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The use of organic waste for removing Pb^{2+} ions from aqueous solutions

Introduction

Nowadays the aqueous environment is highly exposed to considerable quantities of anthropogenic pollutants. These include agricultural effluents, industrial chemicals, pollutants discharged by households, etc. A particularly dangerous group of substances that reach natural waters are heavy metals such as lead, cadmium, mercury, etc. Most often these pollutants reach natural waters along with effluents from the metallurgical, chemical and electronic industries, leachates from landfills and rainwater (Toksykologia 1999; Uluozlu et al. 2008; Świderska-Bróż 1980).

Heavy metals can cumulate in living organisms and thus cause permanent damage to cells, leading to various disorders and diseases or even death (Toksykologia 1999). All over the world an important issue of public health is the toxicity of lead for the environment (Ahamed, Siddiqui 2007). This element is listed by the Agency for Toxic Substances and Disease Registry among the twenty [as the second] most toxic metals (Ravi Raja et al. 2008). Once it enters an organism, lead penetrates the blood system, where 99% of its total amount is built into red blood cells (Toksykologia 1999; O'Neill 1997). Subsequently, carried by the bloodstream, lead enters all the organs. Its toxic effect manifests itself in disorders of the haematopoietic system, heme synthesis, inhibition of the haemoglobin synthesis and shortening of the erythrocyte life span (Toksykologia 1999). In advanced stages, anaemia develops. Lead has a toxic effect also on other systems, e.g., it damages the nervous and

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immune systems, causes kidney and alimentary system disorders (Toksykologia 1999; Ravi Raja et al. 2008).

Because of such a serious risk to human health, there has been an increased interest in the presence of heavy metals in drinking water in recent years. The monitoring of these pollutants in natural waters and their efficient elimination have become a challenge for all of us.

Among classical methods of elimination of heavy metals are chemical precipitation, reverse osmosis, adsorption and ionic exchange (Uluozlu et al. 2008; Kyzioł-Komosińska et al. 2008; Sanak-Rydlewska 2007; Al-Qodah 2006). These methods have many advantages, yet they often turn out to be low effective and too costly. This is one of the reasons why a great interest in alternative, biosorption-based methods has been noted.

The essence of biosorption is that properties of organic matter are employed to bind and concentrate heavy metals from their solutions (Sanak-Rydlewska 2007; Qi, Aldrich 2008; Han et al. 2005). Various materials can act as biosorbents: this includes organic waste such as straw, fruit and vegetable peelings, nut shells, fruit stones, straw and bark. Also efficient as sorbents are algae, seaweeds and mushrooms. Many interesting studies are reported in the literature, where natural sorbents are used to eliminate heavy metals from water and effluents (Uluozlu et al. 2008; Sanak-Rydlewska 2007; Al-Qodah 2006; Qi, Aldrich 2008; Han et al. 2005; Li et al. 2007; Zhu, Wei 2006; Pino et al. 2006; Meunier et al. 2002; Low et al. 2000; Nadeema et al. 2008). Listed below are some examples:

— biosorption of heavy metals on powder obtained from green coconuts (Pino et al. 2006),
— the use of natural sorbents, such as powdered cacao bean shells and powdered cedar tree bark to eliminate toxic metals (Meunier et al. 2002),
— adsorption of heavy metal ions (Pb^{2+}, Cu^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+}) on waste tobacco powder (Qi, Aldrich 2008),
— elimination of Pb^{2+} and Cd^{2+} by means of brewery spent grain (Low et al. 2000).

Most of the studies conducted so far have confirmed that the efficiency of natural sorbents mainly depends on such factors as the solution's pH and ionic strength, the amount and particle size distribution of the sorbent, the time of contact between the sorbent and the solution, and the initial concentration of the ions to be adsorbed (Uluozlu et al. 2008; Al-Qodah 2006; Han et al. 2005; Pino et al. 2006; Low et al. 2000; Nadeema et al. 2008).

