions are complexed with chloride ions, there is an overall increase in ion size and an overall decrease in the charge density on the ion. The effect of strong complexing ligands on cadmium adsorption had also been reported previously, although the interactions observed were different than in the present study [4]. In that study, chloride and other complexing agents appeared to increase the adsorption of cadmium ions, thereby reducing their susceptibility to being extracted.

The amount of cadmium ions adsorbed was also highly dependent on the total concentration of cadmium ions in the solution entering the column. The total amount of cadmium adsorbed was 26% less with a solution containing $250 \text{ mg Cd}^{2+}\cdot\text{dm}^{-3}$ than with a solution containing $500 \text{ mg Cd}^{2+}\cdot\text{dm}^{-3}$ (Fig. 2).

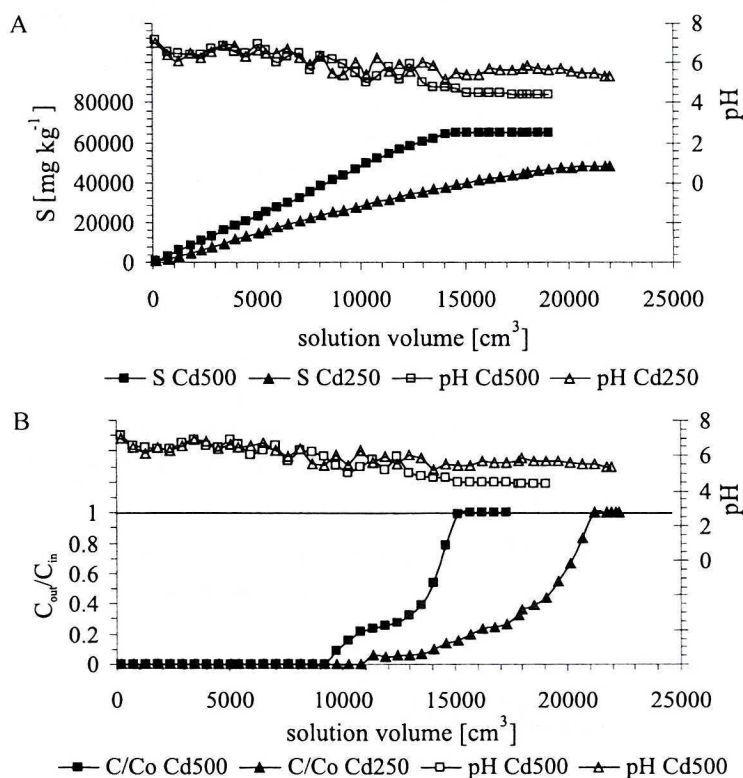


Fig. 2. Effect of cadmium ion concentration in the test solution on cadmium adsorption capacity in Alder Peat under dynamic flow conditions: A – total amount of cadmium ions adsorbed, B – dimensionless liquid phase concentration

In order to determine the degree to which other cations compete with cadmium for binding sites on the adsorbent, solutions containing cadmium ions alone or in combination with Zn(II) and Cu(II) ions were passed through the column. The concentration of each ion was $500 \text{ mg}\cdot\text{dm}^{-3}$. The total amount of cadmium adsorbed was $65260 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing only cadmium sulfate ions, $47750 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and zinc sulfate, and $20660 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and copper sulfate (Fig. 3).

