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Removal of lead ion from aqueous solutions using nanogel of sesame waste

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ABSTRACT

Increasing industrial activities of humans lead to the entry of heavy metals in to the environment. Heavy metals are important pollutants of natural water and treated water and a serious problem for the health of humans and other living being. Among heavy metals, lead (Pb) is of particular importance. The use of adsorbent is an effective technology for the removal of heavy metals. The purpose of this study was to use nanogels prepared from sesame residue to remove lead (Pb) from aqueous solutions and to investigate the effect of the pH, contact time, initial concentration and absorbent dosage. This research was conducted on laboratory scale and a batch system. The isotherms of experimental data was evaluated by Langmuir and Freundlich models. According to the results of this study, the highest lead (Pb) adsorption efficiency was 93.45% at pH 6 and time 90 min. Results showed that lead (Pb) absorption follows Langmuir isotherm and second-order kinetic, and the maximum absorption capacity (Q_{max}) was13.831 mg/g. Due to unique features of nanogels made from sesame waste like, availability, high water adsorption capacity, being cheap, has good ability to remove heavy metals from aqueous solutions.

Keywords: Adsorption; Aqueous solutions; Lead; Sesame waste

1. Introduction

Water is one of the first necessities of life and one of the most useful materials of nature, which is vital for human life and civilization. Nowadays, the supply of healthy, safe and satisfactory drinking water is considered as one of the main factors in maintaining the health and development of communities' economies, and the importance of this vital fluid in human life is so great that it is sometimes difficult to fully express and justify [1]. Heavy metals are the natural components of the earth's crust. But human activities, geochemical and biochemical cycles upset the balance of these metals and cause their release into the environment. The presence of natural and artificial pollutants and factors affecting the spread of pollution is the basis of all environmental pollution with this type of metal [2]. Among heavy metals, lead (Pb) is of particular importance. Lead (Pb) is a metal that can be piped, hammered, cracked and has a

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high resistance to erosion. After iron, it is the second most widely used industrial metal, which is produced from ore [3]. It is one of the most important heavy and toxic elements that enters the body through water and food and through respiration. Once ingested, the metal is converted to biotoxic compounds by the body's bio-molecules such as proteins and enzymes. As a result, their structure changes and their biological interactions become problematic. Lead (Pb) contamination prevents the synthesis of hemoglobin, damage to the function of the kidneys, joints and cardiovascular system [4,5]. Industrial and research centers show that the treatment field is one of the most widely used areas of nanotechnology in the water industry, which, by using it, water treatment costs are significantly reduced. The problem of removing heavy metals from effluents is one of the problems that is hoped to be solved with the help of nanotechnology. In order to solve this problem, one of the ideas in the field of nanotechnology that can be suggested as an efficient method to remove heavy metal cations from the aqueous environment, is the use of nanoparticles as adsorbents of these cations [6]. Today, due to some of the advantages of using agricultural waste in water treatment such as simple technique, short process, high adsorption capacity, low price, almost free access and easy recovery, extensive research has been done on plant adsorption [7]. Nanogels are particles with a three-dimensional network that are formed by crosslinking polymer chains by chemical or physical crosslinking agents. Nanogels have good transfer properties and excellent penetration capacity due to their small particle size. Nanogels also have good composition, high biocompatibility and biodegradability [8]. Various industries have diverted the lives of many living organisms to water resources. Various researchers have used the adsorption process to remove various contaminants, including organic substances, dyes and drugs [9–12]. Therefore, after that, researchers have studied to remove pollutants from water sources [13]. Sayadi et al. [14] used nano-silicate activated with rice husk to remove chromium and copper. The influence of various factors such as the amount of adsorbent, initial concentration and equilibrium of chromium and copper and contact time on the removal of heavy metals copper and chromium were tested. The results of removal of contaminants showed that the maximum adsorption of chromium and copper at the optimal time was 100 min while the optimal amount of adsorbent for chromium and copper was 125 and 100 mg, respectively. The optimal conditions were the highest amount of chromium and copper adsorption at a concentration of 2 mg/L. The adsorption isotherms of chromium and copper follow the Langmuir model [14]. Mousa et al. [15] used rice paddy to remove the heavy metals lead (Pb) and cadmium (Cd). The results showed that lead (Pb) metal, like cadmium, has the best equilibrium time of 90 min and the best pH of 5. Lin et al. [16] used modified cellulose as an adsorbent to adsorb hexavalent chromium from aqueous solutions. Experiments were performed to evaluate the effect of initial pH and initial chromium concentration on adsorption performance. The optimum pH for adsorption of hexavalent chromium was in the range of 2–3 and the maximum adsorption of hexavalent chromium from 500 mg/g solution at pH = 3 and temperature was 50°C. The Langmuir and Freundlich isotherms were used in the adsorption process and the thermodynamic parameters were calculated. Kinetic studies showed that the experimental data are in good agreement with the pseudo-second-order kinetics and the adsorption process is possible. The aim of this research, has been investigated the removal of lead (Pb) ions dissolved in aqueous media on nanogels prepared from sesame waste.

