



Removal of Lead Ions from Industrial Wastewater Using Magnetite Loaded on Silica Support



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A SERIES of magnetite supported silica ($\text{Fe}_3\text{O}_4/\text{SiO}_2$) nanoparticles with different Fe_3O_4 loadings was prepared by co-precipitation method. Structural characteristics of the prepared adsorbents were investigated by X-ray diffraction (XRD), and transmission electron microscope (TEM). The magnetic property was also determined. The adsorption performance of the samples was assessed in the removal of lead ions from aqueous solution. Experiments have been performed to study the effects of several parameters including solution pH, time, initial metal ion concentration and adsorbent weight on the sorption efficiency. The results of the adsorption experiments indicated that the obtained adsorbents have excellent adsorption ability for the removal of lead ions.

Keywords: Magnetite, Wastewater, Adsorption, Lead ions removal.

Introduction

The environment and all the life forms on earth face a very serious threat from the heavy metal pollution due to their toxicity and strong tendency to concentrate in environment. At recent years heavy metals are classified as toxic and half of these are emitted into the environment in quantities that pose risk to human health [1]. These toxic metals can cause accumulative poisoning, cancer, and brain damage when found above the tolerance levels [2]. Lead (II) is one of the most toxic heavy metals causing health problems when present in high amounts in water. The presence of lead in drinking water causes adverse health effects such as anemia, hepatitis and nephritic syndrome [2,3].

Water is essential to all forms of life. It is also a vital resource for agriculture, manufacturing and other human activities. Therefore, the development accurate, rapid and selective determination method for removal Pb(II) from polluted water due to its extreme toxicity and indispensable[4].

Removal of metals from wastewater principally achieved by the application of several processes such as precipitation, electroplating, chemical coagulation, ion-exchange, membrane separation and adsorption [5,6].

Recently, it was reported that using superparamagnetic (Fe_3O_4) nanoparticles as an effective adsorbent for heavy metals removal from water, may result in development of a new and efficient technology in industrial wastewater treatment. The major advantages of technology are its effectiveness in reducing the concentration of heavy metal ions such as Pb(II), avoid the generation of secondary waste, produced no contaminants, has the capability of treating large amount of wastewater within a short time, good selectivity and the adsorption materials employed in this method can be recycled and used easily on an industrial scale[4].

Development of novel and cost-effective nanomaterials for environmental remediation, pollution detection and other applications has

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attracted considerable attention. Recent advances suggest that many of the issues involving water quality could be resolved or greatly ameliorated using nanoparticles, nanofiltration or other products resulting from the development of nanotechnology [7-8].

The present work devoted to study the removal of Pb(II) from wastewater using magnetite loaded on silica at ambient temperature. The study deals with adsorption isotherms, Langmuir model and Freundlich model

Materials and Methods

Materials (chemicals).

Ferrous Chloride (FeCl_2), Provided from LOBA Chemic, Ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), provided from LOBA Chemic, Ammonium hydroxide NH_4OH 33% and Sodium silicate provided from laboratory Rasayan. All the chemicals were of analytical grade and were used without further purification.

Methods:

Preparation of adsorbent:

Preparation of SiO_2 nanoparticles support from sodium silicate:

1. Dissolve 500 gram sodium silicate in one liter of distilled water.
2. Add 1.5 M HCl drop by drop until the pH* become 6-6.5.
3. Wash the precipitate many times with distilled water. Filtrate the precipitate and dry it in the oven at 100°C for 12 hour and calcinat for 2 hour at 500°C .

*The pH of the solution was adjusted by using a pH meter (ThermoScientific Orion 2-Star Benchtop pH Meter Kit, with Refillable Glass pH Electrode).

Preparation of magnetite nanoparticles:

Magnetite nanoparticles were prepared by co-precipitation method reported earlier [9,10]. This method may be the most promising one because of its simplicity and productivity. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ were dissolved in a 2:1 molar ratio in distilled water with stirring under nitrogen and this solution which contains both ions was then heated up to 80°C . After heating, Ammonium hydroxide was added drop-wise to raise the pH up to 8.0 with continuous stirring at 80°C for 30 minute. At this pH the solution turned from brown to black. Subsequently, black particles were separated and quickly washed three times with distilled water. The magnetite was then dried in hot air oven at 100°C for 24 hours. [10,11].

Preparation of a series of SiO_2 supported magnetite ($\text{Fe}_3\text{O}_4/\text{SiO}_2$) with different ratios

Silica support powder was simply added into the mixed solution of ferric chloride and ferrous chloride with stirring under nitrogen before the addition of ammonia solution. The process followed the same steps as the preparation of Fe_3O_4 . The magnetite was supported by silica at different ratios, that is to say; 7.5%, 15%, 30% $\text{Fe}_3\text{O}_4/\text{SiO}_2$.