By ensuring optimal conditions for biosorption, it is possible attain a high degree of removal of heavy metals from the solution. For instance, for powdered cacao bean shells the maximum reduction in Cd^{2+} was about 98% and about 89% for Cr^{3+} under the same conditions (Pino et al. 2006). In the case of cacao bean shells and cedar tree bark, the maximum degree of Pb^{2+} removal from a model solution under the same conditions was about 88% and 59%, respectively (Meunier et al. 2002).

To increase the natural sorption properties of biomass, its surface can be modified by chemical or physical methods. An example is a study conducted on distillery residue (Nadeema et al. 2008). The biomass used in the study was subjected to thermal treatment (heated in a furnace and boiled in water), pressure treatment and chemical treatment

(the action of such chemicals as HCl, H_2SO_4, HNO_3, NaOH). The results obtained confirmed that both physical and chemical modification of the sorbent's surface increases its sorption capacity. The results also demonstrated that chemical treatment is more effective.

The process of boiling and pressure treatment removes mineral components from the sorbent, thus opening the adsorption centres on the surface. The heating in the furnace decomposes organic matter and thus improves the access of ions to the biosorbent active sites. Acids increase the sorption capacity by increasing the biomass active surface area and porosity, whereas treatment with alkalis can, for instance, damage lipids and proteins, which mask the biosorbent active sites (Nadeema et al. 2008).

Natural sorbents are also a raw material feedstock for obtaining activated charcoal used for removal of various pollutants from aqueous solutions. Activated carbon can be obtained by physical or chemical activation (Banat et al. 2003; Juang et al. 2000; Girgis et al. 2002; Suárez-Garcia et al. 2002; Lafi 2001; Williams, Reed 2006). Thermal activation involves pre-carbonisation (at temperatures of up to 700°C) and the subsequent gasification of the starting material with an oxidising gas at high temperatures (up to 1100°C) (Banat et al. 2003, Girgis et al. 2002; Williams, Reed 2006). In chemical methods, strongly dehydrating chemicals (e.g.,:H_3PO_4, $ZnCl_2$) (Girgis et al. 2002; Suárez-Garcia et al. 2002; Williams, Reed 2006) are used for the production of activated carbon. The process of both chemical and chemical activation is conducted in special reactors at high temperatures.

Methods employing natural waste are very promising (Sanak-Rydlewska 2007; Zhu, Wei 2006). They allow the recovery of heavy metals and re-use of the biosorbent in the ion exchange process. These methods are relatively inexpensive and allow the use of waste produced by the fermentation industry, whose disposal generates serious problems. An advantage of these methods is also that the biomass used undergoes biodegradation.

1. Goal of the study

The main goal of the study was to demonstrate that plum stones can bind Pb^{2+} from aqueous solutions. This fact was indicated by preliminary studies conducted, e.g., in paper (Sanak-Rydlewska 2007). An advantage of using organic waste is the possibility to re-use them repeatedly, a high degree of ion removal from the solution and their natural bio-degradation in the environment.

2. Materials and methods of the study

2.1. Preparation of biosorbent

Plum stones were used for the study as natural sorbent. Two particle size classes were used 0.50–1.25 mm; 1.25–2.50 mm, as well as whole stones. The procedure used to prepare

an appropriate class of the biosorbent consisted of several stages. First, dried stones were crushed and the kernels were separated from the dry shells. The crushed shells were milled and passed through appropriate sieves. Before the sorption, the stones were treated with 0.001 mol/L hydrochloric acid to remove surface impurities, and then washed with reverse osmosis water until the pH attained that of pure reverse osmosis water, and next dried at temperatures of up to 323 K (50°C).

