Original Research

Adsorptive Removal of Pb²⁺ Ions from Aqueous Solutions by Peat

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Abstract

Peat has been evaluated as an efficient sorbent. In this paper, peat for removal of Pb^{2+} ions from aqueous solution was studied. Peat samples were analyzed for total lead, pH, humidity, and TC (total carbon). Major factors involved, including initial Pb concentration, contact time, and pH, also were investigated. The aqueous solution of lead(II) (0.25 mg/L, 25.0 mg/L, 50.0 mg/L, 75.0 mg/L, and 100.0 mg/L) was agitated at a fixed speed of 20 rpm for 5, 10, 20, 45, 90, 180, and 360 minutes, respectively. At the end of each contact time, the solid was removed by filtration through a 0.45 μm glass filter and then analyzed with an atomic absorption spectrophotometer. Adsorption capacity of peat increases with increasing contact time, initial lead(II) concentration, and was significantly affected by initial pH value in the range from 4.0-7.0. The amount of Pb adsorbed on peat was greatest at pH 6.0, 360 min contact time, and 100 mg/L lead(II) concentration, i.e. 9.489 mg/g. The Freundlich and Langmuir models were used to interpret the sorption behaviour of Pb^{2+} ions, and the results showed that the Freundlich model shows a better fitting than the Langmuir model according to correlation coefficients ($R^2 > 0.98$, Freundlich model, $R^2 > 0.58$ Langmuir model).

Introduction

The increasing industrial activities and development of social agglomerations brings many negative impacts on the environment. Liquid and solid wastes have become the main problem in recent years. Storage and manipulation with this heterogeneous matter entails many complications focused on entry of a wide range of contaminants into soil or water recipients. Organic pollutants can decompose, but inorganic pollutants will be subsequently redistributed in different parts of the environment [1]. Metals are considered to be among the most dangerous environmental pollutants, because they do not disintegrate with physical processes and therefore remain for a long time period [2]. Solubility and availability are the crucial factors that determine their potential environmental risk [1].

Elevated environmental levels of lead ions come from a variety of sources. Lead (Pb) is a metal that occurs naturally in the Earth's crust and is used in industrial manufacturing. Lead can be released directly to water from human activities like production of pulp and paper, light industry, and processing of metals, and from wastewater treatment plants. Lead is a metal ion toxic to the human biosystem, and is among the common global pollutants arising from increasing industrialization. The assimilation of relatively small amounts of lead over a long period of time in the human body can lead to malfunctioning organs and chronic toxicity. The toxic effects of lead ions on humans, when present above the threshold level in the hydrosphere, are

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known [3]. Precipitation and adsorption are two of the common methods for the treatment of heavy metal-containing water [4]. Previously, various researchers have studied the adsorption of lead ions on activated carbon [3, 5-8], fruit shells [4], zeolite [8-10], silica [10], phosphogypsum [11], chitosan [12-14], orange bark [15], etc. The identification of effective filtering materials is imperative for cleaning wastewater before its discharge [16]. The present work we investigates the sorption properties of peat for lead as a model of toxic metal. The aim of this research was to evaluate the adsorption behaviour of Pb^{2+} onto sphagnum moss peat (Lithuania). The influence of experimental conditions-contact time, pH solution, and initial Pb^{2+} concentration on the adsorption behaviour was investigated.

Materials and Methods

A commercially available sphagnum peat moss (Lithuania) was used as the sorbent. The sorbent was not modified. All chemicals were of analytical-reagent grade and were used without previous purification. Deionized water prepared by Demiwa 3 roi was used for the preparation of all solutions (<0.05 μS/cm, ISO 3696:1987). The bulk standard solution and calibration solutions of Pb(II) were prepared by dilution of Certified Reference Material (C_{Pb}=1000±2 mg/L, Merck). The sorption of lead ions to peat was studied by batch experiments. The sorption of heavy metals ions from aqueous solutions by sorbent was typically investigated with respect to temperature, pH, and sorbent doses [17, 18]. The factors that were examined were: the initial lead ion concentration, contact time between lead(II) and peat, and pH of the aqueous solution.

