Pb(II) and Cu(II) Sorption from Aqueous Solutions Using Activated Red Mud – Evaluation of Kinetic, Equilibrium, and Thermodynamic Models

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Abstract

We investigated the influence of contact time, sorbent dose, and initial Cu(II) or Pb(II) concentrations on the removal of Cu(II) and Pb(II) from aqueous solution by the batch sorption technique using activated red mud (aBauxsol) as a low-cost sorbent. Sorption equilibrium was achieved after 1 h at pH 5.5 for both ions. Pseudo first and pseudo second order models were used for analyzing the kinetics of sorption processes. The rate of both sorption processes is controlled by diffusion in the film fluid and diffusion within particles. The processes of sorption follow the Langmuir model.

Keywords: aBauxsol, lead, copper, sorption, kinetics

Introduction

Heavy metals are a major pollutant. They are found in the natural environment as a result of mineral extraction, industrial production, urban activities, and similar activities [1]. They are extremely toxic, and unlike organic pollutants they are not biodegraded by natural processes, but accumulated into soil by intervention of living organisms [2]. Through the food chain, heavy metals enter into organisms of a large number of plants and animals accumulating in the animals that are at the top of the chain. In this way heavy metals enter the human body through the food chain.

Copper and its compounds are widely used in many industries, such as electroplating, mining, electrical, iron and steel production, and the printing and photographic industries, and there are many potential sources of copper pollution [3]. Lead comes into water through the combustion of fossil fuels and the smelting of sulfide ore. Process industries, such as battery manufacturing and metal plating and finishing, also are a prime source of lead pollution [2]. High concentrations of copper can cause numerous disorders in the human body: fatigue, depression, anorexia, allergies, migraine headaches, and many others [4], while high concentrations of lead can cause severe damage of liver, kidney, brain, and nervous system [5]. According to the World Health Organization, the permissible limit of copper in drinking water is 2 mg/L [6], and for the lead is 10 µg/L [7]. Studies have proven that these metals are commonly found in water and wastewater. So the removal of these very toxic metals from wastewater is necessary.

For removing heavy metals from aqueous solutions various methods have been used: chemical precipitation [8], flotation [9], membrane separation [10], ion

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exchange, and electrolysis. However, adsorption is generally preferred for the removal of heavy metal ions due to its high efficiency, easy handling, availability of different adsorbents, and cost effectiveness [6]. Various sorbents like alumina [11], kaolinite [12], illite [13], bentonite [14], zeolite [15], diatomite [16], and activated carbon [4, 17] are used. Recently, there has been an increasing emphasis on adsorbents with low cost, like plant wastes, or industrial by-products, such as granular red mud, chitosan, potato peel, peanut husks, carbon, pomegranate peel, and waste tire rubber ash [18].

Red mud (an aluminium industry waste) has received wide attention as an effective adsorbent for various aquatic pollutants [19]. The use of red mud as an adsorbent for heavy metals (Cu(II), Pb(II), Cd(II), Cr(V), As(III), As(V), Ni(II), Zn(II)) has been advocated [2, 6, 20-25]. The neutralization or modification of raw red mud with acid or heat treatment was found to considerably improve sorption capacity in many studies [19].

In this work the sorption potential of activated red mud (aBauxsol) for the removal of Pb(II) and Cu(II) from aqueous solutions is examined. The influence of contact time, sorbent dose, and initial Cu(II) or Pb(II) concentration on the removal of Cu(II) and Pb(II) from aqueous solution by the batch sorption was investigated.

Materials and Methods

Apparatus

Lead and copper was analyzed using flame atomic absorption spectrophotometry (FAAS) (Perkin Elmer AAnalyst 300). A laboratory pH meter (Metrohm 827) was used for pH measurements.

Preparation of aBauxsol Sorbent

The red mud used in this study was obtained from the aluminium plant in Podgorica, Montenegro. Seawater-neutralized red mud (Bauxsol) was prepared by adding 4 L of seawater to 4 kg of red mud. The solution was stirred for 2 h until the pH reached an equilibrium of 9.0±0.2. Subsequently, the solution was dried. The obtained Bauxsol powder was sieved through a 0.2 mm screen before further application. The acid treatment method of Pratt and Christoverson [26] was applied for the preparation of activated adsorbent (aBauxsol) in the following way: the initial Bauxsol powder was refluxed in 20 mL of 20% HCl for 20 min. The acid slurry was filtered and the residue was washed with deionized water to remove residual acid and soluble Fe and Al compounds. Finally, the residue was dried at 40ºC, then sieved again through a 0.2 mm screen and used for the experiments.

The aBauxsol is primarily composed of Fe_2O_3 – 41.76%, Al_2O_3 – 18.2%, SiO_2 – 11.3%, and TiO_2 – 6.13%. The specific surface area of aBauxsol was 40 $m^2 \cdot g^{-1}$ [27].