2.2. Reagents

The lead(II) solutions used for the study were obtained by dissolving appropriate amounts of lead(II) nitrate(V) (POCh) in nitric acid(V). The pH of thus prepared lead(II) solutions ranged from 2.0 to 5.0. The ionic strength was controlled with a 0.04 mol/L solution of potassium nitrate(V) (POCh). Standard solutions for the calibration curve were prepared from a certified standard with a lead(II) concentration of 1000 mg/L (WChA). The samples were prepared using hydrochloric acid (37%) manufactured by Merck. All solutions were prepared using reverse osmosis water with a conductivity of 0.01 μS.

2.3. Methods used

The lead(II) content of the solutions after adsorption was determined by flow-through coulometry using EcaFlow 150 GLP apparatus manufactured by POL-EKO. Measurements were performed in an EcaCell 353c cell. In the three-electrode system employed, the working electrode was a carbon electrode type E56 LMF. Elemental lead was deposited on the working electrode at an accumulation potential of 1400 mV. Six measurements were performed for each sample. The results presented in this paper are thus the arithmetic means of six measurements; the plot also shows measurement uncertainty, which is represented by error bars.

2.4. Adsorption process

Adsorption of Pb^{2+} was performed from 100 mL samples of the solution in 250 mL beakers. The pH of the studied solutions ranged from 2.0 to 5.0. The amount of the biosorbent varied from 1.0 to 20.0 g. The content of the beakers was continuously stirred with a mechanical stirrer at 120 rpm at 297±1K (24±1°C). The experiments lasted for 1 hour. The study was performed for the Pb^{2+} concentration ranging from 1 to 175 mg/L. Each solution's pH was measured before and after the experiment. After the study, the stones were rinsed with reverse osmosis water.

2.5. Analytical procedure

After adsorption, the solutions were passed through a filter paper to remove solid particles. Samples for coulometric analysis were prepared in 50 mL calibrated flasks. To each

flask 0.4 mL of concentrated hydrochloric acid was added and an appropriate amount of the sample (so that the expected concentration should be within the range of the calibration curve) and next the flasks were filled up to the mark with the osmosis water. A blank sample was prepared in the same way as the samples analysed, except that osmosis water was used-added instead of the analyte.

3. Discussion of the results

The study conducted, whose aim was to take advantage of the sorptive properties of plum stones to remove Pb^{2+} ions from aqueous solutions, was arranged in several stages. In the first stage optimal conditions for adsorption were selected. The optimisation procedure involved studying the effect of the amount of biosorbent used, its particle size and the pH of the solution on Pb^{2+} adsorption. Subsequently, the sorption mechanism was determined (adsorption was described by the Freundlich and Langmuir equations).

The amount of lead(II) ions, m_a, adsorbed on the biosorbent's surface was calculated from this formula:

$$m_a = A \cdot m = V(c_0 - c_k) \tag{1}$$

where:

A – is the sorptive capacity (mg of the substance adsorbed per gram of the adsorbent,

V – is the volume of the solution in the flask (L);

c_o and c_k – are the initial and final concentrations of lead(II) ions (mg/L);

m – is the quantity of dry mass of the adsorbent (g).

3.1. The effect of biosorbent on the adsorption of Pb^{2+} ions

The study of the dependence of the degree of removal of lead(II) ions on the amount of plum stones used was performed for 1, 2, 5, 10, and 20 g of the biosorbent. The initial concentration of lead in the solutions studied was 25 mg/L. The pH of the solutions prepared for analysis was 4.0. Stones in the size range of 1.25–2.50 mm were used in the experiments. The results are shown in Fig. 1.