Preparation of lead ion solution (adsorbate):

A stock solution of Pb(II) (1000 mg/l) was prepared. Different concentrations of Pb(II) were prepared by dilution from this stock by distilled water, to be utilized for adsorption experiments.

Characterization of magnetite:

Perkin-Elmer 2380 atomic absorption spectrometer (AAS) was used for the determination of lead using a flame type air/acetylene. Philips 1390 X-ray powder diffractometer was used for structural identification (XRD), Transmission electron microscopy (TEM) images were taken using TEM model EM10 Zeiss instrument with 40000 magnification and 60kV power.

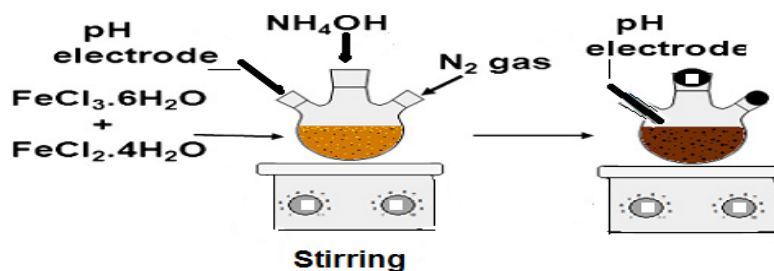


Fig. 1. Preparation of magnetite (Fe_3O_4) by co-precipitation method.

Adsorption of lead ions:

Batch adsorption experiments of the lead ions by $\text{Fe}_3\text{O}_4/\text{SiO}_2$ adsorbents were carried out at room temperature by shaking a series of bottles each containing the desired quantity of the adsorbent in a predetermined concentration of lead ion solutions. Samples were withdrawn at different time intervals; the supernatant was separated and analyzed for remaining heavy metal content. The percent removal of heavy metal from solution was calculated by the following equation [12]:

$$\% \text{Adsorption} = \frac{C_o - C_e}{C_o} \times 100$$

where; C_o is initial concentration of heavy metal, C_e is final concentration of heavy metal.

Adsorption isotherms:

The adsorption isotherm that describes the adsorption pattern between the Pb(II) adsorbed metal ions on the nano $\text{Fe}_3\text{O}_4/\text{SiO}_2$ adsorbent and the residual metal ions in the solution during the surface adsorption was conducted. Equilibrium isotherms are measured to determine the capacity of the adsorbent for metal ions. The most common types of models describing this type of system are the Langmuir and Freundlich models [13]. The adsorption capacity q_e (mg/g) after equilibrium was calculated by a mass balance relationship equation as follows:

$$q_e = (C_o - C_e) \frac{V}{W}$$

Where C_o is the initial and C_e is the equilibrium concentrations of the test solution (mg/L), V is the volume of the solution (L) and W is the mass of adsorbent (g).

Langmuir model

Langmuir adsorption model is based on the assumption that the maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. Langmuir equation can be described by the linearized form [14,15]:

$$\frac{1}{q_e} = \left(\frac{1}{q_m K_L} \right) \left(\frac{1}{C_e} \right) + \frac{1}{q_m}$$

Where, C_e is the equilibrium concentration of metal ions in solution (mg/L), q_e is the amount of metal ion adsorbed on adsorbents (mg/g), and q_m and K_L are the monolayer adsorption capacity (mg/g) and Langmuir equilibrium constant (L/mg) which indicates the nature of adsorption, respectively. The values of q_m and K_L were determined graphically. The plot of $\frac{1}{q_e}$ versus,

yields a straight line having slope equal to $\frac{1}{q_m K_L}$ and intercept q_m , which corresponds to complete monolayer coverage.

Freundlich model

Freundlich adsorption isotherm represents the relationship between the amount of metal adsorbed per unit mass of the adsorbent q_e and the concentration of the metal in solution at equilibrium. Freundlich equation can be described by the linearized form [16];

$$\text{Log } q_e = \text{log } K_F + \frac{1}{n} \text{log } C_e$$

Where k_f and n are Freundlich constants. The values of k_f and n were determined graphically. A plot of $\text{log } q_e$ versus $\text{log } C_e$ gives a straight line of slope $\frac{1}{n}$ and the intercept is $\text{log } K_F$. The value of K_F indicates the adsorption capacity while $\frac{1}{n}$ is indicative of the intensity of the reaction.