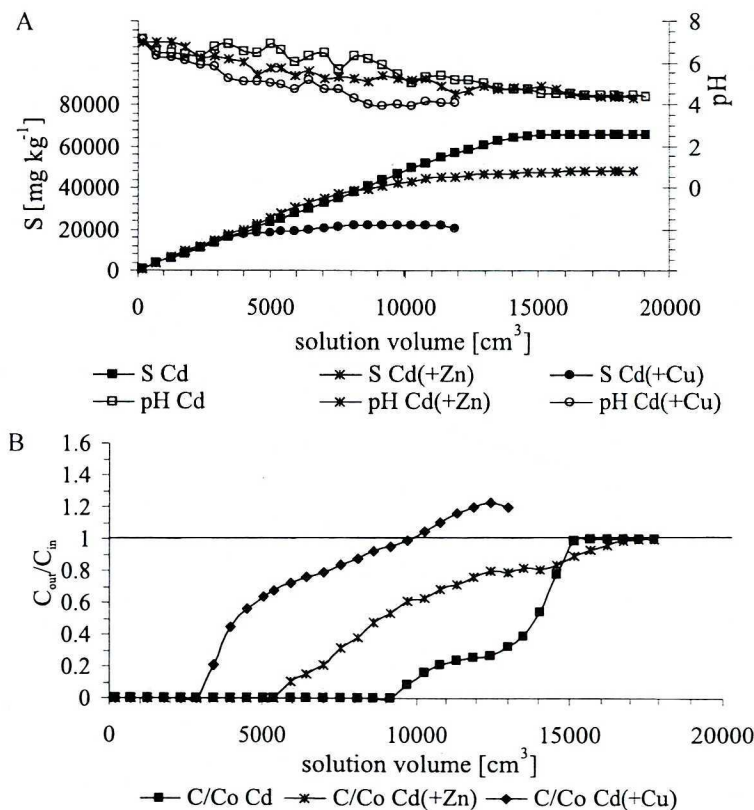


Fig. 3. Effect of zinc(II) and copper(II) ions on cadmium adsorption capacity in Alder Peat with solutions prepared using sulfate salts under dynamic flow conditions: A – total amount of cadmium ions adsorbed, B – dimensionless liquid phase concentration

The total sorption capacity for cations occurring in solution was $116 \text{ cmol}(+)\cdot\text{kg}^{-1}$ with the solution containing only cadmium sulfate ions, $210 \text{ cmol}(+)\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and zinc sulfate, and $154 \text{ cmol}(+)\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and copper sulfate. The differences in total cation exchange capacity were due to differences in the ionic strengths of the solutions.

The amount of cadmium adsorbed during the full adsorption phase was $46780 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing only cadmium sulfate ions, $30325 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and zinc sulfate, and $15710 \text{ mg}\cdot\text{kg}^{-1}$ with the solution containing cadmium sulfate and copper sulfate (Fig. 3A, B).

The proportion of the total amount of cadmium ions adsorbed that were adsorbed during the full adsorption phase was 72% with the solution containing only cadmium sulfate ions, 63.5% with the solution containing cadmium sulfate and zinc sulfate, and 76% with the solution containing cadmium sulfate and copper sulfate.

The pH of the eluate leaving the column fell from 6.43 to 4.60 with the solution containing only cadmium sulfate ions, from 6.51 to 4.40 with the solution containing cadmium sulfate and zinc sulfate, and from 6.32 to 4.03 with the solution containing cadmium sulfate and copper sulfate (Fig. 3A).

With the solution containing cadmium sulfate and copper sulfate, the concentration of cadmium ions in the eluate leaving the column was higher than in the solution entering the column when the pH fell below 4.25 (Fig. 3B). This was due to displacement of the cadmium ions already adsorbed on the column by copper ions.

The same general patterns were observed in solutions prepared using chloride salts (Fig. 4A, B).