2. Materials and methods

2.1. Equipment and devices

The equipment and devices used are:

- Electronic balance model LT3002T made in China for weighing metals salt.
- Universal model centrifuge at a speed of 4,000 rpm, for 5 min to separate suspended particles from the solution.
- Raymand Co. shaker device for mixing adsorbent and absorbent at different times at a speed of 400 rpm.
- pH meter model AZ-86502 made by AZ Taiwan Company to measure pH.
- Atomic Absorption Unit Model Unicam-919 Made in England to obtain the final equilibrium concentration.
- The X-ray diffraction spectroscopy device, model x Pertpro, is made by the Dutch company Analytical to study the structure of the absorber.
- Fourier-transform infrared spectroscopy (FTIR) device model RX1 Spectrum made by American Analytical Company to identify organic compounds and functional groups and determine the groups involved in the adsorption of heavy metals.
- Multi-mode atomic force microscope (ARA-AFM) to observe the physical shape of the surface of adsorbent samples and shape.

2.2. Used materials

The materials used include lead nitrate ($Pb(NO_3)_2$) for preparing stock solution, HCl and NaOH for set of pH.

Nanogels prepared from sesame waste were purchased from Nano Novin Polymer Company in Sari (Mazandaran Province of Iran).

2.3. Absorbent characteristics

To determine the adsorbent characteristics X-ray diffraction (XRD) spectra, AFM and FTIR were taken. The XRD peaks in the range of 2θ times showed 15, 16, 22.5 and 35. These peaks indicate that the adsorbent is irregular and indicate that the resulting material is only adsorbent and no other impurities have been observed. In addition, the peak in 22.5 is related to the crystal structure of the adsorbent, which indicates its high degree of crystallinity due to internal and intermolecular hydrogen bonds. Fig. 1 shows the XRD image of the adsorbent.

Atomic force microscopy was used to observe the morphology and physical shape of the surface of the prepared adsorbent sample, which is shown in Fig. 2.

FTIR analysis was used to determine the effective groups on adsorption. Fig. 3 shows the FTIR spectra of gels

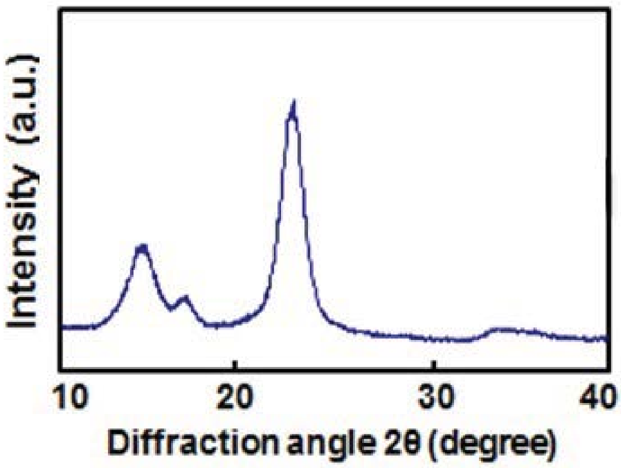

Fig. 1. X-ray diffraction (XRD) analysis of nanogels prepared from sesame waste.