Results and Discussion*Characterization of magnetic iron oxide:**X-ray diffraction (XRD).*

Purity and crystalline structures of SiO_2 support, unsupported Fe_3O_4 and SiO_2 supported Fe_3O_4 with different ratios were examined using powder X-ray diffraction (XRD), Fig. 2 shows that the broad background peak around 22° is due to poorly crystallized SiO_2 and shows the formation of Fe_3O_4 , based on the comparison of their XRD patterns with the standard pattern of Fe_3O_4 (JCPDS 75-0033). The diffraction peaks corresponding to (220), (311), (400), (511), (440) are quite identical to characteristic peaks of the Fe_3O_4 crystal with the cubic spinel structure [17-19].

Transmission electron microscopy (TEM).

Transmission electron microscope (TEM) was used to examine the supported and unsupported Fe_3O_4 . The TEM micrographs are shown in Fig. 3. From this figure, the used silica is porous and detectable changes in the surface morphology are observed when the silica was coated with magnetite. This technique is a very useful tool for detecting and differentiating between different ratios of magnetite which was loaded on silica support and it can be seen that Fe_3O_4 nanoparticles loaded on silica were agglomerated in the form of spots increased by increasing the percentage of magnetite loaded. The pure magnetite appeared

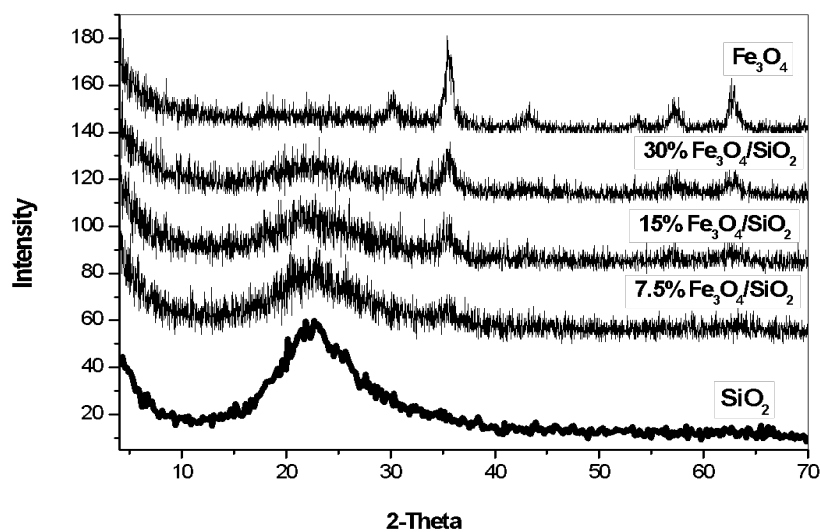


Fig. 2. X-ray diffraction patterns of SiO₂ support, unsupported Fe₃O₄ and SiO₂ supported Fe₃O₄ with different ratios.

