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Selective removal of lead ions from aqueous solutions using SiO_2–MoO_3: Isotherm, kinetics and thermodynamic studies

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ABSTRACT

This study reveals novel data on adsorptive removal of Pb(II) from single metal solution, as well as from multi-metal solution containing Cd(II), Co(II), Cr(III), Cu(II), Fe(III), Mn(II), Ni(II) and Zn(II) ions along with Pb(II) using inorganic SiO_2–MoO_3 sorbent in a batch system. The results of multi-metal solution system were strongly pH-depended and due to occurred precipitation of Fe(III) in the form of hydroxide. The parameters affecting the sorption efficiency (contact time, sorbent dosage and initial analyte concentration) were evaluated for single solution system. The optimal conditions for SiO_2–MoO_3 sorbent indicated high affinity toward Pb(II) ions within following process parameters; pH value 3, sorbent dosage 50 mg and contact time 60 min for maximum Pb^{2+} concentration of 100 mg/L. The adsorption data for Pb(II) utilizing SiO_2–MoO_3 best fit to pseudo-second-order kinetic model (R^2 = 0.9998) and Langmuir isotherm model (R^2 = 0.9320) with an adsorption capacity of 222.20 mg/g and R_{eff} >95%. According to thermodynamic study, the adsorption process is feasible, spontaneous and exothermic ($\Delta G = -18.6$ kJ/mol, $\Delta H = -7.5$ kJ/mol and $\Delta S = 37.32$ J/mol K). The results of present study demonstrated that SiO_2–MoO_3 material as a renewable sorbent for Pb(II) removal.

1. Introduction

Wastewater categories differ in terms of contaminants type/nature such as pesticides, various organic dyes and pharmaceuticals etc. [1,2]. Among them, heavy metals, because of their non-degradable features are widely present in Earth's atmosphere, hydrosphere, biosphere and lithosphere. Due to general toxicity of the heavy metals and their harmful effects even at very low concentrations, these are classified as huge and serious environmental problem [3,4], which has initiated many studies aimed to solve problems on quantification, distribution, mobility and removal of these pollutants [5,6,7]. The challenges associated with the treatment of lead (Pb)-containing wastewater is important because of its ecological impact and effect on humans' health [8]. Adsorption has become one of the alternative methods and in recent years the need for new, unexplored, cheaper and more effective sorbents to solve these aforementioned problems have been increased [9,10]. In this regard, opportunities to assess the feasibility of new metal-oxide functionalized silica sorbents for heavy metal removal is still in the research domain [11,12].

Recently, molybdenum (VI) oxide-modified silica gel material was synthesized, characterized and used for the preconcentration of very low heavy metals concentrations from river water [13], however, has not been used in a batch removal system. In comparison with literature review [11,14–16] the proposed synthesis of SiO_2–MoO_3 sorbent is less demanding resulting in material rich in chemically reactive oxygen containing functional groups. The additional novelty of the research is aimed towards the examination of reducing efficiency or complete removal of Pb(II) ions from single metal (just Pb ion), as well as from multi-metal aqueous solutions (Containing Cd, Co, Cr, Cu, Fe, Mn, Ni, and Zn ions along with Pb) using SiO_2–MoO_3 sorbent in the pH range 3–5. In present study, the removal of Pb(II) was studied under various other key adsorption conditions (time, mass of sorbent, initial ion concentration and temperature) to evaluate the nature of binding mechanisms of Pb(II) onto used sorbent. The affinity of the sorbent was determined based on the value of removal efficiency (%), R_{eff} (%) and adsorption capacity, q (mg/g).

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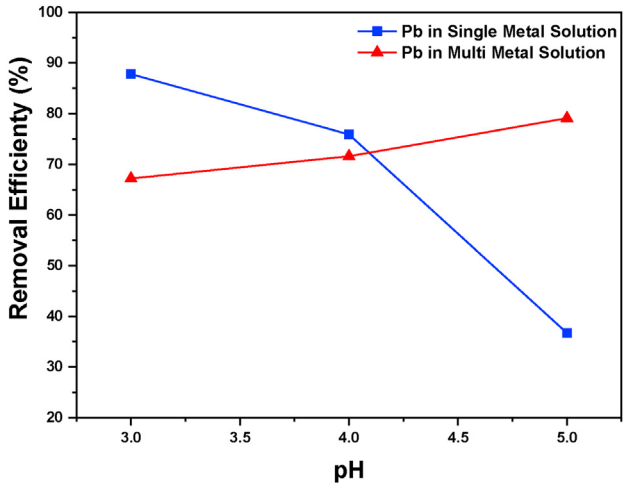

Fig. 1. Effect of initial pH on Pb^{2+} adsorption from single metal and multi-metal solutions using SiO_2–MoO_3 (n = 3).