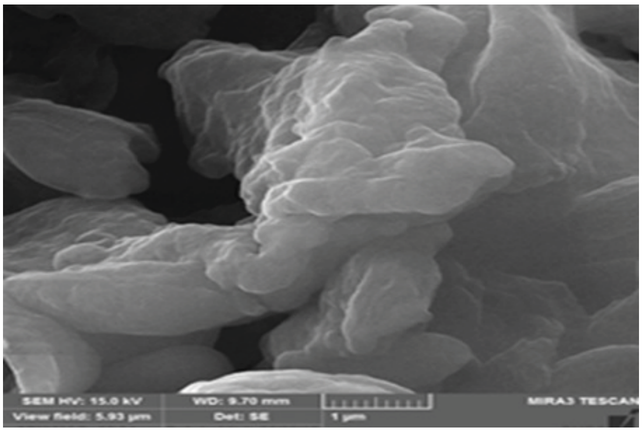

Fig. 2. Microscopic image of nanogels prepared from sesame waste.

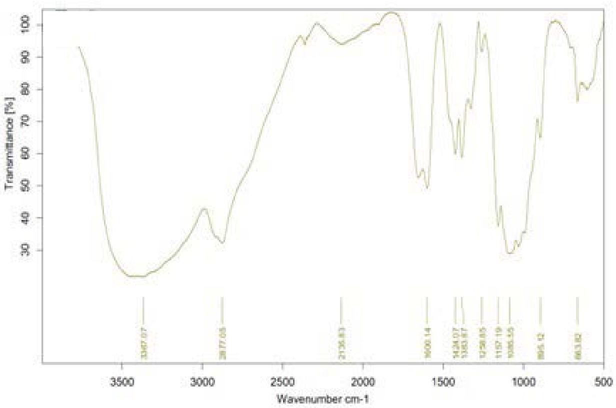

Fig. 3. FTIR nanogels prepared from sesame waste.

prepared from pre-adsorption sesame waste. According to Fig. 3, the 3,500 cm^{-1} wide peak belongs to the functional group O–H and N–H. The 2,900 cm^{-1} peak can be related to C–H vibration. The next strong band 1,600 cm^{-1} can be considered as the C=O tensile vibration in carboxylic. The peak 1,000 cm^{-1} also belongs to the bending C–O factor group.

2.4. Preparation of solutions containing metal ions

This study was an experimental study and all adsorption experiments were performed in a batch system in which the performance of nanogels prepared from sesame waste as an adsorbent in the removal of lead (Pb) from aqueous solutions on a laboratory scale was studied.

The effects of pH, initial metal ion concentration, adsorbent dose and contact time on the adsorption rate were investigated and the adsorption isotherm was evaluated using the Langmuir and Freundlich adsorption isotherm models. The sample volume used for the experiments was 100 mL.

In this study, nanogels prepared from sesame waste were prepared from Nano Novin Polymer Company of Sari. The standard solution was 1,000 mg/L of lead nitrate ($Pb(NO_3)_2$) salt. Other required solutions were prepared by diluting the stock solution with deionized water. To prepare a solution of 1,000 mg/L, 1.59 g of lead nitrate salt was dissolved in deionized distilled water and brought to a volume of 1,000 mL. For 0.1 normal solutions of sodium hydroxide and hydrochloric acid were used to adjust the pH.

To determine the optimal pH at this stage, the pH is variable and other parameters are constant. The studied heavy metal solution was prepared separately with an initial concentration of 20 mg/L. For 0.1 M HCL and NaOH solutions were used to adjust the pHs of 3, 4, 5, 6, 7 and 8. To the dish, 0.1 g of nanogel adsorbent prepared from sesame waste was added, then the pH determined solutions. The solutions were placed on a Raymand Co., shaker for 60 min at 100 rpm, then the solutions were placed in a centrifuge set at 4,000 rpm for 5 min. The supernatant was then separated and its concentration was read by atomic absorption spectrometer. To determine the optimal adsorbent dose at this stage, a solution of the studied metal (lead) with an initial concentration of 20 mg/L was prepared. Different values (0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g to each of the determined pH solutions). The periods of time are 15, 30, 45, 60, 75 and 90 min. The solution of heavy metal (lead) with an initial concentration 10, 20, 30, 40 and 50, 100 mg/L was prepared.

Eqs. (1) and (2) were used to determine the percentage of lead (Pb) removal in equilibrium and to determine the lead adsorption capacity of lead metal by nanogels prepared from sesame waste [17].

$$R\% = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (2)$$

where R is the removal percentage, where, q_e the adsorption capacity of the equilibrium in each sample in mg/g, C_0 the initial concentration of the heavy metal in solution in mg/L, C_e the final equilibrium concentration in mg/L, V sample volume in L, M is adsorbent mass in grams [17].