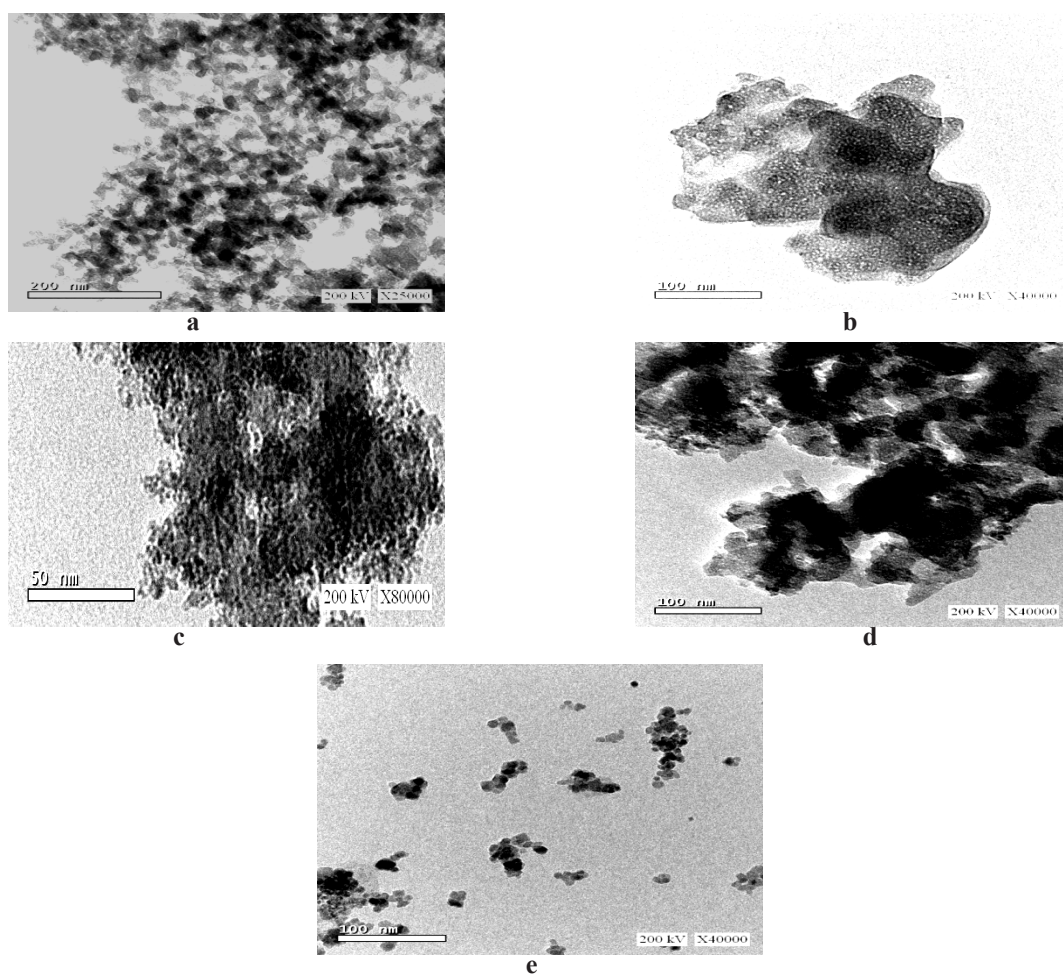


Fig. 3. TEM image of (a) Silica support (b) 7.5% Fe₃O₄/SiO₂ (c) 15% Fe₃O₄/SiO₂ (d) 30% Fe₃O₄/SiO₂ (e) unsupported Fe₃O₄

as nanoparticles of less than 20 nanometer.

Vibrating sample magnetometer (VSM) techniques.

The saturation magnetization were determined by vibrating sample magnetometer (VSM)[20]. Typical ferrimagnetic behavior exerts coercivity and remanence (retentivity) as displayed in Fig. 4. As particle size is decreased, the amount of exchange-coupled spins resisting spontaneous magnetic reorientation is decreased, tending towards superparamagnetic magnetization. Consequentially, decreasing magnetite particle size demonstrates enhanced superparamagnetic behavior.

It is well known that magnetite particles with size of less than 30 nm have a large surface area and exhibit super paramagnetic properties that make them prone to magnetic fields and they do not become permanently magnetized without an external magnetic field to support them [21]. The results of the TEM showed that the prepared magnetite has small particle size. Normally, the obtained magnetite nano-particles exhibited overall magnetic behavior characteristic of soft magnetic particles, with a very narrow hysteresis cycle, and a small coercive field and remnant magnetization [22].

Metal Removal Experiments:

The effect of contact time on removal percentage of Pb(II) by different ratios of Fe_3O_4/SiO_2 :

The effect of the variations in the percentage of removal of Pb(II) with contact time was shown in Fig. 5. The reaction was occurred by 0.025g of 30% 15% and 7.5% Fe_3O_4/SiO_2 per 25ml solution, respectively when the initial lead ions concentration was 100 mg/l and the pH of

the solution equals 4.8 at room temperature.

From this figure, it is observed that the removal percentages of Pb(II) are increased gradually from 56.23 % to 60.04 % for 30% Fe_3O_4/SiO_2 , from 63.49 % to 67.05% for 15% Fe_3O_4/SiO_2 and from 44.3 % to 52.54 % for 7.5% Fe_3O_4/SiO_2 , respectively. By increasing the contact time from 10 to 360 minute the removal efficiency of Pb(II) have shown a fast rate of adsorption during the first 30 minute of the adsorbate-adsorbent contact and the rate of removal becomes almost insignificant due to a quick exhaustion of the adsorption site. The equilibrium reached at 30 min. and was taken as the optimal contact time for the subsequent experiments.

Also the comparison between different ratios of magnetite loaded on the surface of silica support was shown in Fig. 5 at different time. It was found that the removal percentage of Pb(II) was increased by increasing the ratio of the magnetite on the silica support from 7.5% Fe_3O_4/SiO_2 to 15% Fe_3O_4/SiO_2 . This due to the increase of active sites of magnetite which were dispersed on the surface of the silica support with high surface area. However, further increase of magnetite on the surface of silica support from 15% Fe_3O_4/SiO_2 to 30% Fe_3O_4/SiO_2 leads to a decrease in the adsorption efficiency. This can be attributed to coagulation of magnetite on the silica which leading to decrease the number of the exposed active sites so that the adsorption capacity and removal percentage of Pb(II) was decreased. Concequently 15% Fe_3O_4/SiO_2 is considered to be the suitable ratio of magnetite supported on silica for the adsorption of Pb(II) and therefore it was taken for the next studies.