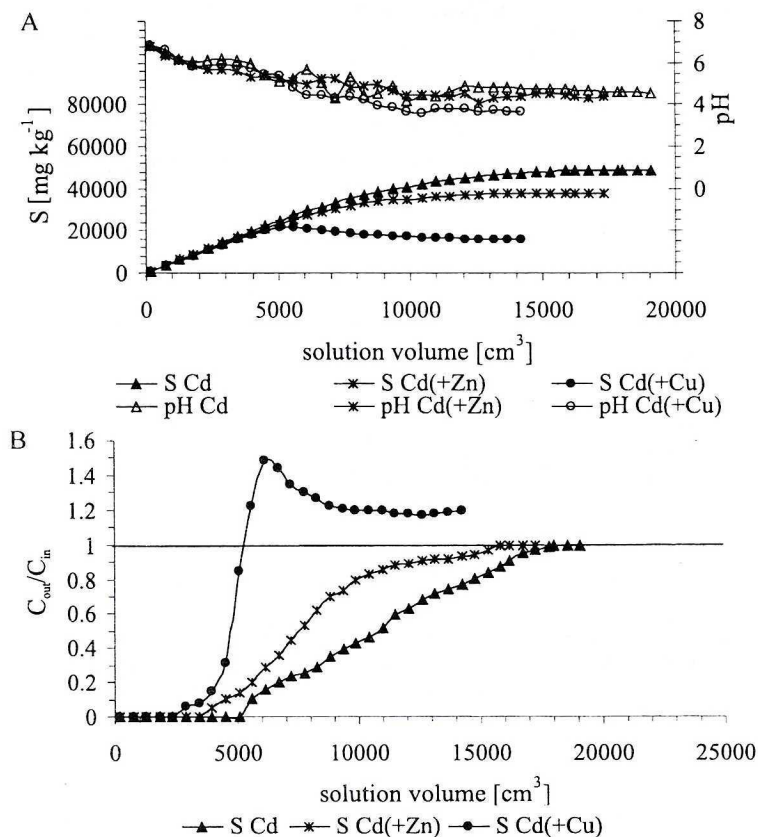


Fig. 4. Effect of zinc(II) and copper(II) ions on cadmium adsorption capacity in Alder Peat with solutions prepared using chloride salts under dynamic flow conditions: A – total amount of cadmium ions adsorbed, B – dimensionless liquid phase concentration

The total amount of cadmium adsorbed was $47300 \text{ mg} \cdot \text{kg}^{-1}$ with the solution containing only cadmium chloride ions, $37520 \text{ mg} \cdot \text{kg}^{-1}$ with the solution containing cadmium

chloride and zinc chloride, and 15680 mg·kg⁻¹ with the solution containing cadmium chloride and copper chloride.

The total sorption capacity for cations occurring in solution was 84.5 cmol(+)·kg⁻¹ with the solution containing only cadmium chloride ions, 202.0 cmol(+)·kg⁻¹ with the solution containing cadmium chloride and zinc chloride, and 149.0 cmol(+)·kg⁻¹ with the solution containing cadmium chloride and copper chloride.

The pH of the eluate leaving the column fell from 6.45 to 4.26 with the solution containing only cadmium chloride ions, from 6.39 to 4.20 with the solution containing cadmium chloride and zinc chloride, and from 6.12 to 4.11 with the solution containing cadmium chloride and copper chloride (Fig. 4A).

With the solution containing cadmium chloride and copper chloride, the concentration of cadmium ions in the eluate leaving the column was higher than in the solution entering the column when the pH fell below 4.27.

With the highly acidic wastewater from the galvanizing plant, the total amount of cadmium ions adsorbed by the adsorbent was only 2080 mg·kg⁻¹, which represents only 3% of the total cation exchange capacity of the peat. As the pH of the eluate leaving the column fell, the cadmium ions were displaced from the adsorbent by oxonium ions (Fig. 5).