2.5. Adsorption isotherms

Adsorption isotherms are mathematical equations that show the relationship between the amount of adsorption of an ion by a solid phase and its concentration in equilibrium solution at constant temperature and as tools for describing and predicting the amount of adsorption, type and intensity of interaction. They are absorbent and absorbate. In the present study, Langmuir and Freundlich models were used to analyze the experimental data and describe the equilibrium state in the adsorption between solid and liquid phases [18].

2.5.1. Langmuir isotherm

The Langmuir adsorption isotherm model is related to single-layer adsorption and it is assumed that bio-sorption occurs in special (uniform) sites. The general form of the Langmuir equation is given in Eq. (3):

$$q_e = \frac{q_m b C_e}{1 + bC_e} \tag{3}$$

The Langmuir relation can be arranged as Eq. (4):

$$\frac{1}{q_{\max}} C_e + \frac{1}{bq_{\max}} \tag{4}$$

where C_e is the equilibrium concentration of metal ions in mg/L, q_e is the amount of ions adsorbed in mg/g, q_m is the maximum adsorption capacity of metal ions in mg/g, b is the equilibrium constant of Langmuir adsorption in mg/mg [19].

2.5.2. Freundlich isotherm

Freundlich's experimental relationship is based on multilayer adsorption on non-ionic surfaces and inhomogeneous energy distribution on active adsorbent sites. The general form of the equation is Eq. (5):

$$q_e = K_f C_e^{1/n} \tag{5}$$

where C_e is the equilibrium concentration of metal ions in mg/L, q_e are the amount of ions adsorbed in mg/g, K_f and n are Freundlich constants, and capacity and adsorption indices, respectively [19].

Eq. (6) can be used to obtain the Freundlich linear relation:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{6}$$

2.6. Absorption kinetics

In the present study, the most commonly used pseudo-first and pseudo-second-order kinetic models were used to investigate the factors affecting the reaction rate of the lead adsorption process on a nanogel adsorbent prepared from sesame waste.

2.6.1. Pseudo-first-order kinetic equation

$$\ln(q_e - q_t) = \ln q_e - K_1 t \tag{7}$$

where q_e is the amount of mg absorbed per unit of adsorbent mass in equilibrium, q_t is the amount of mg adsorbed per unit of adsorbent mass at time t, k_1 is the constant rate of the adsorption process.

2.6.2. Pseudo-second-order kinetic equation

$$\frac{t}{q_t} = \frac{1}{(k_2 q_e^2)} - \frac{t}{q_e} \tag{8}$$

where k_2 is the constant of the adsorption process speed [18].

2.7. Data analysis

All experiments were repeated three times and the mean data and results were used. Excel and SPSS (20) software were used to draw the diagram and analyze the data.

3. Results

In this section, the parameters affecting the removal process including pH, adsorbent dose, contact time and initial concentration will be studied.

3.1. Effect of pH on lead removal

The results of investigating the effect of solution pH on the adsorption rate of metal ions are shown in Fig 4. By increasing the pH of the solution from 3 to 6, the removal process and adsorption capacity increased and at pH 6 the highest removal percentage and lead adsorption capacity of 93.45% was obtained and then took a decreasing trend so that in pH 8, removal percentage and lead adsorption capacity was 84.05.

3.2. Effect of adsorbent dose on Lead (Pb) removal

The effect of adsorbent on lead (Pb) adsorption is shown in Fig. 5. The results show that the amount of lead (Pb) removed increases with increasing the amount of

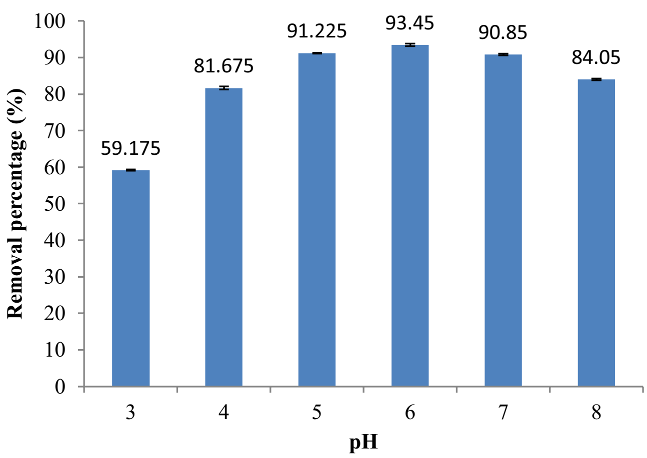

Fig. 4. Effect of pH on lead removal percentage.

adsorbent from 0.1 to 0.6 g and to it reaches 97.275% and also the adsorption capacity increases with increasing the adsorbent dose from 0.1 to 0.6, so that at 0.6 g the adsorption capacity is equal to 19.455 mg/g.