Batch Experiments

Lead(II) stock solution was prepared by dissolving $Pb(CH_3COO)_2 \cdot 3H_2O$ in water to a concentration of 0.10 $mol \cdot L^{-1}$ lead. This solution was used for the preparation of test solutions with lead concentrations ranging $5 \cdot 10^{-5}$-$5 \cdot 10^{-3}$ $mol \cdot L^{-1}$ lead. Copper(II) stock solution was prepared by dissolving $CuCl_2 \cdot 2H_2O$ in water to a concentration of 0.10 $mol \cdot L^{-1}$ copper. This solution was used for the preparation of the test solutions with copper concentrations ranging $5 \cdot 10^{-5}$-$5 \cdot 10^{-3}$ $mol \cdot L^{-1}$ copper.

When the effect of contact time was analyzed, 0.2 g of the adsorbent was added to plastic bottles containing 50 mL of a solution of lead or copper at an initial concentration ranging from $5 \cdot 10^{-5}$ to $5 \cdot 10^{-3}$ $mol \cdot L^{-1}$. The bottles were shaken with a mechanical shaker at room temperature (23±1ºC) until equilibrium was attained. The equilibrium contact times were found by taking sub-samples from the bottles several times over 2 h (5, 10, 15, 30, 60, 90, and 120 min.).

When the mass of adsorbent was varied, 1.0 to 5.0 g of aBauxsol adsorbent was added to plastic bottles containing 100 mL of a solution of copper or lead at an initial concentration of $5 \cdot 10^{-4}$ and $2.5 \cdot 10^{-3}$ $mol \cdot L^{-1}$ for copper and lead, respectively. Then the bottles were shaken with a mechanical shaker at room temperature (23±1ºC) for 1 h.

When the initial concentration of lead or copper was varied, 0.4 g of adsorbent aBauxsol was added to plastic bottles containing 100 mL of solution with an initial lead or copper concentration ranging from $5 \cdot 10^{-5}$ to $5 \cdot 10^{-3}$ $mol \cdot L^{-1}$.

Sample pHs (pH=5.5±0.1) are adjusted through the addition of 2 M NaOH standard solutions. The batches were than centrifuged and the lead and copper was analyzed in each supernatant. All glassware and plastic bottles used in the experiments were washed with 6% HNO_3, then with deionised water and dried. All samples were analyzed in duplicate (or triplicate), whereby precision better than ±5% was indicated.

Results and Discussion

Kinetic Studies

The influence of time required to establish equilibrium in the examined systems depends on the initial concentration of the solution ($5 \cdot 10^{-5}$, 10^{-4}, $5 \cdot 10^{-4}$, and $2.5 \cdot 10^{-3}$ $mol \cdot L^{-1}$).

The amount of Pb(II) and Cu(II) ions sorbed over time on aBauxsol are shown in Fig. 1. It is noticeable that the curves have the same form regardless of the type of ions and the initial concentration of solution.

Sorption of both metal ions increases with increases of initial concentrations and contact times. Each curve can be divided into two parts that which respond to fast and slow phases of the process. The rapid initial phase of the process is determined by a number of free active sites on the surface of sorbent. Also, in the beginning of the process the difference of concentrations of metals in solution and the
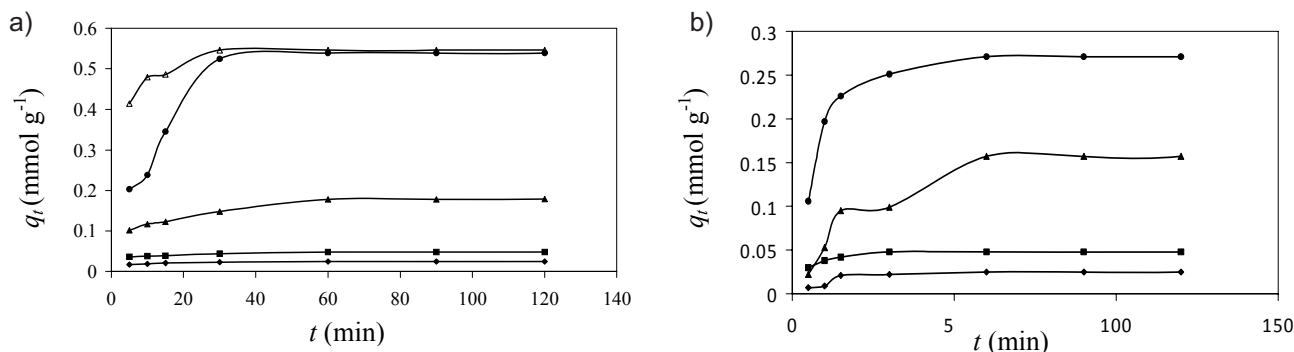
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Fig. 1. (a) Lead and (b) copper sorption using aBauxsol as a function of contact time. Initial lead and copper concentration (mol·dm⁻³): (♦) 5·10⁻⁵; (■) 10⁻⁴; (▲) 5·10⁻⁴; (●) 2.5·10⁻³; (Δ) 5·10⁻³, pH = 5.5±0.1 and sorbent dosage = 4.00 g·L⁻¹.

