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Effective parameters for the adsorption of chromium(III) onto iron oxide magnetic nanoparticle

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Abstract Most of the industrial wastewaters comprise toxic, biologically non-biodegradable, and heavy metals which tend to accumulate in the biological organisms causing different diseases. There are some novel technologies and strategies to remove these pollutants. Using the magnetic nanoparticles which are cheap, recyclable, and reusable can be considered as an effective method for removing the pollutants as they do not require conservation or complicated equipments. Using this method, dangerous and rare heavy metals can be restored to the industry. In this study, magnetic nanoparticles with the size of 30 nm were prepared and used for the removal of chromium from synthetic wastewater polluted by chromium sulfate. For this purpose, removal of various concentrations of chromium(III) from wastewater was investigated. The best concentration was achieved in the removal efficiency of 99.1 %. The optimal values of pH, rotation speed of magnetic stirrer, time, temperature, and the amount of nanoparticles were determined according to the primary concentration (500 mg/L). The mechanism of chromium adsorption onto iron oxide (Fe₃O₄) magnetic nanoadsorbent was also investigated. The results showed both Freundlich and Longmuir isotherms to be the best fit for the chromium adsorption, with Freundlich isotherm being more suitable.

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Introduction

In recent decades, the environment exposure to heavy metals has triggered worldwide concerns, resulting in utilizing a host of treatment techniques and processes (Shen et al. 2009). One of these techniques is the process of electrocoagulation which is a novel and efficient method for heavy metals removal (Akbal and Camc 2011; Arroyo et al. 2009; Bhatti et al. 2009; Schulz et al. 2009; Sun et al. 2009). Other methods for removing metal ions from wastewater are deposition, evaporation, solvent extraction, ion exchange, reverse osmosis, separation by membrane, etc. Most of these methods have some shortcomings such as high cost of operation, high required investment, and disposal of metal sludge; therefore, attempts have been made to develop cheap materials for removing pollutants from aquatic environments (Dizadji and Abootalebi Anaraki 2011; Malakootian et al. 2009; Shen et al. 2009). The new advancements in using nanoparticles have solved some of the problems in this regard (Ambashta and Sillanpää 2010).

Having appropriate adsorption surfaces, nanoparticles enjoy unique characteristics (Faraji et al. 2010; Xu et al. 2012). Metal nano-oxides were also applied to remove heavy metals from water and wastewater (Hua et al. 2012); for instance, iron oxide (Fe₃O₄) magnetic nanoparticle is efficient not only in the adsorption of metals, but also in medical (Shen et al. 2009) and biotechnological processes (Yavuz et al. 2009). Another advantage of magnetic nanoparticles is that they can be easily separated from liquid via external magnetic field (Shen et al. 2009). In



comparison with common treatment methods, utilization of magnetic nanoparticles brings about many advantages, such as requiring simple equipments, facilitated operation, high efficiency, and high potentials for restoration and reusability of magnetic beds, which have made them well qualified to be widely applied. They can be used not only in the treatment of wastewater contaminated with heavy metals, but also in major industries owing to their reusability and metal recyclability (Chen et al. 2011).