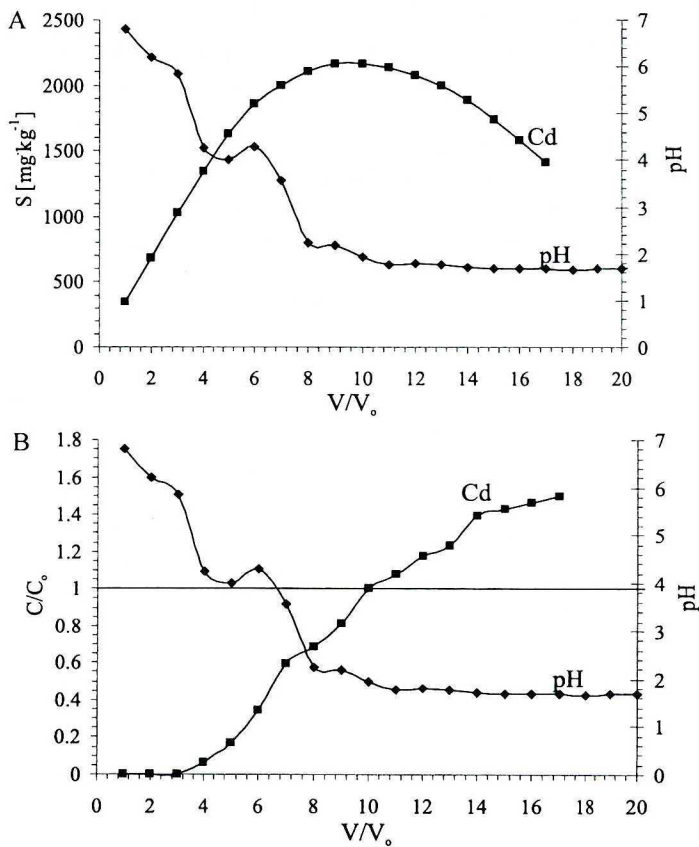


Fig. 5. Cadmium adsorption capacity in Alder Peat with highly acidic wastewater from a galvanizing plant: A – total amount of cadmium ions adsorbed, B – dimensionless liquid phase concentration

The high degree of antagonism between cadmium and zinc ions which was responsible for a significant reduction in the amount of cadmium ions adsorbed had also been observed in a previous study [5]. Displacement of cadmium(II) ions by copper(II) and chromium(III) ions has also been observed with loess, sandy soil, and soil with a high content of organic substances [9, 22].

Susceptibility of adsorbed cadmium ions to extraction

The results of the seven-step sequential extraction procedure showed that the same set of factors which affected the amount of cadmium adsorbed by the peat also affected the susceptibility of adsorbed ions to extraction. These factors included pH, the anions present in the solution, and other metal ions present in the solution.

The concentration of a particular metal ion in the first fractions to be eluted, F0, F1 and F2, represents those ions which were relatively weakly bound to the adsorbent, and consequently more susceptible to extraction. These fractions are particularly important for determining the mobility of a particular metal in soils.

In this study, most of the cadmium ions were eluted in F0, F1 and F2, which indicates that they had been only weakly bound to the peat. Fraction F6 in the studied organogenic material does not occur.

The cadmium ions adsorbed on the column were bound to the peat predominantly by relatively weak interactions, which agree well with previous reports [16]. They were therefore easily displaced by other cations, which agrees well with the results of a study on cadmium desorption in subsurface soils [23].

With the solutions prepared using chloride salts, the cadmium adsorbing capacity of the peat was lower than with the solutions prepared using sulfate salts. Consequently, the proportion of the adsorbed cadmium ions eluted in fractions F0, F1 and F2 was higher than with the solutions prepared using sulfate salts. In other words, the adsorbed cadmium ions were more susceptible to extraction with the solutions prepared using chloride salts than with the solutions prepared using sulfate salts (Fig. 6).