2. Experimental

2.1. Batch adsorption experiments

Stock single-element atomic absorption standard solutions (1000 mg/L, Certipur grade, Merck, Germany) of each Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb and Zn were used. Working solutions containing different tested Pb(II) ion concentrations were prepared by dilution of mentioned stock solutions. The details about the synthesis and characterization of the silica gel modified with molybdenum(VI) oxide can be found in the previously published study [13]. A suspension of 50 mg of SiO_2–MoO_3 sorbent in 50 mL of a multi-metal solution containing 2 mg/L each of nine aforementioned metals (18 mg/L in total) was initially tested over the pH 3 to 5. After investigation of possible competitive adsorption at multi-metal solution on Pb adsorption over different pH values. Other key parameters influencing adsorption of Pb onto SiO_2–MoO_3 were tested in a single-Pb^{2+} metal (non-competitive) solution that included optimization of contact time, mass of the sorbent, initial concentration of Pb(II), capacity as well as the reusability of SiO_2–MoO_3 sorbent. The quantification of metal contents after adsorption process was carried out by Varian SpectrAA model 240FS flame atomic absorption Spectrometer. The removal efficiency (%), R_{eff} (%) of lead ions was calculated according to Eq. (1); while the sorption capacity, q (mg/g) was calculated according to Vanderborght and Van Griekenm [17], Eq. (2):

$$R_{eff} = \frac{c_i - c_f}{c_i} \mathrm{n}100 \quad (1)$$

$$q = \frac{(c_i - c_f)\mathrm{n}V}{m} \quad (2)$$

where c_i and c_f are initial and final concentrations of metal ion (mg/L) respectively, V represents the volume of solution (L) and m refers to the mass of sorbent (g). To understand the biosorption mechanism of Pb(II) ions onto SiO_2–MoO_3, linearized equations of Langmuir, Freundlich and Temkin isotherm models as well as pseudo-first-order, pseudo-second-order and intraparticle diffusion kinetic models were applied. In order to evaluate the applicability of each named model to the obtained experimental data, Chi square test (X^2), normalized deviation (ND) and the sum of squares error (SSE) were applied. The aforementioned parameters were calculated according to the expressions given by Šabanović et al. [18] and Sehar et al. [19]. Furthermore, thermodynamic parameters, ΔH°, ΔS° and ΔG° were calculated according to Nuhanović et al. [20].

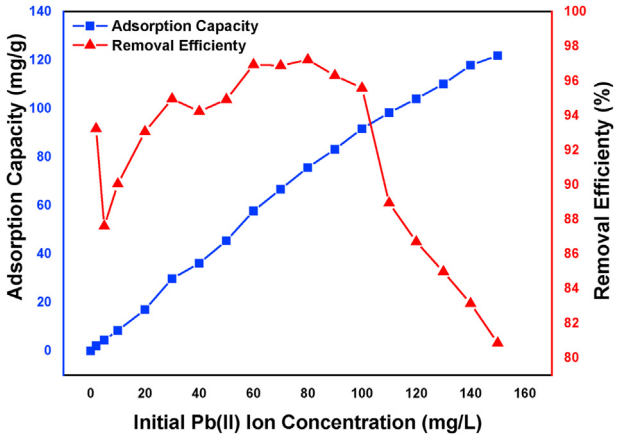

Fig. 2. The effect of initial Pb(II) ion concentration on adsorption capacity and removal efficiency using SiO_2–MoO_3 (Conditions: pH 3, contact time 60 min under 250 rpm, sorbent mass 50 mg and sample solution 50 mL).

3. Results and discussion

3.1. Effect of pH

Initial pH of multi-metal competitive (Cd^{2+}, Co^{2+}, Cu^{2+}, Cr^{3+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, Pb^{2+} and Zn^{2+}) and single-metal noncompetitive solution (Pb^{2+}) was varied from 3 to 5 according to the batch procedure and obtained results are shown in Fig. 1.