Determination of Peat pH

pH_{H_2O} of peat was measured using peat-water ratio 1:5 and a glass electrode pH meter S47K, which was standardized using buffer solutions of different pH values (4.01 and 7.00). 20 g of the peat was mixed with 100 ml deionized water and was shaken for 1 hour using a Gerhardt Rotoshake RS 12 mechanical shaker. The mixture was left for 60 minutes to allow it to settle [19]. PH_{KCl} of peat was measured by analogy instead of deionized water using 1 M KCl solution.

Determination of Moisture

Peat samples were sieved through 1 mm diameter sieve (Retsch, As 200) and dried at 100-105ºC for 24 h and then allowed to cool in the desiccators. According to mass difference of sample, before and after heating quantity of moisture in peat (%) was determined. Heated sorbent was cooled up to room temperature. It was weighed out 1±0.001 g of peat with the analytical balance KERN-770 for the sorption of Pb from an aqueous solution research.

Determination of Total Carbon (TC) of Peat

About 30±0.05 mg of dry peat was weighed. The total carbon content was analyzed with total organic carbon analyzer (TOC-V analyzer at 900ºC temperature).

Determination of Pb Quantity in Peat

Total Pb quantity determination in peat analysis was performed with atomic absorption spectrophotometry (Buck Scientific 210 VGP) after peat digestion in a mixture of HNO_3:H_2O_2 (10 ml, 65%: 2 ml, 30%) extraction solution using ETHOS microwave digestion system (Milestone). Then the mixture of the sample and acid was filtered through 0.45 μm glass filters, diluted with deionized water to 50 ml, and lead concentration determined using atomic absorption spectrophotometry applying the GAAS method [20].

Kinetics Sorption Studies

Kinetics sorption studies were carried out at room temperature (20±1.0ºC) using peat samples under static conditions. All the tests were done in capped conical 250 ml flasks filed with 100 mL of Pb(II) ion solution containing 1.0 g of peat, adjusting the pH to the required value (pH=4.0-7.0) by using buffer solution consisting of orthophosphoric acid (85%, Merck), boric acid (Merck), and acetic acid (30%, Merck) mix with sodium hydroxide solution (Merck). Additional quantity of NaOH 0.1 N or HNO_3 0.1 N solutions was added if needed for pH regulation. Lead concentration was in the range 0.25-100.0 mg/L. The mixture was agitated with a RS-12 Rotoshake rotary shaker (Gerhard, Germany) at a fixed speed of 20 rpm for 5, 10, 20, 45, 90, 180, and 360 minutes, respectively. At the end of each contact time, the solid was removed by filtration through a 0.45 μm glass filter and then analyzed using AAS (FAAS and GFAAS). Control samples containing all other reagents except adsorbent also were analyzed.

Each pH, lead concentration in polluted water and in peat, and total carbon and humidity of peat were measured five times. The differences were considered significant at p<0.05. Statistical data (average, confidence interval, standard deviation) were calculated using Microsoft Office Excel 2013.

Results and Discussion

The adsorption of lead to peat can be explained on the basis of the constituent of peat. Peat contains polar functional groups such as aldehydes, ketones, acids, and phenolic. These groups can be involved in chemical bonding and are responsible for the cation exchange capacity of the peat [21, 22]. Metal ion adsorption from an aqueous solution is affected by pH, properties of adsorbent, adsorbate concentration, and the presence of co-ions in solution.

The sorption process can be grouped as: physical and chemical properties of metal ions (i.e., molecular weight, ionic radius, oxidation state); properties of biosorbent (i.e., the structure of the biomass surface); and the experimental conditions (i.e., pH, temperature, concentration of biosorbent, the concentration of sorbate).