Analysis of the dependence obtained shows that the degree of removal of lead(II) ions increases with the amount of stones used for adsorption. The least removal of 82.3% occurred for 1 g of stones, whereas the highest one, 99.8%, occurred for 20 g of the sorbent at the same volume of the initial solution (100 mL) and the same concentration of the initial solution in contact with the adsorbent (25.0 mg/L). It was also deduced from this plot that the optimal amount of stones was 10 g. Conducting the sorption with 10 g of biosorbent leads to a relatively high removal, reaching 98.8% and saves the sorbent, whose preparation is
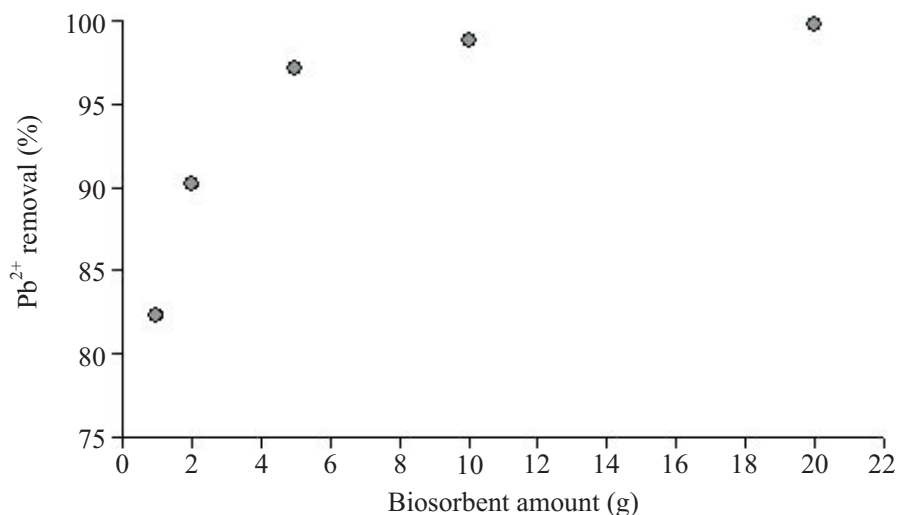
Fig. 1. The effect of the amount of biosorbent on Pb^{2+} removal from the solution (initial Pb^{2+} concentration 25.0 mg/L; pH of 4.0; ionic strength 0.02 mol/L; sorbent's particle size class 1.25–2.50 mm; T = 298 K, time of adsorption 1 h; mixing rate 120 rpm)

Rys. 1. Wpływ ilości biosorbentu na redukcję jonów Pb^{2+} z roztworu (początkowe stężenie metalu 25,0 mg/dm^3; pH 4,0; siła jonowa 0,02 mol/dm^3; klasa ziarnowa sorbentu 1,25–2,50 mm; T = 298 K, czas adsorpcji 1 h; szybkość mieszania 120 obr./min)

time-consuming. With the use of 20 g of the biosorbent, the removal of lead(II) ions is only by 1 percent lower whereas the consumption of the biosorbent is two times higher.

3.2. The effect of pH and particle size on Pb^{2+} adsorption in the biosorbent studied

Another factor which plays an important role in the adsorption of heavy metals is the solution's pH. As mentioned earlier, the optimal pH was selected from among three values: 2.0; 3.0; 4.0 and 5.0. Also studied was the effect of the stone particle size on the Pb^{2+} sorption. The experiments were conducted for two particle size classes 0.50–1.25 mm; 1.25–2.50 mm and for whole stones. The results are shown in Fig. 2.