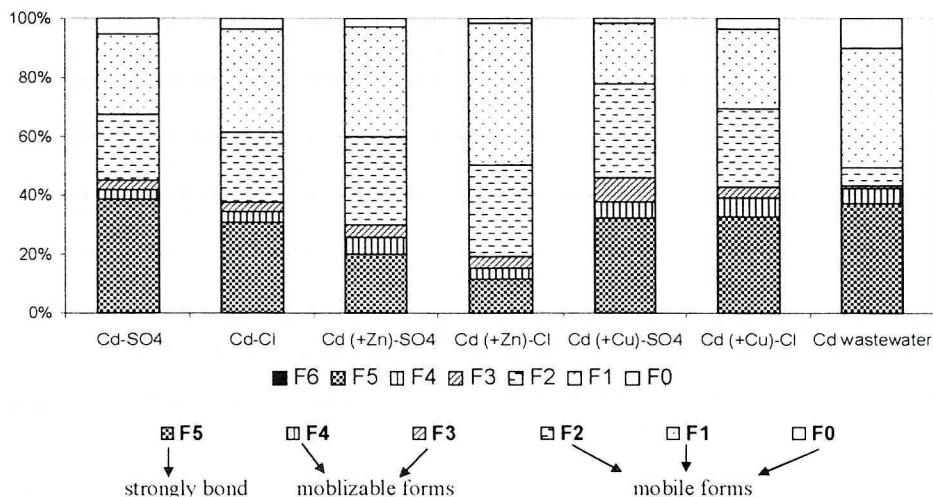


Fig. 6. Distribution of cadmium ions among the fractions collected during sequential extraction; strength of cadmium-peat interactions ranges from very weak in F0 to strong in F5

The proportion of the adsorbed cadmium ions eluted in fractions F0, F1 and F2 was also higher in solutions containing cadmium ions in combination with copper(II) ions. This is because the amount of cadmium ions which were tightly bound to the peat was lower because they were displaced by more competitive smaller ions.

CONCLUSIONS

1. The peat used as the adsorbent in this study had a high capacity for adsorbing the ions tested.
2. The cadmium adsorbing capacity of the peat was significantly affected by pH, the anions present in the solution and other cations present in the solution.
3. The cadmium adsorbing capacity of the peat was significantly lower at lower pH because of displacement by oxonium ions.
4. The amount of cadmium adsorbed by the peat was lower with solutions prepared using chloride salts than with solutions prepared using sulfate salts. This was because cadmium has a high capacity for forming stable complexes with chloride ions.
5. The cadmium adsorbing capacity of the peat was significantly lower in the presence of other metal cations such as zinc(II) and copper(II) because these cations effectively compete with the cadmium ions for binding sites on the peat.
6. With the highly acidic wastewater from the galvanizing plant, the cadmium adsorbing capacity of the peat was greatly reduced because of competition by other cations in the solution, including zinc(II), copper(II) and oxonium ions, which prevented the cadmium ions from forming strong interactions with the peat.
7. Peat is a good adsorbent for use in purification processes designed to remove cadmium ions. Because the cadmium ions are predominantly loosely bound to the peat, they are easily extracted. This means that the cadmium adsorbing capacity of the peat is regenerated so that it can be used in further purification cycles.

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WIĄZANIE JONÓW Cd(II) ZE ŚCIEKÓW PRZEMYSŁOWYCH PRZEZ TORFY WYSTĘPUJĄCE W NADKŁADZIE ZŁÓŻ WĘGLI BRUNATNYCH

Wyznaczono pojemność sorpcyjną dla torfu olesowego występującego w nadkładach złoża węgla brunatnego w Bełchatowie w stosunku do jonów Cd(II). Badania przeprowadzono w dynamicznych warunkach kontaktu faza stała – roztwór. W badaniach wykorzystano zarówno wzorcowe roztwory chlorków i siarczanów jak i kwaśne ścieki polimetaliczne z zakładu galwanizerskiego. Ponadto określono podatność na ługowanie zasorbowanych jonów Cd(II). Stwierdzono znaczny wpływ pH, rodzaju anionu i kationów współwystępujących na ilość wiązanych jonów Cd(II). Wiązanie jonów Cd(II) na pozycjach wymiennych kompleksu sorpcyjnego wpływa na ich wysoką podatność do uwalniania i pozwala na kilkukrotne użycie sorbentu w cyklu sorpcja – desorpcja. Przeprowadzone badania pokazują, że torfy występujące w nadkładzie złóż węgla brunatnych charakteryzują się wysoką skutecznością usuwania jonów Cd(II) z roztworów, co pozwala na wykorzystanie ich jako efektywnych sorbentów w oczyszczaniu wód i ścieków.