The most significant divergence of lead ion percentage removal efficiency was observed at pH 5: 79.1% (multi-metal solution) and 36.7% (single Pb^{2+} solution). The apparent "increase" of R_{eff} value (specifically at pH 5) due to possible co-precipitation of Pb(II) ions at iron(III) hydroxide caused a decrease in Pb(II) ion concentration resulting in higher obtained R_{eff} value. High R_{eff} at pH 3, for both solutions could contribute towards better driving force that causes better diffusion to the binding sites over the sorbent surface. Considering presented results, it can be concluded that SiO_2–MoO_3 sorbent is suitable for Pb^{2+} ion removal from both, multi-metal solution as well as single solution at pH 3 and 4, while pH 5 should be avoided due to significant co-precipitation of Pb onto Fe(III) hydroxide precipitate.

3.2. Kinetic studies

Kinetic experiments were conducted at 5, 10, 20, 30, 40, 50, 60, 90, 120 and 180 min (n = 3). Initial sorption of Pb(II) ion onto SiO_2–MoO_3 sorbent was rapid after only 5 min (R_{eff} = 89.72%, q = 43.07 mg/g) followed by slow stabilization and equilibrium at 60 min (R_{eff} = 99.17%, q = 47.60 mg/g). Pseudo-second-order model kinetic parameters indicated its applicability since the experimental value of adsorption (47.78 mg/g) was found to be very close to the model predicted value (48.07 mg/g) as well as high linearity agreement recording correlation coefficient (R^2) and constant (k_2) values of 0.9998 and 0.0148 g/mg min, respectively. Additionally, R^2 for intraparticle diffusion model were 0.8593 (I phase), 0.7567 (II phase) and 0.9693 (III phase) indicating that Pb(II) ions adsorption was more present on the pore surface of the sorbent (phase III) as well as by external diffusion and exchange with easily exchangeable ions (phase I), while exchange with more difficulty exchangeable ions (phase II) was negligible. Furthermore, low values of X^2, ND and SSE of statistical analysis (0.0017; 0.0551 and 0.026 respectively) supported and validated pseudo-second-order model based on chemisorption as best to describe the adsorption process of Pb(II) ion onto SiO_2–MoO_3 surface.

Table 1
Parameters of Langmuir, Freundlich and Temkin isotherm models for Pb(II) sorption onto SiO_2–MoO_3 ($q_{e,exp}$ = 121.94 mg/g).

Langmuir				Freundlich			Temkin		
q_{max} (mg/g)	K_L (dm^3/g)	R^2	R_L	K_F (mg/g) (dm^3/mg)$^{1/n}$	R^2	1/n	A_T	RT/b	R^2
222.20	0.06	0.932	0.893	15.49	0.783	0.841	2.947	27.26	0.822

Table 2
Comparison of solution pH and Langmuir adsorption capacity values.

Sorbent	q_{max} (mg/g)	pH	Reference
Imprinted silica sorbent	61.90	4.5	[14]
SiO_2/lignin	89.02	5.0	[15]
SiO_2–Al_2O_3	37.03	8.0	[16]
CuO nanosorbent	125.00	6.5	[21]
Crystalline mesoporous ZrO_2–Al_2O_3	110.49	5.0	[22]
Graphene oxide-MgO	196.1	6.5	[23]
Pb(II) imprinted carbon nanofiber	47.00	6.0	[24]
Fe_3O_4@DHAQ_GO nanocomposite	239.4	5.5	[25]
CeO_2–ZrO_2 coated ACFs	61.54	6.5	[26]
Polypyrrole-based activated carbon	50.00	5.5	[27]
SiO_2–MoO_3	**222.20**	**3.0**	**This work**

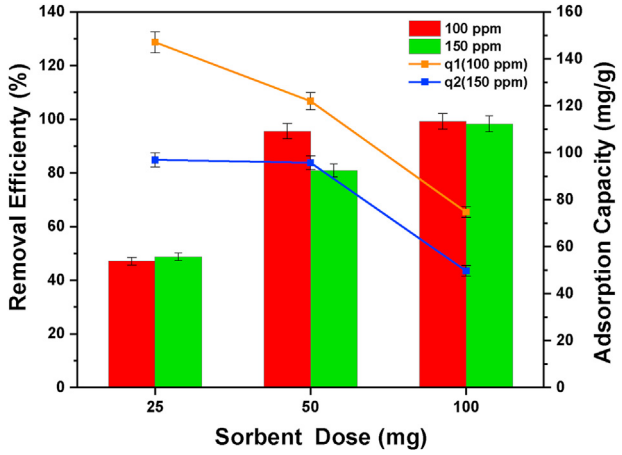

Fig. 3. The effect of sorbent dosage on the adsorption of Pb(II) on SiO_2–MoO_3 at pH 3, contact time 60 min under 250 rpm and with a sample volume of 50 mL.