3.3. Effect of contact time on lead removal rate

The effect of lead (Pb) contact time on the removal process efficiency and adsorption capacity is shown in Fig 6. The results showed that with increasing contact time, removal percentage and adsorption capacity increase. In the 90 min removal time, 93.9% adsorption of lead (Pb) were obtained.

3.4. Effect of initial lead concentration on removal rate

The effects of changes in initial lead (Pb) concentration on removal percentage and adsorption capacity by nanogel prepared from sesame waste are shown in Fig. 7. According to Fig. 4, with increasing initial lead (Pb) concentration, the removal percentage and the adsorption capacity were decreases. So that at a concentration of 10 mg/L, the percentage of lead (Pb) removal is equal to 95.975% and with increasing the initial concentration and reaching a concentration of 100 mg/L, the percentage of lead (Pb) removal reaches 72.625. Also, the adsorption capacity increases with the initial concentration.

3.5. Effect of initial lead concentration on removal rate

The effects of temperature on removal percentage and adsorption capacity by nanogel prepared from sesame waste are shown in Fig. 5. According to Fig. 8, with increasing temperature, the removal percentage and the adsorption capacity were increase.

3.6. Adsorption isotherms

The results of isotherm studies are presented in Table 1.

Table 1
Adsorption isotherms

Isotherm	Langmuir			Freundlich		
Parameter	q_m (mq/q)	b	R^2	K_f	n	R^2
Pb	13.83	2.51	0.995	19.35	6.8	0.903

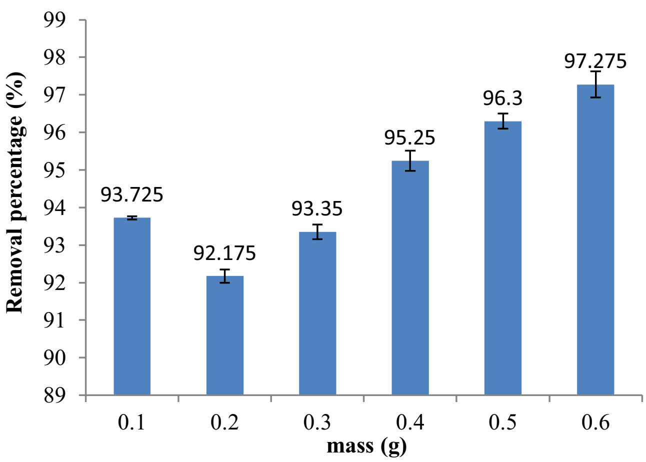

Fig. 5. Effect of absorbent mass on lead removal percentage.

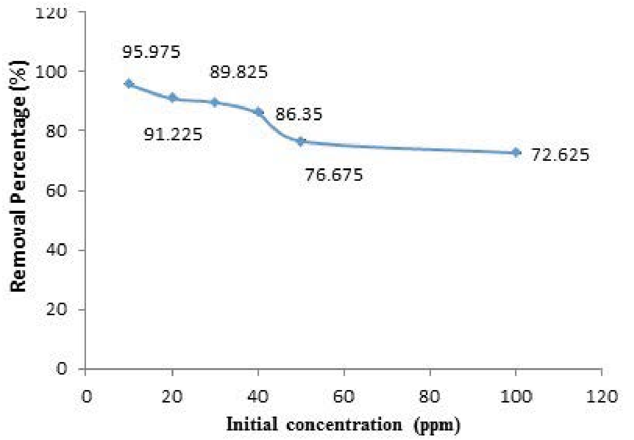

Fig. 7. Effect of initial concentration mass on lead removal percentage.