In the pH range studied, small changes in the removal of Pb^{2+} were noted for sorbents differing in particle size. In very acidic solutions, for pH of 2.0, these differences were the greatest. It turned out, that in such conditions the finest particle size 0.50–1.25 mm produced worse results than the particle size 1.25–2.50 mm. For pH 2.0 the removal was also the poorest as compared to other conditions. With increasing pH of the solution, the concentration of Pb^{2+} increases. The smallest (insignificant) differences in removal for various particle size classes and the highest removal degree were noted for pH 4.0. A further increase in the solution's alkalinity worsened the results.
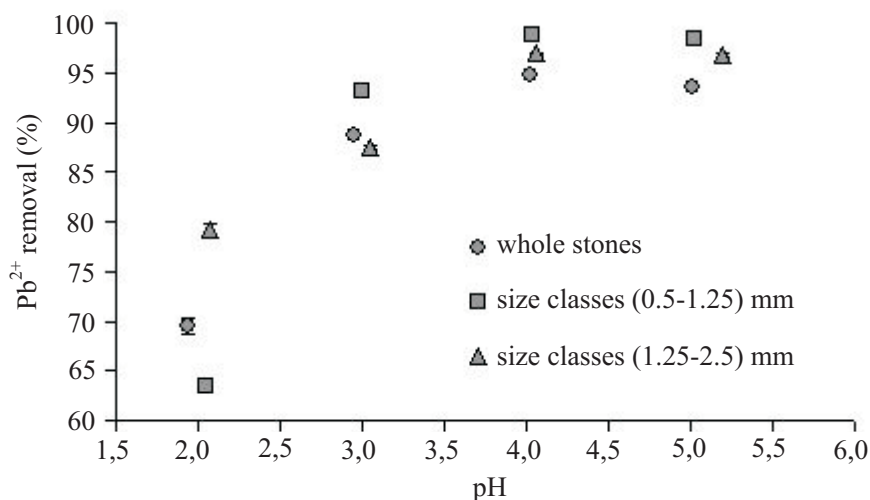
Fig. 2. The effect of pH and particle size on the removal of Pb^{2+} on plum stones (initial Pb^{2+} concentration of 25.0 mg/L; the amount of sorbent 10 g; the ionic strength 0.02 mol/L; T = 298 K, adsorption time 1 h; mixing rate 120 rpm)

Rys. 2. Wpływ pH i uziarnienia na redukcję jonów Pb^{2+} na pestkach śliwek (początkowe stężenie metalu 25,0 mg/dm^3; ilość biosorbentu 10 g; siła jonowa 0,02 mol/dm^3; T = 298 K, czas adsorpcji 1 h; szybkość mieszania 120 obr./min)

Studies conducted recently on natural sorbents turn out to be helpful in explaining the results obtained in this study. As reported in the literature, various functional groups are present on the plum stone surface, e.g., carboxyl and hydroxyl groups (Sanak-Rydlewska 2007; Pino et al. 2006; Wachowski et al. 2007). Most important for sorption of heavy metal ions are weak acidic groups (mainly carboxyl) (Sanak-Rydlewska 2007; Li et al. 2007). In more acidic solutions (at pH 2.0) some of surface groups are probably strongly associated with hydronium ions (H_3O^+), which creates a barrier exerting a repulsive force, which hinders the access of metal ions to the sorbent's surface (Pino et al. 2006). It is also known that in strongly acidic solution, carboxyl groups are not dissociated, which additionally hinders the binding of metal ions from the solution.

The pH of the solution from which the ions are adsorbed also affect the form of lead present in the solution. At low pH only Pb^{2+}ions are present. Gradual increase in the solution's alkalinity initiates hydrolysis and contributes to the formation of $Pb(OH)^+$ ions and ultimately a deposit of $Pb(OH)_2$. As calculated, for the studied Pb^{2+} concentration of 25 mg/L, lead hydroxide should deposit at a pH as high as bout 6.0, but it cannot be excluded that intermediate forms of lead are formed in the solution at pH 5.0, which may decrease their removal.

As at pH 4.0 sorbents of various particle size do not show considerable differences in their ability to remove Pb^{2+} ions, a 1.25–2.50 mm particle size class was selected for further study. This particle size class ensures an appropriate removal degree (96.9%) and shortens the biomass preparation time.

3.3. Isotherm of Pb²⁺ adsorption on plum stones

To describe the adsorption of lead(II) ions on the studied biosorbent, the two most common adsorption models were used: the Langmuir and the Freundlich isotherms. Their characteristics are shown in Table 1.