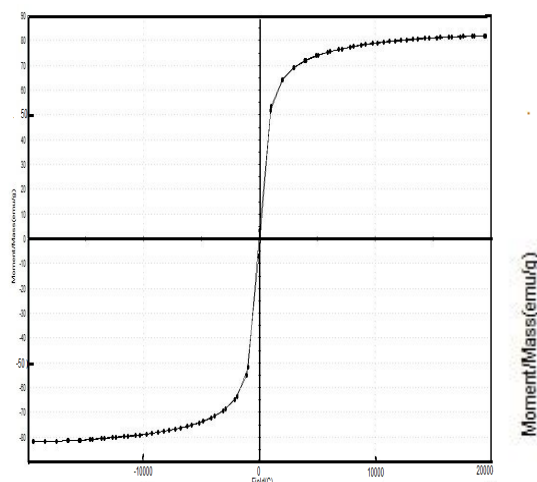


Fig. 4. The absence of hysteresis implies superparamagnetism

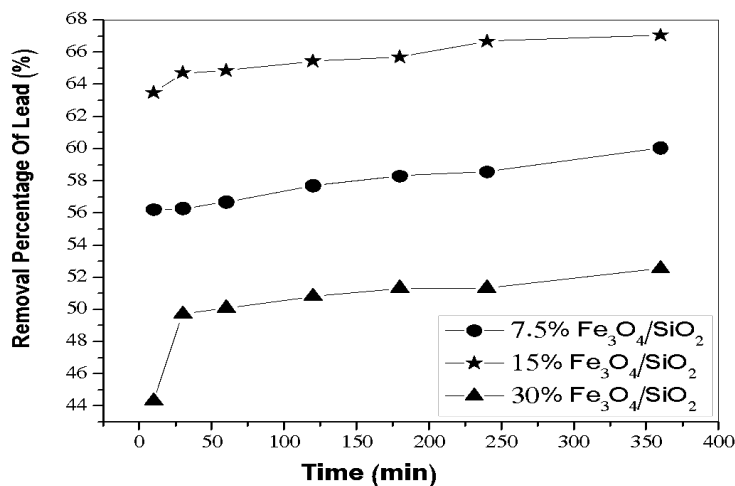


Fig. 5. The effect of contact time on removal percentage of lead by 0.025g of 30%, 15% and 7.5% Fe₃O₄/SiO₂/25ml solution when initial Pb (II) concentration was 100mg/l and the pH of the solution was 4.8 at room temperature.

Effect of pH of the solution on the adsorption of Pb(II):

The effect of pH on the adsorption of Pb(II) by 15% Fe₃O₄/SiO₂ nanoparticles was studied by varying the pH between 2.0 and 5.2 because of at higher pH more than 5.2 the lead ion may be precipitated as Pb(OH)₂. The reaction was carried out for 30 min. when the initial concentration of Pb(II) was 100mg/l, the dose of 15% Fe₃O₄/SiO₂ nanoparticles was 0.025g/25ml.

As shown in Fig. 6, the removal percentage of Pb(II) was increased from 44.08% to 64.76% with increasing the pH of the solution from 2.0 to 4.8 and it was decreased to 63.7 with further increase in the pH of the solution to 5.2.

The removal percentage of Pb(II) decreased at highly acidic conditions is probably due to the presence of high concentration of H⁺ ions on the adsorbent surface, competing with Pb(II) for adsorption sites. So, the maximum removal of Pb(II) was achieved at pH 4.8.

The effect of adsorbent weight on the adsorption of Pb(II):

The determination of the optimal adsorbent weight is another main parameter in the removal percentage of Pb(II). The effect of different weight of 15% Fe₃O₄/SiO₂ was studied at a series ranging between 0.025g/25ml to 0.25g/25ml. The adsorption was carried out when the initial concentration of Pb(II) was 100 mg/l and the pH of the solution was 4.8 at room temperature for 30 minute.

Figure 7 shows that the percentage of removal of Pb(II) was increased from 64.72% to 99.84 by increasing the adsorbent weight from 0.025g/25ml to attain maximum at 0.125g/25ml this due to that the increase of adsorbent weight, the number of the available active sorption sites increases, leading to an increase in the adsorption capacity and removal percentage of Pb(II) and then remained almost constant by further increase in adsorbent weight. Therefore, 0.125g/25ml weight of adsorbent was considered as the optimum weight.