Peat can be modified in order to reduce several deficiencies, which are: low chemical stability, low mechanical strength, and the tendency of its particles to expand or shrink. The influence of initial metal ions concentration and equilibrium contact time was studied in a series of batch experiments, in comparison with natural peat. An increase in adsorption capacity of alkaline-treated peat was obtained up to 23.07% for Pb(II). Based on the results of the determination of adsorption activity and microcalorimetric studies, it was established that physical absorption processes predominate on the interaction of the modified peat sorbents with a model organic substance and the aqueous solutions of metal salts [23]. Peat properties were determined: $pH_{KCl} = 4.80 \pm 0.059$, $pH_{H_2O} = 5.90 \pm 0.072$, φ (moisture) = 44.67±1.11%, TC (total carbon) = 51.83±0.19%, and Pb (lead) =3.31±0.313 ppm.

The influence on adsorption by pH was studied over the pH range 4.0-7.0, because at low pH values (≤3.0) lead adsorption is small, even desorption of adsorbed lead ions starts and at higher pH (>7.5) sedimentation of hydroxide of lead starts in solution. The adsorption of lead is dependent over the pH range from 4.0 to 7.0. Inside selected pH domain, the lead exists predominantly as a divalent free ions (Pb^{2+}) so that it can be involved in chemical processes [24]. Using peat in conditions of higher concentrations of lead cations is connected with solution pH decrease. This fact is due to releasing hydrogen ions from peat into solution. The higher the initial metal concentration, the stronger the pH decrease, because the ion exchange is more intensive. At lower concentrations the ion exchange isn't so dominant and can't decrease the pH value. Decreasing the solution pH effect was small due to the impact of used buffer solutions; it reaches up to 0.10-0.15 of pH value. PH value of solution has a big influence on adsorption of lead(II) ions. Peat exhibited relatively higher metal removal efficiencies at pH 4.5 compared to pH 2.9 [25]. The most effective pH value for peat was about 6.0.

The influence of lead concentration on adsorption was studied over the lead concentration range 0.25-100.0 mg/L. With reference to the requirements for sewage cleaning in Lithuania, maximum allowable concentration (MAC) of lead to the system of sewage collection is equal to 0.5 mg/L (1 MAC). The concentrations of lead selected for researches were equal to these values: 1 MAC, 50 MAC, 100 MAC, 150 MAC, and 200 MAC. Lead removal efficiency decreases with increasing initial metal ion concentration. This fact is caused by limitation of peat sorption capacity.

Adsorption capacity of peat increases with increasing contact time, initial lead(II) concentration, and was significantly affected by initial pH value in the range from 4.0-7.0.

Conditions of lowest peat adsorption: contact time 5 min, initial lead(II) concentration 0.5 mg/L, pH 4.0 adsorption capacity-0.09973 mg/g, pH 5.0 adsorption capacity-0.09979 mg/g, pH 6.0 adsorption capacity-0.09980 mg/g, and pH 7.0 adsorption capacity-0.09978 mg/g. When contact time was 360 min, initial lead(II) concentration was 100 mg/L, pH was 4.0 adsorption capacity was 7.283 mg, when pH was 5.0 adsorption capacity was 9.233 mg, and when pH was 7.0 adsorption capacity was 8.244 mg. The amount of Pb adsorbed on peat was the greatest at pH 6.0, 360 min contact time, and 100 mg/L lead(II) concentration, i.e. 9.489 mg/g.

Freundlich Isotherms

Equilibrium relationships between sorbents and sorbates are described by sorption isotherms, which give the capacity of a sorbent for a sorbate. Isotherms can be obtained by examining batch reactions at fixed temperatures. Linear regression is frequently used to determine the best-fitting isotherm. The linear least-squares method with linearly transformed isotherm equations has also been widely applied to confirm experimental data and isotherms using coefficients of determination [25]. The adsorption kinetics can be described by the Langmuir or Freundlich equations [26]. These isotherms were selected for lead adsorption onto peat mathematical describing. Not always do adsorption isotherms fit the typical Freundlich and Langmuir equations [27]. The results show that the adsorption rates are rapid: equilibrium is reached in 90-180 minutes.