TABLE 1

Characteristics of adsorption isotherms (Al-Qodah 2006; Atkins 2007; Ignatowicz 2008)

TABELA 1

Charakterystyka modeli izoterm adsorpcji (Al-Qodah 2006; Atkins 2007; Ignatowicz 2008)

Isotherm	Langmuir	Freundlich
Assumptions	Monolayer adsorption on a uniform surface	Adsorption on a non-uniform surface; empirical
Equation	$A = \dfrac{a \cdot c_k}{(1 + k \cdot c_k)}$	$A = a \cdot c_k^k$
where:	A – adsorption (milligrams of adsorbed substance per 1 g of adsorbate); c_k – final concentration of Pb²⁺ ions (mg/L); a and k – adsorption coefficients	

In this part of the study, experiments were conducted at an optimal pH of 4.0, using 10 g of stones of the particle size class 1.25–2.50 mm. The concentration of lead(II) ions in the prepared solutions ranged from 1 to 175 mg/L. The results were described by the Langmuir equation (Fig. 3) and the Freundlich equation (Fig. 4). The fit of the isotherms to the to the results of lead(II) adsorption on the studied sorbent is compared in Fig. 5.

The a and k coefficients of the Langmuir equation were determined from the linear form of the isotherm:

$$\frac{1}{A} = \frac{1}{a} \cdot \left(\frac{1}{c_k} + k \right) \tag{2}$$

In order to calculate the constants a and k from the Freundlich equation, the logarithmic form of the equation was used:

$$\log A = \log a + k \cdot \log c_k \tag{3}$$

All the parameters and their uncertainties were calculated using EXCEL software. The isotherm coefficients obtained, their errors and correlation coefficients R are given in Table 2.

A better function fit to the measurement data was obtained for the Freundlich isotherm, where the correlation coefficient was 0.975. The Langmuir model provided a worse description of the adsorption of the studied ions on the natural sorbent used. The results show
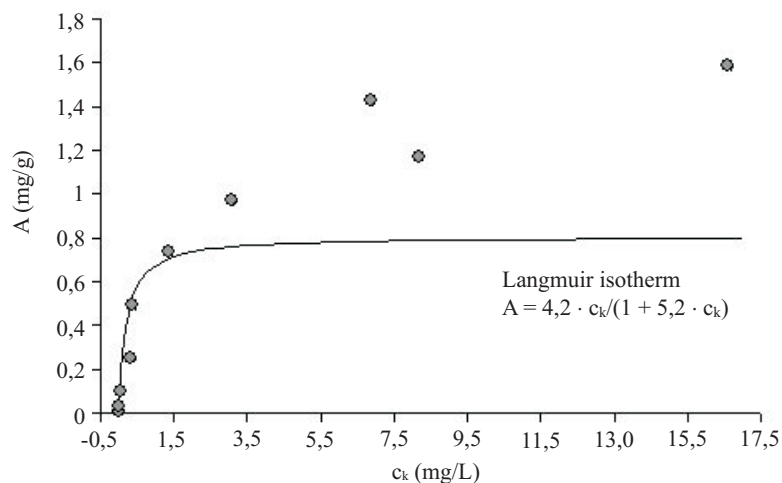
Fig. 3. Langmuir isotherm for Pb^{2+} adsorption on plum stones (pH 4.0; the amount of sorbent 10 g; biosorbent particle size class 1.2–2.50 mm; ionic strength 0.02 mol/L; T = 298 K, adsorption time 1 h; mixing rate 120 rpm)