sorbent surface (i.e. driving force for mass transfer) is the largest one. During the time, driving force is reduced, making it a condition of slower mass transfer in the second phase of the process. The amount of Cu(II) and Pb(II) sorbed increases over time until the moment of established equilibrium. That time is 60 min for both ions at all tested concentrations, which is in accordance with literature data [28].

The obtained kinetic data was analyzed by application of the pseudo-first-order and pseudo-second-order models [29].

The linear forms of the pseudo-first-order and pseudo-second-order equations are as follows:

$$\ln\left(q_e - q_t\right) = \ln q_e - k_1 t \tag{1}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{2}$$

...where:

q_t (mmol·g⁻¹) is the sorption capacity at time t

q_e (mmol·g⁻¹) is the equilibrium sorption capacity

k_1 (min⁻¹) is the rate constant of sorption

k_2 (g·mg⁻¹·min⁻¹) is the rate constant of sorption.

The obtained results are presented in Figs. 2 and 3 and in Tables 1 and 2.

Comparing these two models based on parameter R^2, it can be concluded that the pseudo second order model better describes the sorption processes at all tested concentrations of both ions. The pseudo second order model was found to be the most appropriate in many investigations [14, 17].
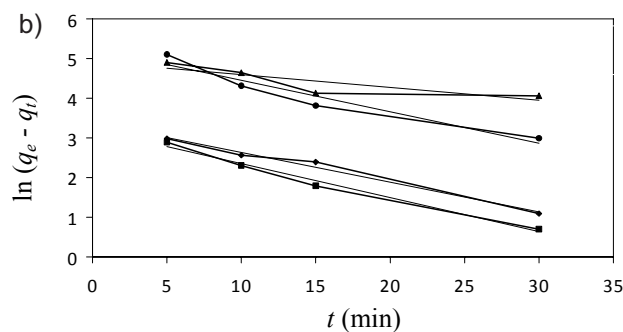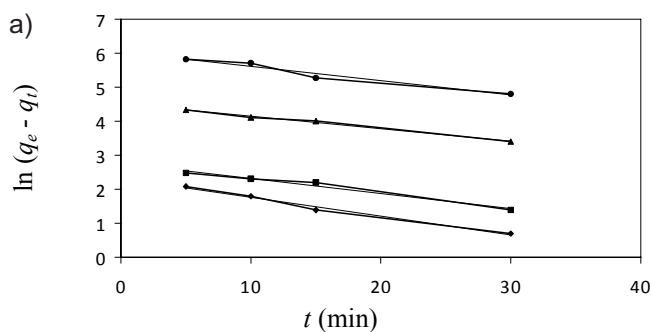
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Fig. 2. Pseudo-first-order modeling of (a) lead and (b) copper sorption kinetics on aBauxsol. Initial lead and copper concentration (mol·dm⁻³): (♦) 5·10⁻⁵; (■) 10⁻⁴; (▲) 5·10⁻⁴; (●) 2.5·10⁻³; pH = 5.5±0.1 and sorbent dosage = 4.00 g·L⁻¹.
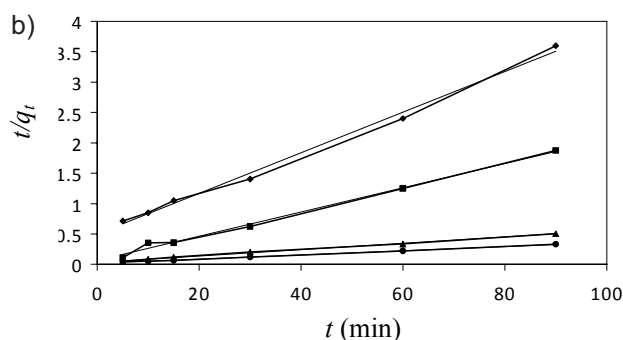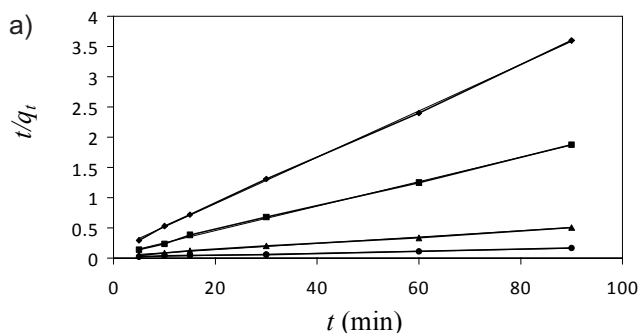
a)

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Fig. 3. Pseudo-second-order modeling of (a)lead and (b)copper sorption kinetics on aBauxsol. Initial lead and copper concentration (mol·dm⁻³): (♦) 5·10⁻⁵; (■) 10⁻⁴; (▲) 5·10⁻⁴; (●) 2.5·10⁻³; pH = 5.5±0.1 and sorbent dosage = 4.00 g·L⁻¹.