Freundlich isotherm is considered as the adsorption characteristics on the heterogeneous surface. Freundlich isotherm curves in the opposite way of Langmuir isotherm and is exponential in form. The heat of adsorption, in many instances, decreases in magnitude with increasing extent of adsorption. This decline in heat is logarithmic, implying that the adsorption sites are distributed exponentially with respect to adsorption energy. This isotherm does not indicate an adsorption limit when coverage is sufficient to fill a monolayer (θ = 1) [28]. The Freundlich isotherm can be calculated by the formula:

$$\log q_e = \log k_F + \frac{1}{n}\log C_e \qquad (1)$$

...where:

q_e – amount of adsorbate adsorbed per unit weight of adsorbent, mg/g

k_F – equilibrium constant indicative of adsorption capacity, (mg/g)(l/mg)1/n

n – adsorption equilibrium constant

C_e – concentration of adsorbate in solution at equilibrium, mg/L

The constants k_F and n, which respectively indicate adsorption capacity and the adsorption intensity, are founded from the intercept and slope of plot log*q* *versus* logC_e accordingly, as shown in Fig. 1 and listed in Table 1.

Table 1. Freundlich isotherm parameters.

	pH = 4.0	pH = 5.0	pH = 6.0	pH = 7.0
k_F	0.069	0.024	0.021	0.297
n	0.786	0.842	0.917	0.777
R^2	0.998	0.993	0.984	0.990

Table 2. The Langmuir isotherm parameters.

	pH = 4.0	pH = 5.0	pH = 6.0	pH = 7.0
Q^o	8.811	3.626	3.957	3.655
b	0.009	0.007	0.005	0.009
R^2	0.584	0.778	0.819	0.734

$$\text{Freundlich isotherm slope} = (1/n) \quad (2)$$
$$\text{and the intercept} = \log k_F \quad (3)$$

The values of R^2 are situated within the range of 0.984 -0.998, demonstrating that the experimental data fit well with the Freundlich isotherm equation and these outcomes evidence that the lead adsorption on the samples followed the Freundlich isotherm model and such adsorption mainly occurred on the heterogeneous surface of the samples (Table 1).

From the Freundlich isotherm which best described the data for the batch isotherm studies, it can be observed that lower k value was obtained at pH 4, which suggested that peat had lower lead adsorption capacity at pH 4. The low adsorptive capacity can be attributed to hydrogen ions that compete with the lead ions on sorption sites. A sharp increase in adsorption was observed within a pH range of 4.0 and 6.0, and maximum adsorption occurred at pH 6. At low pH some functional groups may be positively charged and their interaction with metal ions can be highly reduced.

Langmuir Isotherms

The Langmuir isotherm represents the equilibrium distribution of metal ions between the solid and liquid phases. The Langmuir isotherm equation is represented in formula (4) [29]:

$$\frac{C_e}{q_e} = \left(\frac{C_e}{Q^0}\right) + \left(\frac{1}{bQ^0}\right) \quad (4)$$

...where:

q_e – amount of adsorbate adsorbed per unit weight of adsorbent, mg/g
Q^o – constant related to the adsorption capacity, mg/g
b – Langmuir constant, mg/ml
C_e – concentration of adsorbate in solution at equilibrium, mg/L

The Langmuir model represents theoretical treatments of non-linear sorption and suggests that uptake occurs on a homogenous surface by monolayer sorption without interaction between adsorbed molecules. It assumes that adsorption sites on the adsorbent surfaces are occupied by the adsorbate in the solution. Therefore the Langmuir constant (b) represents the degree of adsorption affinity the adsorbate. High value of b indicates much stronger affinity of metal ion adsorption. The shape of the isotherm (assuming the (x) axis represents the concentration of adsorbing material in the contacting liquid) is a gradual positive curve that flattens to a constant value. The Langmuir isotherm parameters Q^o and b were evaluated from the slope and intercept by formulas below and listed in Table 2.