The effect of the initial concentration of lead on the adsorption of Pb(II):

The effect of the variation of the initial concentration on the removal percentage of Pb (II) was studied at different initial concentrations ranging from 50 to 500 mg/l, when the dose of adsorbent was 0.125g/25ml, and the pH of the solution was 4.8 at room temperature. The results are graphically repressed in Fig. 8.

From this figure, it can be seen that the adsorption decreases gradually from 99.84% to 14.62% with increasing initial lead concentration from 50 to 300 mg/l, and remained constant up to 500 mg/l. This is due to sufficient adsorption sites available at lower initial concentration of Pb(II), but at higher initial concentrations the adsorption capacity and removal percentage of Pb(II) ions decreased due to the saturation of active binding sites of adsorbent and initial concentrations of Pb(II) are greater than adsorption sites

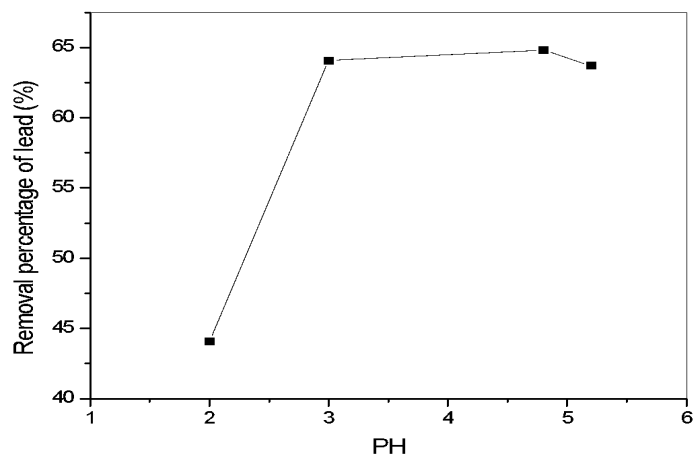


Fig. 6. The effect of the variation of pH value on the removal percentage of lead by 0.025g/25ml of 15% $\text{Fe}_3\text{O}_4/\text{SiO}_2$ when the contact time 30 min. and the initial Pb (II) concentration was 100 mg/l at room temperature.

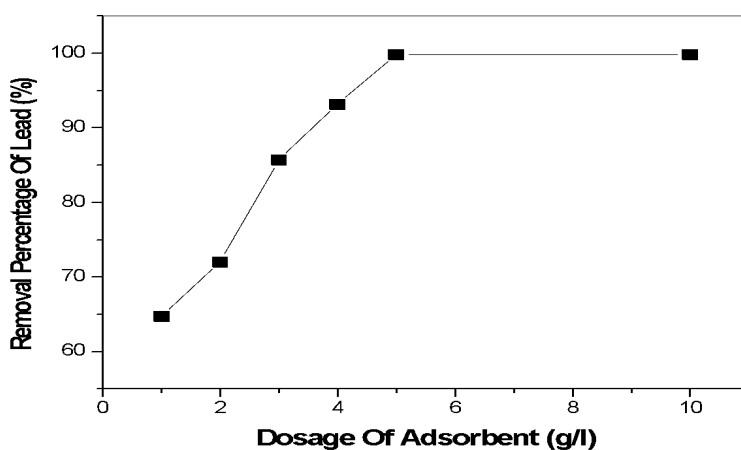


Fig. 7. The effect of variation of the adsorbent weight on the removal percentage of lead when the initial Pb (II) concentration was 100 mg/l, the contact time 30 min., and the pH of the solution was 4.8 at room temperature.

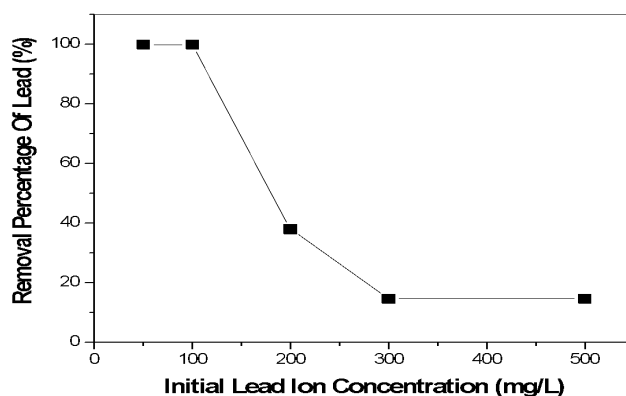


Fig. 8. The effect of variation of the initial lead concentration on the removal percentage of lead by 0.125g/25ml of 15% $\text{Fe}_3\text{O}_4/\text{SiO}_2$ when the contact time 30 min., pH of solution was 4.8 at room temperature.