Table 1. Kinetic model parameters and correlation coefficients for Pb(II) sorption on aBauxsol.

Kinetic model	Parameter	Concentration (mmol·L^{-1})			
		$5 \cdot 10^{-5}$	$1 \cdot 10^{-4}$	$5 \cdot 10^{-4}$	$2.5 \cdot 10^{-3}$
Pseudo first-order	K_1 (min^{-1})	-0.055	-0.044	-0.039	-0.042
	q_e (mmol/g)	0.010	0.016	0.090	0.580
	R^2	0.987	0.977	0.993	0.957
Pseudo second-order	K_2 (g/mmol·min)	0.645	11.206	0.700	5.800
	q_e (mmol/g)	0.050	0.026	0.200	0.100
	R^2	0.999	0.999	0.996	0.989

Table 2. Kinetic model parameters and correlation coefficients for Cu(II) sorption on aBauxsol.

Kinetic model	Parameter	Concentration (mmol·L^{-1})			
		$5 \cdot 10^{-5}$	$1 \cdot 10^{-4}$	$5 \cdot 10^{-4}$	$2.5 \cdot 10^{-3}$
Pseudo first-order	K_1 (min^{-1})	0.085	0.075	0.032	0.079
	q_e (mmol/g)	0.240	0.290	0.140	0.380
	R^2	0.986	0.987	0.739	0.932
Pseudo second-order	K_2 (g/mmol·min)	2.226	6.558	0.757	0.090
	q_e (mmol/g)	0.030	0.050	0.200	0.300
	R^2	0.994	0.994	0.997	0.997

Mechanisms of Mass Transfer

Diffusion in the Fluid Film

The results obtained by kinetics of below-mentioned sorption processes were used for calculation of mass transfer coefficients.

The external mass transfer model is defined by the Mathews-Weber model:

$$\ln \frac{c_t}{c_0} = -k_f at \qquad (3)$$

By applying this model to the sorption process Pb(II) and Cu(II) ions (Fig. 4), volumetric mass transfer coefficients that characterize the diffusion of metal ions through fluid boundary layer around the sorbent (Table 3) were calculated. The table notes that with an increase of initial concentration of the solution mass transfer coefficients decrease in both studied systems. This phenomenon can be explained by the fact that the bonding mechanism of Cu(II) and Pb(II) ions to the surface of sorbent is ion exchange, where there is a release of alternate cations from the surface of sorbent. This creates a concentration gradient of opposite direction than the one obtained by diffusion of Cu(II) or
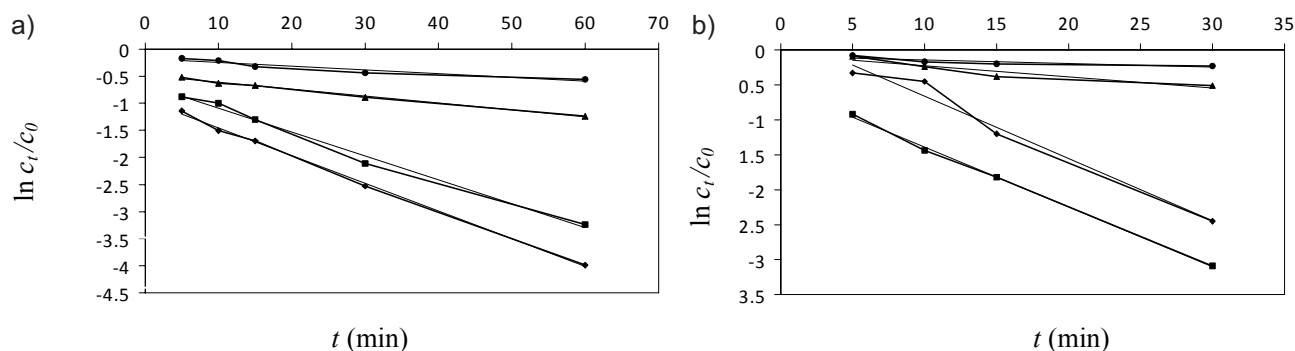

Fig. 4. Fitting of the experimental results of sorption (a) lead and (b) copper ions on aBauxsol using linear forms M & W equation: Initial lead and copper concentration (mol·dm^{-3}): (♦) $5 \cdot 10^{-5}$; (■) 10^{-4}; (▲) $5 \cdot 10^{-4}$; (●) $2.5 \cdot 10^{-3}$; pH = 5.5 ±0.1 and sorbent dosage = 4.00 g·L^{-1}.