$$\text{Slope of } (C_e/q_e) \text{ versus } C_e \text{ curve} = \frac{1}{Q^0} \quad (5)$$

$$\text{Intercept of } (C_e/q_e) \text{ versus } C_e \text{ curve} = \frac{1}{bQ^0} \quad (6)$$
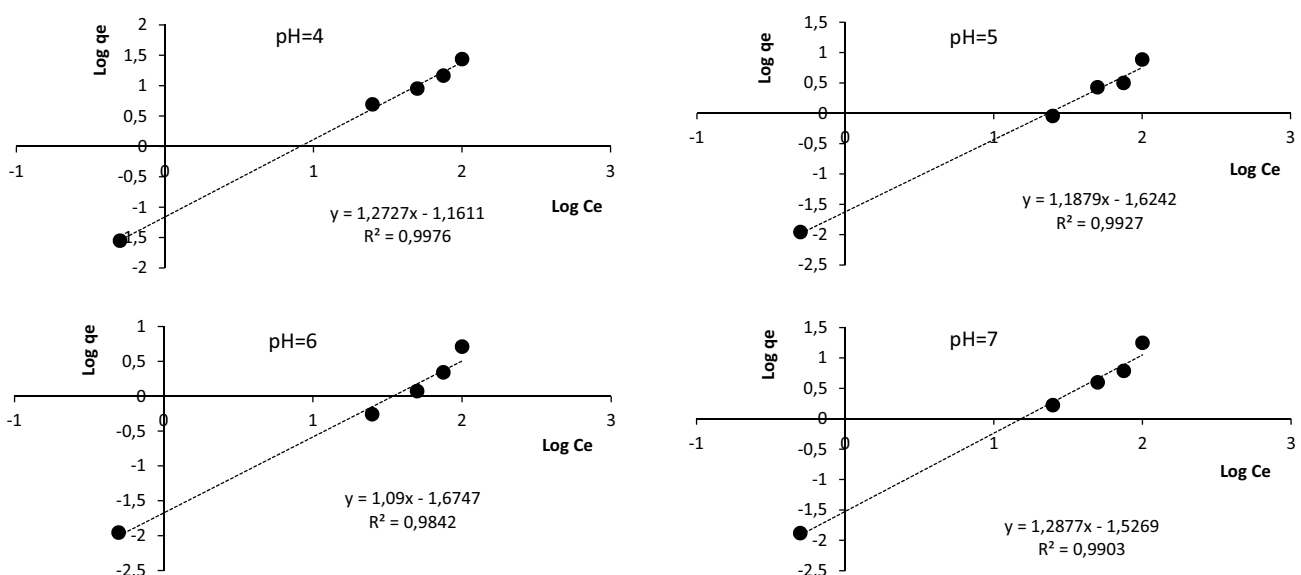