Rys. 3. Izoterma Langmuira jonów Pb^{2+} na pestkach śliwek (pH 4,0; ilość biosorbentu 10 g; klasa ziarnowa sorbentu 1,25–2,50 mm; siła jonowa 0,02 mol/dm^3; T = 298 K, czas adsorpcji 1 h; szybkość mieszania 120 obr./min)
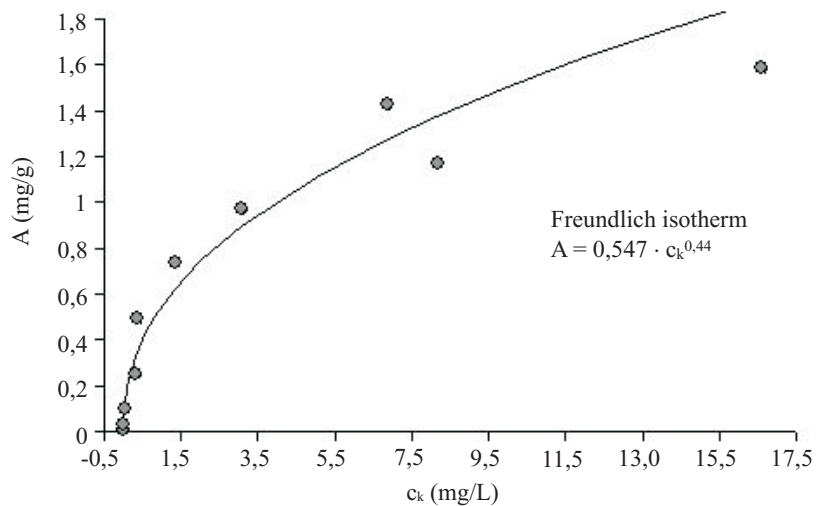

Fig. 4. The Freundlich isotherm for Pb^{2+} adsorption on plum stones (pH 4.0; the amount of sorbent 10 g; biosorbent particle size class 1.25–2.50 mm; ionic strength 0.02 mol/L; T = 298 K, adsorption time 1 h; mixing rate 120 rpm)

Rys. 4. Izoterma Freundlicha jonów Pb^{2+} na pestkach śliwek (pH 4,0; ilość biosorbentu 10 g; klasa ziarnowa sorbentu 1,25–2,50 mm; siła jonowa 0,02 mol/dm^3; T = 298 K, czas adsorpcji 1 h; szybkość mieszania 120 obr./min)
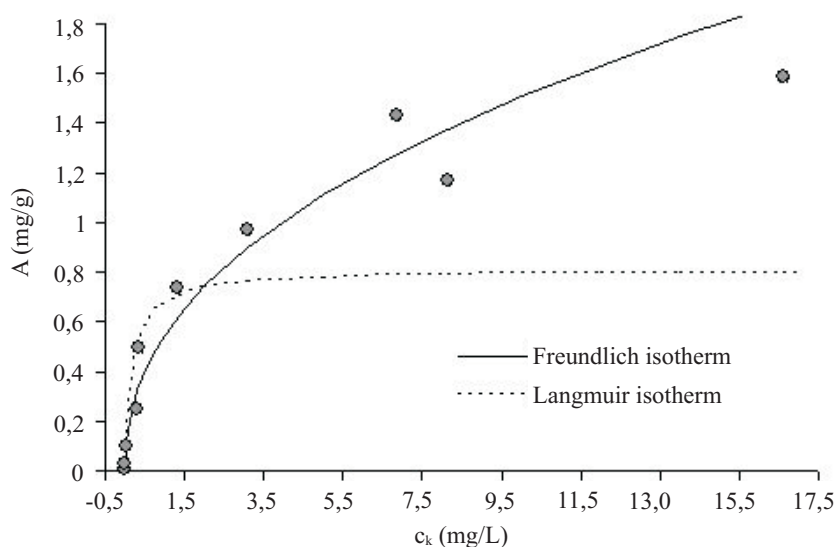
Adsorption isotherm coefficients and their uncertainties

Współczynniki izoterm adsorpcji i ich niepewności

Isotherm	a	Δa	k	Δk	R
Freundlich	0.547	0.05	0.44	0.04	0.975
Langmuir	4.2	0.5	5.2	1.5	0.964

Fig. 5. Comparison of adsorption isotherms fits for Pb^{2+} adsorption on plum stones

Rys. 5. Porównanie dopasowania izoterm procesu adsorpcji jonów Pb^{2+} na pestkach śliwek

that the plum stone surface is not non-uniform (adsorption centres on the surface are of different strength). In the course of adsorption, the active centres with the highest adsorption energy are probably covered first. It is only in the subsequent stages that centres of increasingly lower energy are covered. The energetic nonuniformity of the stone surface has also been confirmed by studies conducted in other research units. Those results show that there are various functional groups on the plum stone surface, e.g., carboxyl, phenol, carbonyl, and hydroxide groups, which form bonds of various energy with metal ions (Li et al. 2007; Wachowski et al. 2007). The data given in Table 3 (Wachowski et al. 2007) indicate the presence of these functional groups.