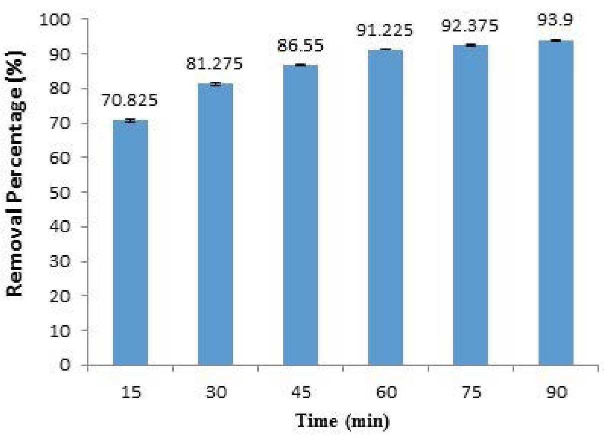

Fig. 6. Effect of contact time on lead removal percentage.

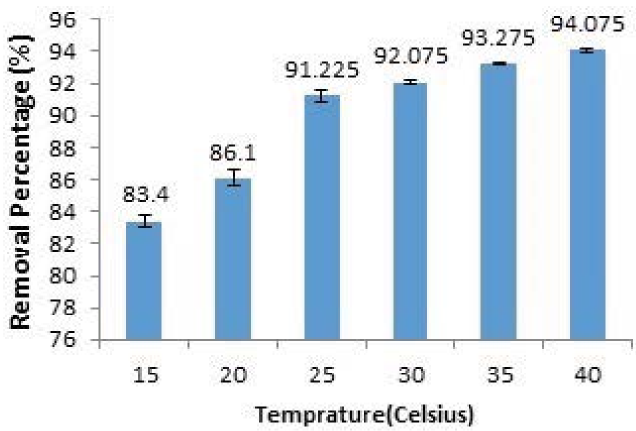

Fig. 8. Effect of temperature mass on lead removal percentage.

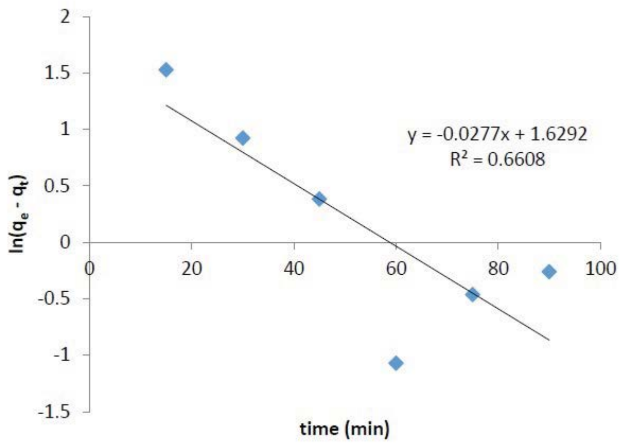

Fig. 9. Pseudo-first-order synthetic model curve for lead.

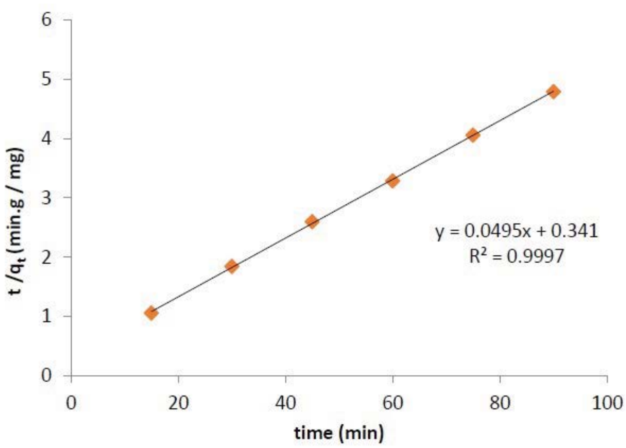

Fig. 10. Pseudo-second-order synthetic model curve for lead.

3.7. Adsorption kinetics

The results of pseudo-first-order kinetics and pseudo-second-order kinetics are presented in Figs. 9 and 10.