Fig. 1. The Freundlich adsorption isotherms for Pb²⁺ ion adsorption by peat at different pH.
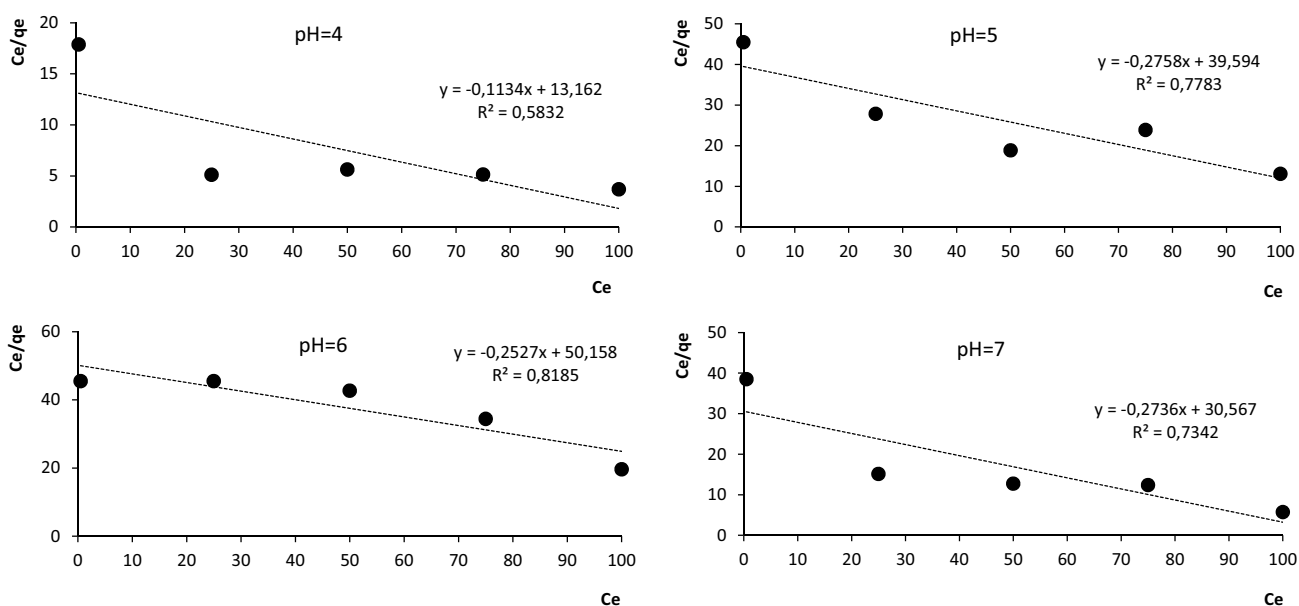
Fig. 2. The Langmuir sorption isotherms for Pb²⁺ ion adsorption by peat at different pH.

A plot of C_e/q_e versus C_e gives a straight line of slope $1/Q^o$ and intercept $1/(Q^o \times b)$, for example, as shown in Fig. 2.

It can be found that the values of R^2 are located in the range of 0.584-0.819, suggesting that these experimental data fitted worse with the Langmuir isotherm equation. From the isotherm shape it can be seen if the sorption system is "favourable" or "unfavorable." Hall with colleagues in 1966 proposed a dimensionless separation factor R, which is based upon an initial assumption of the applicability of the given data to Langmuir isotherm model to predict if an adsorption system is "favourable" or "unfavourable." If $R>1$, adsorption is unfavourable; if $R=1$, adsorption is linear; if $0<R<1$, adsorption is favourable; if $R=0$, adsorption is irreversible [30]. So, when concentration of the lead is $C_o = 100$ mg/l, R calculated: at pH = 4.0, $R = 0.5263$, at pH = 5.0, $R = 0.5882$, at pH = 6.0, $R = 0.6667$, at pH = 7.0, and $R = 0.5882$. Based on the separation factor R calculations, values are in the range of $0 <R< 1$, which indicates that the adsorption of lead ions is favourable.

From Table 1 and Table 2 it is found that the adsorptions of lead correlated better on Freundlich isotherm model ($R^2 >0.98$) than on Langmuir ($R^2>0.58$). The experimental equilibrium data better correlated to the Freundlich equation, but the Langmuir model fits also well. This study showed the possibility of peat utilization as a low-cost sorbent for Pb(II) removal from model solutions.

Conclusions

1. Peat as a natural material can be used as a low-cost and usable material to control toxic metals (Pb) in liquid solutions. Adsorption capacity of peat increases with increasing contact time and initial lead(II) concentration, and was significantly affected by initial pH value in the range from 4.0-7.0. The amount of Pb adsorbed on peat was the greatest at pH 6.0, 360 min contact time, and 100 mg/L lead(II) concentration, i.e. 9.489 mg/g.

2. The Langmuir isotherms model was statistically significant, correlation coefficients R^2 at pH 4.0-0.584, at pH 5.0-0.778, at pH 6.0-0.819, and at pH 7.0-0.734.

3. The model parameters obtained for the Freundlich model were statistically significant, correlation coefficient R^2 at pH 4.0-0.998, at pH 5.0-0.993, at pH 6.0-0.984, and at pH 7.0-0.990.

4. The equilibrium adsorption data fit well to the two models (Langmuir and Freundlich), but gave a better fit with the Freundlich model, as evidenced by the higher value of R^2-0.98.

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