Based on the found parameters of the Freundlich equation (a and k) – Table 2, it is possible to assess the sorption capacity of plum stones. Presumably it will be possible to obtain better results after activation of the surface with physical or chemical methods.

TABLE 3

XPS analysis of the plum stone surface (Wachowski et al. 2007)

TABELA 3

Analiza powierzchni pestek metodą XPS (Wachowski et al. 2007)

Bonding	C–C, C–H	C=OH	C=O	O=C–O
Relative intensity of peaks [%]	46.3	9.8	11.9	5.9

Summary

1. Plum stones can be used for effective adsorption of lead(II) ions from solutions. The sorption efficiency ranges from 60% to 99%.
2. As part of the study also optimal conditions were identified for the adsorption process (pH 4.0; the amount of biosorbent of 10 g and the particle size class of the plum stones (1.25–2.50 mm).
3. The process of lead(II) adsorption on the biosorbent studied was described by the Langmuir and Freundlich models. A better fit of the function to the measurement data was obtained for the Freundlich isotherm ($R = 0.975$). This testifies to the energetic nonuniformity of the stone surface (e.g., diverse chemical composition, the so-called surface groups).
4. Based on the results, the biosorbent's sorptive capacity was determined. This results are, unfortunately, not satisfactory, which demonstrates that the surface must be activated using chemical or physical methods.

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THE USE OF ORGANIC WASTE FOR REMOVING Pb^{2+} IONS FROM AQUEOUS SOLUTIONS

Key words

Wastes, heavy metals, biosorption

Abstract

This article presents the use of organic waste substances for the sorption of toxic metal ions from aqueous solutions.

Experiments were conducted concerning the sorption of Pb^{2+} ions on plum stones to study the effect of various factors, such as the pH, the quantity and particle size distribution of the natural sorbent, and the concentration of ions undergoing adsorption in the solution to be purified.

Optimal conditions for adsorption were selected (a pH of 4.0, an amount of biosorbent of 10 g and the particle size class of plum stones (1.25–2.50 mm). Pb^{2+} ions were shown to undergo chemisorption with an efficiency of 60 to 99%. The process of Pb^{2+} ion sorption on the natural sorbent studied was described by the Langmuir and Freundlich model.

WYKORZYSTANIE ODPADÓW ORGANICZNYCH DO USUWANIA JONÓW Pb^{2+} Z ROZTWORÓW WODNYCH

Słowa kluczowe

Odpady, metale toksyczne, biosorpcja

Streszczenie

W artykule zaprezentowano zastosowanie odpadowych substancji pochodzenia organicznego do sorpcji jonów metali toksycznych z roztworów wodnych.

Wykonano doświadczenia dotyczące sorpcji jonów Pb^{2+} na pestkach śliwek w zależności od szeregu czynników takich jak: wartość pH środowiska, ilość i klasa ziarnowa sorbentu naturalnego oraz stężenie adsorbowanych jonów w roztworze poddawanym oczyszczaniu.

Dobrano optymalne warunki przeprowadzania procesu adsorpcji (pH środowiska 4,0; ilość biosorbentu 10 g oraz klasa ziarnowa pestek (1,25–2,50) mm). Wykazano, że jony Pb^{2+} sorbują się chemicznie w wydajnością od 60 do 99%. Proces adsorpcji jonów Pb^{2+} na badanym sorbencie naturalnym opisano za pomocą modelu Langmuira i Freundlicha.