Therefore, no more adsorption of Pb(II) ions can be accompanied with increasing the initial concentration. Therefore, the availability of active sites of adsorbent is low at higher concentration of Pb(II) eventually decreasing the adsorption capacity and removal percentage of Pb(II) ions. Thus it can be said that removal of lead is highly concentration dependent.

Adsorption Isotherms:

Langmuir and Freundlich isotherms for the adsorption of Pb(II) on 15% Fe₃O₄/SiO₂ are shown in Fig. 9 and 10 respectively. The values of the equilibrium parameters of both models, K_L , q_{max} , K_F and n , together with the corresponding regression coefficient (R^2) were computed and provided in Table 1. The Langmuir model represents a much better fit for the experimental equilibrium adsorption data of Pb(II) adsorption on the surface than Freundlich model as evidenced by relatively higher value of R^2 . This result indicated that monolayer adsorption of Pb(II) takes place on well-defined energetically equal sites and there is no subsequent interaction between the adsorbed Pb(II) molecules.

Adsorption kinetics:

Adsorption kinetics were investigated for a better understanding of the mechanism of adsorption. In the present study, the adsorption data of Pb (II) at different time intervals were analyzed by the pseudo-first-order kinetic model (Eq. 1) and pseudo-second-order kinetic model (Eq. 2).

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (1)$$

$$t/q_t = 1 / k_2 (q_e)^2 - t/q_e \quad (2)$$

Where k_1 (min⁻¹) and k_2 (g/mg/min) are the equilibrium rate constants of pseudo-first- and pseudo-second-order rate equation, respectively.

From Fig. 11 we can see the pseudo-first-order model did not fit well with the kinetics data as was evidenced from the low value of the regression coefficient ($R^2 = 0.9307$). On the other hand, the good linear plot of t/q_t versus t based on pseudo-second order kinetic model, as shown in Fig. 12, with extremely high regression coefficient near to unity ($R^2 = 0.999$).

TABLE 1. Parameters of Langmuir and Freundlich isotherms

Langmuir isotherm			Freundlich isotherm		
q_{max} (mg/l)	K_L (L/mg)	R^2	n	K_F	R^2
25.30364	0.046165	0.96851	32.13368	19.544	0.43456

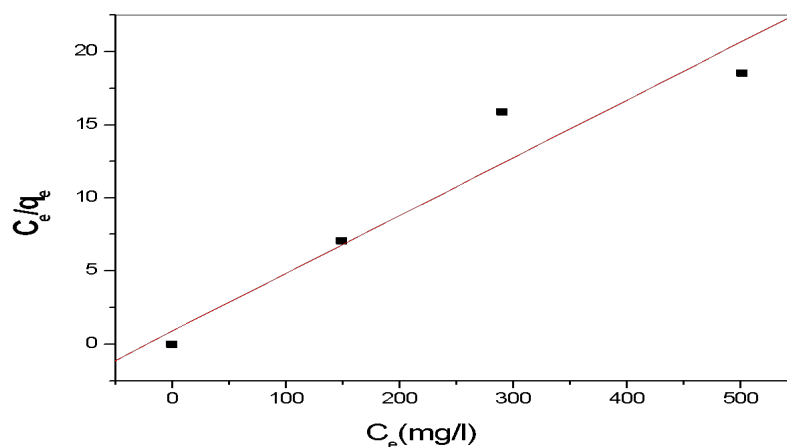


Fig. 9. Langmuir isotherm plot for adsorption of Pb(II) on 15% Fe₃O₄/SiO₂.

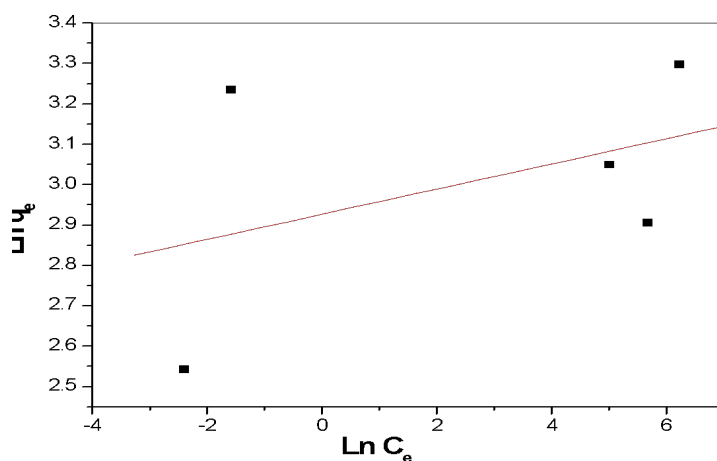


Fig. 10. Freundlich isotherm plot for adsorption of Pb(II) on 15% $\text{Fe}_3\text{O}_4/\text{SiO}_2$.