Table 3. Volumetric mass transfer coefficients through fluid film for the sorption of Cu(II) and Pb(II) ions from solution with different initial concentration ions.

Concentration c_0 (mmol·L^{-1})	k_f·a (1/min)	
	Pb(II)	Cu(II)
5·10^{-5}	0.051	0.089
1·10^{-4}	0.044	0.085
5·10^{-4}	0.012	0.015
2.5·10^{-3}	0.007	0.005

Pb(II) ions. As a result, the transfer of these ions through the film of fluid is aggravated by diffusion of cations in the opposite direction. Diffusion resistance generated by the releasing cations is directly proportional to their concentration. With increasing concentrations of Cu(II) or Pb(II) ions in solution, the amount of discharged cations is increasing and thus the resistance to transport of Cu(II) and Pb(II) ions, which causes a decrease of the volumetric mass transfer coefficients.

Diffusion within Particles

For the definition of current mass transfer of resistance and limited degree of the total sorption process Weber-Morris's model was used for diffusion within the particle:

$$q_t = k_{id} \cdot t^{1/2} + C \qquad (4)$$

...where:
k_{id} is the intraparticle diffusion rate constant (μmol·g^{-1}·h^{-1})
C is a constant for any experiment (μmol·g^{-1}).

Equation (4) is a straight line in a diagram of q_t vs. $t^{1/2}$. If the line passes through the origin, the thickness of the boundary layer is negligible, mass transfer resistance is also negligible, and diffusion within the particles is the only limiting process. If not, diffusion within the sorbent is not the only limited process.

The dependence of q_t vs. $t^{1/2}$ for sorption Cu(II) and Pb(II) ions on aBauxsol are shown in Fig. 5.
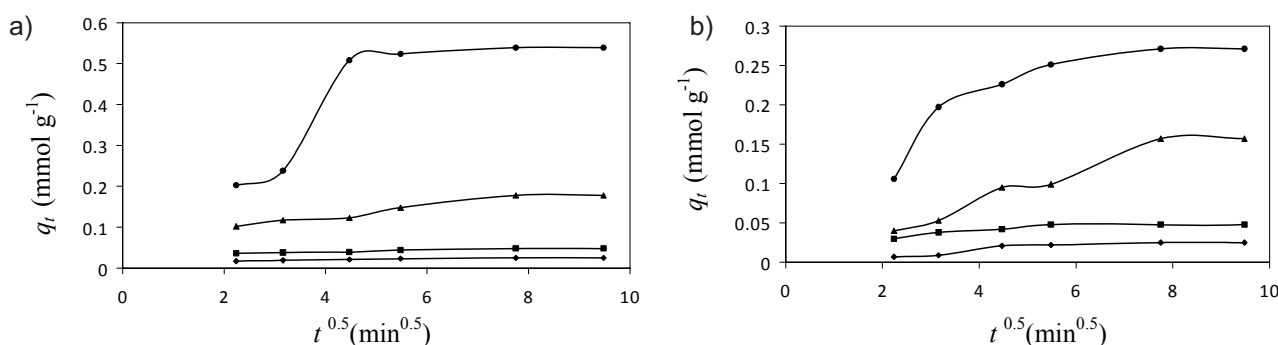
It is shown that obtained dependences are not a straight line and do not pass through the origin. This fact indicates the presence of diffusion through the fluid film, as well as pore diffusion within the sorbent [17, 30]. All curves can be divided into two parts, which can be linearized and the corresponding coefficients of direction, ie. gradients, are k_{id} of the process. The first of different phases of the process (the first segment of the curve) is related to the diffusion of metal ions through the film of fluid, while the second phase corresponds to the diffusion inside the sorbent pores.

K_{id} coefficients for different segments at different concentrations of both ions are given in Table 4.

According to all concentrations, values of $k_{id,1}$ for both ions are higher than relevant values of $k_{id,2}$, which confirms the fact that initial sorption is a fast process. Also, the coefficients of the direction are increasing along with the increase of initial concentration of solution, regardless of the type of ion and the observed segment.

This observation can be explained by the fact that Weber-Morris's model based on Fick's law that increases of the concentration gradient causes faster diffusion and adsorption [30].

Equilibrium Studies

Examination of sorption at different initial concentrations of sorbate provides construction of sorption isotherm.

Adsorption isotherms are important for the description of how molecules of a sorbate interact with a sorbent surface and for the prediction of the extent of sorption [31]. Fig. 6 shows dependencies of equilibrium concentrations of metal ions in the solid phase of equilibrium concentration of Cu(II) and Pb(II) ions in solutions. In both cases pictures with the same quality are obtained: the two curves have a much higher slope at lower initial concentrations, which correspond to lower values of equilibrium concentration. With the increase of initial concentration, the curves slope downward and transfer into the plateau area.