3.3. Adsorption isotherms

According to Fig. 2, when the initial Pb(II) concentration was increased from 2 to 150 mg/L, the adsorption capacity increased from 2.16 to 121.94 mg/g, but at the same time, the removal efficiency (%) decreased from 93.25 to 80.86%. At lower concentrations up to 100 mg/L, the metal ion could be quantitatively adsorbed on the abundantly available active sites. However, with higher concentrations of Pb(II), a saturation of binding sites occurred resulting in R< 90%. To infer the adsorption effect mechanism by characterizing the adsorption behaviour of SiO_2–MoO_3, different adsorption isotherm tests were performed and results are presented in Table 1. The experimental results fit best to the Langmuir isotherm model based on higher correlation coefficient (R^2 = 0.932).

Langmuir model suggests monolayer Pb(II) uptake without interaction between adsorbed species with a maximum capacity of 222.20 mg/g. Calculated values of R_L confirmed the favorability of the present process. Observed Pb(II) sorption capacity of SiO_2–MoO_3 was found to be higher or comparable to other similar sorbents (Table 2) except for Fe_3O_4@DHAQ_GO nanocomposite. Additionally, SiO_2–MoO_3 sorbent has a stronger affinity to Pb(II) ions in acidic medium, regarding other mentioned sorbents, which is more favourable for the treatment of specific industrial wastewaters based on the high concentration of heavy metals.

Table 3
The thermodynamic parameters of adsorption with 150 mg/L Pb(II) onto SiO_2–MoO_3 (50 mg).

Temperature (K)	q (mg/g)	R_{eff} (%)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol.K)
296.15	121.93	80.90	−18.60	−7.50	37.32
306.65	123.40	81.90	−19.00		
313.15	125.40	83.40	−19.20		
323.15	126.30	84.10	−19.70		

Additionally, regarding lead adsorption, 100 mg of SiO_2–MoO_3 could be successfully used in both column [13] and batch method applied to a concentration range from traces to 150 mg/L based on the high values of preconcentration factor (column: PF = 25, Recovery>95%) and removal factor (batch: R_{eff}>95%).

3.4. Effect of SiO_2–MoO_3 dosage

The increase of sorbent dosage has increased removal efficiency and decreased sorption capacity for both tested initial Pb(II) ion concentrations (Fig. 3).

The optimal dosage for quantitative sorption of 100 mg/L (including lower concentrations) of Pb(II) ions was 50 mg (R>95%), while quantitative sorption (R_{eff} = 98.3%) of 150 mg/L Pb(II) ion was obtained by 100 mg of solid phase.

3.5. Effect of temperature and thermodynamic parameters

According to the results presented in Table 3, the removal of Pb(II) using SiO_2–MoO_3 is not significantly temperature dependent with obtained R_{eff} values > 80%. Table 3 also shows that the increase in temperature caused a decrease in ΔG values, indicating that the adsorption process is feasible and spontaneous, as well as exothermic (-ΔH°). Furthermore, the positive value of ΔS° indicates an increase in the randomness at the adsorbate/solution interface during lead (II) adsorption.

3.6. Regeneration and reuse of sorbent

In the present study, 15 probes were performed to extract Pb(II) ion from the solution to the sorbents surface under optimal conditions (pH = 3, 100 mg/L of Pb(II), during 60 min with 50 mg of sorbent). Afterwards, the sorbent was separated from the aqueous phase, soaked 2 h in 10 mL of 1.0 mol/L HNO_3 washed by distilled water then dried at 80 °C for 24 h and used for new similar sorption cycle. At the second cycle, the R_{eff} decreased to 82.60% corresponding to 82.76 mg/g adsorption capacity. The value remained almost constant for the other two successive adsorption-desorption cycles.

4. Conclusion

Sorption equilibrium was established for 60 min at pH 3 for single Pb(II) solution system while pH 5 was more suitable for multi-metal solution. The pseudo-second-order kinetic model and Langmuir isotherm model exhibited an excellent correlation with the experimental data for single solution of Pb(II). The maximum adsorption capacity of SiO_2–MoO_3 for Pb(II) ions was found to be 222.20 mg/g that was higher

or comparable to other similar sorbents. Furthermore, SiO_2–MoO_3 sorbent could be regenerated with 1.0 mol/L HNO_3 and reused for four times. A follow up study is strongly recommended on the adsorption efficiency of SiO_2–MoO_3 sorbent for other heavy metals since promising and significant data were obtained for Pb(II) removal or especially toward applications in pilot plants and wastewater treatment of real samples for Pb(II) removal.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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