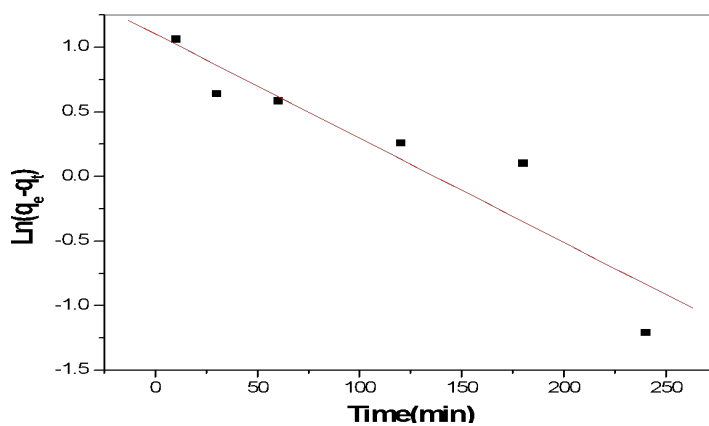


Fig. 11. Pseudo-first-order linear plots for adsorption of Pb(II) on 15% $\text{Fe}_3\text{O}_4/\text{SiO}_2$.

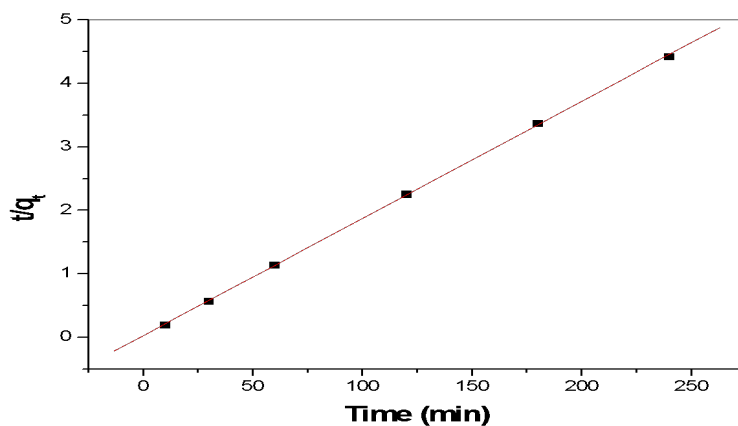


Fig. 12. Pseudo-second-order linear plots for adsorption of Pb(II) on 15% $\text{Fe}_3\text{O}_4/\text{SiO}_2$.

Conclusions

The results showed that 15% Fe₃O₄/SiO₂ nanoparticles prepared by co-precipitation method is more active than that 7.5% and 30% Fe₃O₄/SiO₂ for removal of Pb(II) ions from aqueous solutions. The equilibrium was achieved practically in 30 min when the percentage removal of lead was 99.84%. Langmuir model is found to be in a good agreement with experimental data on adaptive behavior of Pb(II) ions by 15% Fe₃O₄/SiO₂ is then considered as a useful adsorbent for the treatment of wastewater containing lead. The kinetic study indicated that the adsorption of lead followed the second-order adsorption.

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ازالة ايونات الرصاص من مياه الصرف الصناعي باستخدام الماجنتايت المحمل على السليكا

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تم تحضير سلسلة من (SiO₂ / Fe₃O₄) النانوية مع تحميل نسب مختلفة من Fe₃O₄ بواسطة الترسيب المتزامن. تم دراسة الخصائص البلورية والشكلية للمنتجات المحضرة بواسطة حيود الأشعة السينية (XRD)، والمجهر الإلكتروني النافذ (TEM)، وكذلك الخواص المغناطيسية للماجنتايت. تم تقييم أداء الامتزاز للعينات في إزالة أيونات الرصاص من المحلول المائي. تم إجراء تجارب لدراسة تأثير العديد من العوامل بما في ذلك درجة الحموضة الحل، والوقت، وتركيز أيون الرصاص الأولية والجرعة الممتزة على كفاءة الامتصاص. أشارت نتائج تجارب الامتزاز إلى أن أنواع الماجنتايت المستخدمة لديها قدرة امتصاص ممتازة لإزالة أيونات الرصاص.