The sorbed amount of metal ion that responds to the plateau of the curve represents the maximum sorption capacity at given experimental isotherm conditions. Below-mentioned isotherms belong to L-type isotherms.

Fig. 5. The Weber-Morris plot for (a) lead and (b) copper sorption by aBauxsol. Initial lead and copper concentrations (mol·dm^{-3}): (♦) 5·10^{-5}; (■) 10^{-4}; (▲) 5·10^{-4}; (●) 2.5·10^{-3}; pH = 5.5±0.1 and sorbent dosage = 4.00 g·L^{-1}.

Table 4. Weber-Morris model parameters for Pb(II) and Cu(II) sorption on a-Bauksol.

Pb(II)		Cu(II)		Concentration (mmol·L⁻¹)
$k_{id,1}\cdot10^2$ (µmol·g⁻¹·h⁻¹)	$k_{id,2}\cdot10^2$ (µmol·g⁻¹·h⁻¹)	$k_{id,1}\cdot10^2$ (µmol·g⁻¹·h⁻¹)	$k_{id,2}\cdot10^2$ (µmol·g⁻¹·h⁻¹)	
0.6	0.4	0.4	0.3	$5\cdot10^{-5}$
1.02	0.8	1.2	0.4	$1\cdot10^{-4}$
3.8	1.4	3.8	1.2	$5\cdot10^{-4}$
10.6	8.1	5.3	1.5	$2.5\cdot10^{-3}$

The sorption capacity of the aBauxsol for Pb(II) and Cu(II) ions was found to be 0.595 mmol·g⁻¹ (123.28 mg·g⁻¹) and 0.339 mmol·g⁻¹ (21.56 mg·g⁻¹), respectively. The aBauxsol sorbent analyzed exhibited high sorption capacity for Pb(II) and Cu(II) ions compared to red mud sorbents recently investigated [2, 6, 19]. The Langmuir and Freundlich isotherms are commonly used adsorption isotherms for assessing the potential use of an adsorbent for different applications. The Langmuir isotherm model is found to be suitable for the description of chemisorption. The Freundlich isotherm could also be applied to a sorption system describing the physical sorption of sorbate [32, 33]. The experimental data obtained for Pb(II) and Cu(II) were fitted by the linearized Langmuir and Freundlich isotherms, which are described by the following equations, respectively:

$$q_e = \frac{q_o\,K_L C_e}{1+K_L C_e} \qquad (5)$$

$$\frac{C_e}{q_e} = \frac{1}{q_o\,K_L} + \frac{C_e}{q_o} \qquad (6)$$

$$\ln q_e = \ln K + \frac{1}{n}\ln C_e \qquad (7)$$

...where:

C_e is the equilibrium concentration in µmol·L⁻¹

q_e is the amount adsorbed at equilibrium in µmol·g⁻¹

q_o is the adsorption maximum in µmol·g⁻¹

K_L is Langmuir equilibrium constant in L·µmol⁻¹

K is Freundlich constant in L·g⁻¹

n is constant related to sorption intensity of sorbent in g·µmol⁻¹.

Table 5. Langmuir and Freundlich model parameters for Pb(II) and Cu(II) sorption on a-Bauksol.

Model isotherm	Parameter	Cu(II)	Pb(II)
	K_L (L/mmol)	9.420	4.050
Langmuir	q_e (mmol/g)	0.338	0.620
	R_L	0.175	0.330
	R^2	0.998	0.998
Freundlich	K_F (mmol/g)	0.260	0.492
	n	3.540	2.630
	R^2	0.895	0.936

The linearized Langmuir isotherm for Cu(II) and Pb(II) are given in Fig. 7, and linearized isotherm plots of the Freundlich model are presented in Fig. 8.

According to obtained dependencies, isotherm parameters were calculated for both ions, and the results summarized in Table 5.