4. Discussion

One of the important parameters in the adsorption process is the initial pH of the solution. The pH of the solution plays an essential role in determining the concentration of cation species in the solution [20]. The optimum pH for the removal of the heavy metal lead in the synthetic solution was 6. The reason for this is the high efficiency of adsorption of bio-sorbents in acidic environments, in which case metal ions are well absorbed on the specified adsorbent bands that have H^+. Examining other studies, it is observed that in most cases, the maximum removal of lead occurred in the range of 5–6 (acidic), which is consistent with the results of the present study. In Islam et al. [21] study "Pre-concentration of metal ions through chelation on a synthesized resin containing O, O donor atoms for quantitative analysis of environmental and biological samples", he optimum pH range for the maximum sorption of Ni(II), Mn(II), Cu(II), Zn(II), Cd(II), Cr(III), and Co(II) was observed at pH 5.5–8.0. It was consistent with the results of research conducted by Abdel-Ghani and Elchaghaby et al. [22] on the removal of lead on rice husk and indigo rose and Zafarzadeh et al. [5] on the removal of lead from the synthetic solution by citrus cool and activated carbon [5,22]. Increasing the pH increases the negative charges on the adsorbent surface, which leads to an intensification of electrostatic forces in the process of adsorption of more metal ions [5,22,23]. Increasing the adsorption efficiency by increasing the adsorbent dose, it is justified that at high doses, lead ions are more likely to be trapped inside the adsorbent and in fact the available surface for adsorption of metal ions is higher [24]. Reaction time is one of the conditions for creating a suitable environment for the removal of heavy metals. In the present study, lead removal in a period of 15–90 min was investigated which increases with increasing removal time. Numerous similar studies have been performed by researchers to examine contact time on lead removal rates, and the results are varied. For example, the optimal contact time in the study, (Mousa et al. [15]) 90 min (Mohammadpour et al. [18]) 30 min (Bulut and Tez [25]) is 60 min (11, 14 and 20). Lead removal decreased with increasing initial concentration. As the initial concentration increases, the available sites of the adsorbent decrease and thus the removal efficiency decreases, because at low concentrations, access to the available surfaces of the adsorbent is higher. In the present study, with increasing the initial concentration from 10 to 100, the adsorption efficiency for lead had a decreasing trend. Research conducted by Nassehinia et al. [26] showed that the removal efficiency decreases with increasing initial concentration. With increasing the initial concentration, the removal efficiency of metals decreased. This is due to the smaller ion exchange bands, which is consistent with the study conducted by Kumar et al. [27] and Abdel-Ghani and Elchaghaby et al. [22]. In Pant et al. [28] (study "Enhancement of biosorption capacity of cyanobacterial strain to remediate heavy metals", was shown maximum removal of copper (87.50%), chromium (82.96%), lead (86.44%), zinc (86.59%) from the sugar mill effluent. In this study, according to the correlation coefficients R^2, the adsorption data of this study follow the pseudo-second-order kinetic model and the equilibrium data better follow the Langmuir isotherm, in their study concluded that in the equilibrium test for lead ions, the Langmuir isotherm model is more suitable than Freundlich [24].

The result of this study same to Ahmad et al. [29] study "Chemically oxidized pineapple fruit peel for the biosorption of heavy metals from aqueous solutions", chemically oxidized pineapple fruit peel biomass has been used as a biosorbent for Cd(II) and Pb(II) removal from aqueous solutions. The biosorption efficiency of pineapple fruit peel for Cd(II) and Pb(II) was greatly enhanced after chemical oxidation probably due to introduction of carboxylic and hydroxyl groups onto the biosorbent surface. Biosorption kinetics for both metals was well described by pseudo-second-order kinetic equation and intraparticle film diffusion. Langmuir, Freundlich, and Temkin isotherm models were applied to the biosorption equilibrium data and best results were obtained with Langmuir isotherm model. Maximum monolayer biosorption capacity was found to be 42.10 and 28.55 mg/g for Cd(II) and Pb(II), respectively. Thermodynamic study indicates that the biosorption was exothermic and the spontaneity of the process decreases with the increase in solution temperature [29].

5. Conclusion

The results in this study showed that the highest adsorption efficiency at pH is equal to 6 observations. It was also observed that with increasing the initial concentration, the efficiency of the removal process decreases and increasing the contact time increases the adsorption efficiency. The Langmuir–Freundlich equilibrium data showed that lead uptake followed the Langmuir isotherm. The pseudo-second-order models are most consistent with the experimental data. In general, the results of this study showed that nanogels prepared from sesame waste have a high ability to remove lead.

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Conflict of interests

No conflict of interest has been stated by the authors.

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