Comparing these two isotherms based on factors R^2, it can be said that Langmuir's sorption isotherms better describe both ions on the examined sorbent. This observation is in accordance with literature data [4, 14, 17]. The maximum sorption capacity calculated using Langmuir's isotherm is very close to experimentally determined capacity (0.338 mmol·g⁻¹ for Cu(II) and 0.62 mmol·g⁻¹ for Pb(II)). Although the conditions of running isotherms deviate from real conditions, it is proved that this equation can describe a number of sorption processes.
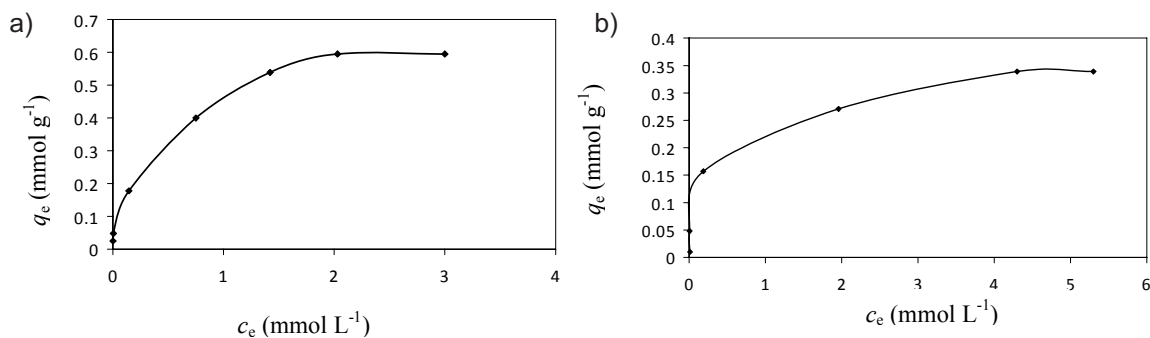

Fig. 6. Sorption isotherm for (a) lead and (b) copper sorption on aBauxsol.
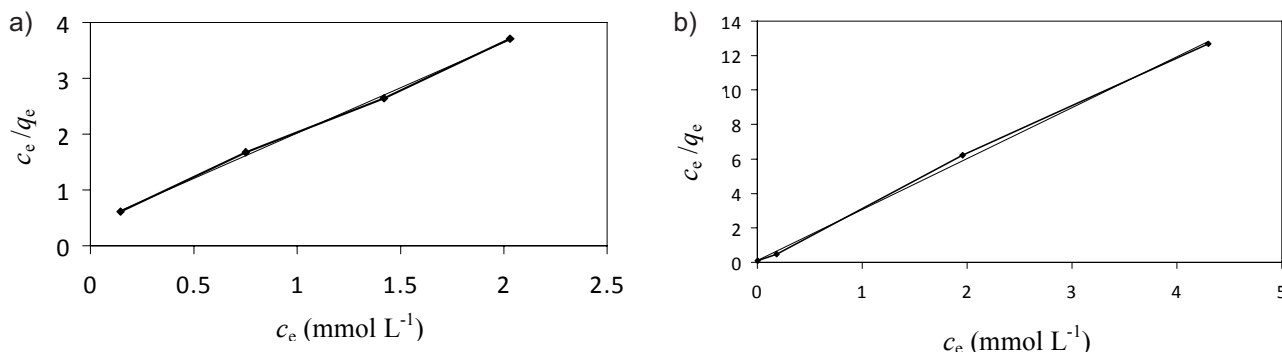
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Fig. 7. Langmuir isotherm for (a) lead and (b) copper sorption on aBauxsol.

The shape of the Langmuir isotherm was investigated by the dimensionless constant separation factor R_L to determine whether Pb and Cu adsorption by aBauxsol is a high-affinity adsorption. The R_L values were calculated as [34]:

$$R_L = \frac{1}{1 + K_L\,C_o} \qquad (8)$$

...where C_0 is the initial arsenate concentration ($\mu mol \cdot L^{-1}$).

The parameter R_L indicates the shape of the adsorption isotherm and $0 < R_L < 1$ corresponds to high-affinity adsorption. Calculated values of R_L are between 0 and 1 for both ions (0.33 to 0.175 for Pb and Cu, respectively). This fact indicates that the processes of sorption of both ions are favored. Favored sorption is reflected in the convex isotherm, which is confirmed experimentally (Fig. 6).

Based on the known values of Langmuir constant-K_L, the change of Gibbs function (ΔG) of examined sorption processes can be calculated. Change of Gibbs function is an important thermodynamic parameter of each process and is given by:

$$\Delta G = -RT \ln K_L \qquad (9)$$

The negative ΔG values for copper (-19.998 kJ·mol⁻¹) and lead (-10.874 kJ·mol⁻¹) indicate that both processes take place spontaneously at room temperature.

The Effect of Sorbent Dose

The influence of sorbent dose on the sorption kinetics of Pb(II) and Cu(II) is given in Fig. 9. The amount of attached ions per unit mass of sorbent decreases with increase of sorbent dose.
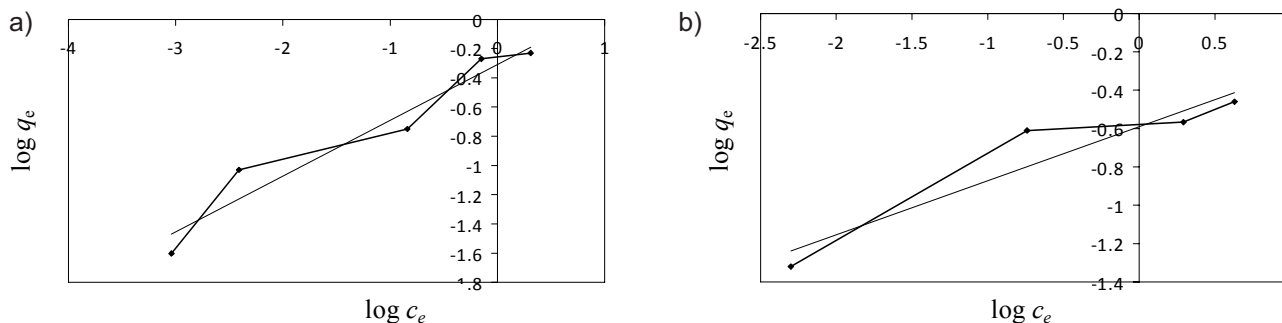
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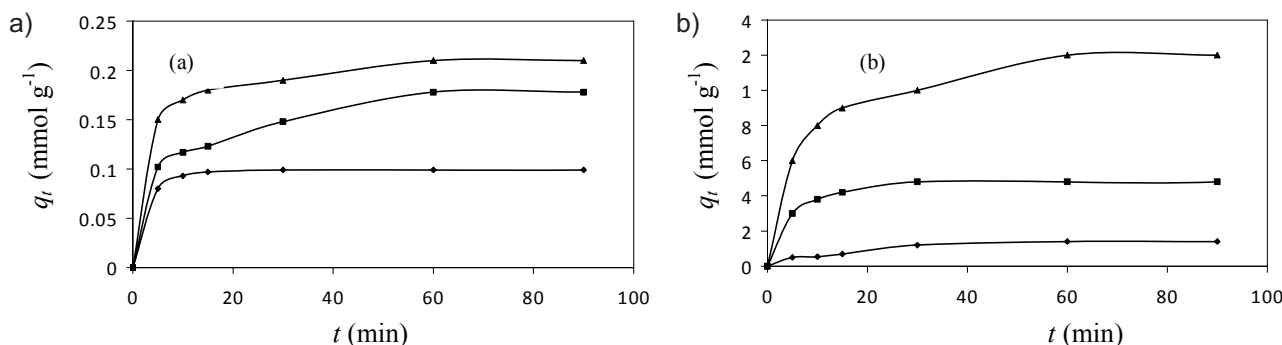Fig. 8. Freundlich isotherms for (a) lead and (b) copper sorption on aBauxsol.

a)

b)

Fig. 9. (a) Lead and (b) copper sorption using aBauxsol as a function of contact time and sorbent dosage: (♦) 50 g·L⁻¹; (■) 20 g·L⁻¹; (▲) 10 g·L⁻¹.

For the sorbent dose, 10, 20, and 50 g·L⁻¹ sorbed amounts of Pb(II) ions after an hour are 0.210, 0.178, and 0.099 mmol·g⁻¹, respectively.

In the same conditions, sorbed amounts Cu(II) ions are 0.120, 0.048, and 0.014 mmol·g⁻¹. The observed effect can be explained by the fact that the unit mass of sorbent binds a larger amount of metal ions at the lowest dose of sorbent. Also, increase of concentration of sorbent reduces the concentration gradient between the sorbent and solution, resulting in a reduction of sorbed metal ions per unit mass of sorbent. This phenomenon can be explained on the basis of the initial concentration gradient between the sorbent and solution. For a given dose of sorbent and volume of initial solution, concentration gradient increases with increases of solution concentrations. A reduced amount of added sorbent has the same effect on the concentration gradient as well as the increase of solution concentration [35].

Conclusions

Activated red mud (Bauxsol) was examined for potential use as low-cost material for the removal of lead and copper from wastewater. Several parameters were studied: contact time, sorbent dose, and initial Cu(II) or Pb(II) concentration on the removal of Cu(II) and Pb(II) from aqueous solution. For both metal ions the sorption efficiencies increased with increasing contact time and initial metal concentration; the maximum observed capacity of the aBauxsol sorbent was 123.28 mg·g⁻¹ and 21.56 mg·g⁻¹ for Pb(II) and Cu(II), respectively. This was achieved within 1 hour-contact with 4 g·L⁻¹ of aBauxsol sorbent at a pH of water 5.5.

Pseudo first and pseudo second order models were used for analyzing the kinetics of sorption processes. The pseudo second order model better describes the sorption processes at all tested concentrations of both ions. The rate of both sorption processes is controlled by diffusion in the film fluid and diffusion within particles. The ΔG^0 values show that both processes are spontaneous at room temperature. It is shown that the efficiency of sorption decreases with increases of sorbent doses for both metal ions. The processes of sorption follow the Langmuir model.

Future research should consider desorption mechanisms of the examined waste material, as well as the problem of disposal of enriched adsorbent. The cost of removal is expected to be quite low, as the sorbent is cheap and easily available in large quantities.

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