Many studies have been carried out showing the high efficiency of magnetic nanoparticles in removing different pollutants, especially heavy metals, from water and wastewater, including oil refinery wastewater treatment using zero-valent iron nanoparticles by ultrasound (Rasheed et al. 2011); paper mill wastewater treatment by iron oxide covered with polyacrylic acid for the purpose of chemical oxygen demand (COD) removal (Zhang et al. 2011); application of Fe_3O_4 magnetic nanoparticles for color removal from water (Absalan et al. 2011; Iram et al. 2010); methylene blue removal from aquatic environment by Fe₃O₄ nanoparticles fixed with pectin (Rakhshaee and Panahandeh 2011); reduction of polybrominated diphenyl ethers (PBDE) by metal nanoparticles (Fang et al. 2011a); adsorption of cadmium from aquatic environment by magnetic nanoparticles and its restoration from industrial wastewater by magnetic particles (Chen et al. 2011; Tu et al. 2012); copper and chromium(VI) adsorption from aquatic environment by iron oxide magnetic nanoparticles with amino factor groups (Shen et al. 2012); chromium(VI) removal from electroplating wastewater by metal nanoparticles (Fang et al. 2011b); iron nanoparticles fixed with pectin to remove color from aquatic solution (Rakhshaee 2011); reduction of decabromodiphenyl ether by zero-valent iron nanoparticles fixed within mesoporous of silica microspheres (Oiu et al. 2011); adsorption of arsenite onto the Fe₂O₃ nanoparticles and arsenic removal from water by Fe₃O₄ nanoparticles (Akin et al. 2012; Prasad et al. 2011); application of Fe₃O₄ magnetic nanoparticles with functional groups of carboxyl, amine, and thiol in removing Escherichia coli and toxic metals ions (Singh et al. 2011); using bakery yeast along with Fe₃O₄ nanoparticles to remove methyl violet from aquatic environment (Tian et al. 2010); lead removal from aqueous solution by magnetic nanoparticles (Tan et al. 2012); removal of heavy metal ions from aquatic environment using Fe₃O₄ magnetic nanoparticles reformed by polymer (Ge et al. 2012); removing ions of nickel, cadmium, and lead from water by Fe₃O₄ nanoparticles reformed by carboxymethyl-\beta-cyclodextrin (Badruddoza et al. 2013); treatment of wastewater by Fe_3O_4 nanoparticles (Shen et al. 2009); and Pb(II) removal from wastewater by magnetic nanoparticles of iron oxide (Nassar 2010).

Chromium is a transition element of VI B periodic table. Chromium sulfate can be mentioned as one of the most important and common chromic compounds (Mortimer 1986).

Investigations imply that chromium(III) removal by Fe_3O_4 magnetic nanoparticles yet has not been carried out. The present study is an attempt to investigate the performance of Fe_3O_4 magnetic nanoparticles in removing chromium(III) as a pollutant with various concentrations. The role of effective parameters including pH, temperature, rotation speed of magnetic stirrer, amount of nanoparticles, and contact time has also been considered. This study was carried out in water and wastewater treatment laboratory at Tehran University on 2012.

Materials and methods

For the purpose of removing chromium pollution, artificial wastewater containing chromium(III) ions with the initial concentrations of 250, 500, 750, and 1,000 mg/L was tested separately. The reason for choosing these concentrations is the variability of chromium concentration in tanning wastewater from 400 to 1,700 mg/L (Ludvik 2000).

In this study, chromium sulfate [Cr₂ (SO₄)₂(OH)₂·16H₂O] made by Rock Chemical Company was used. The size of Fe₃O₄ magnetic nanoparticle used in this study was 30 nm, which is considered an average particle size. The nanopowder was of 99 % purity, with specific surface of 55 m²/g.

To prepare nanoparticles, the mixture of FeCl₂ and propylene glycol was boiled. Thereafter, sodium hydroxide (NaOH) was gradually added to the mixture until pH was raised. The mixture was then stirred and cooled to reach the room temperature, and Fe₃O₄ particles were separated by centrifugal movement. The obtained particles were characterized using a transition electron microscope. The test was conducted under the conditions that optimal time of the experiment and optimal rotation speed of magnetic stirrer were 45 min and 200 rpm, respectively. Before conducting the test and after carrying out the experiment, the concentration of chromium(III) was determined in the samples according to the standard methods of water and wastewater experiments (Franson 2005). The concentration of chromium was determined by atomic absorption spectrophotometry. IKA magnetic stirrer with RCT Basic (made in Germany) and Metrohm 691 pH meter (made in Switzerland) were used. The wastewater obtained from chromium sulfate was touched by Fe₃O₄ magnetic nanoparticles. After the experiment, the required wastewater was immediately collected and its chromium concentration was measured accordingly. The adsorbent comprising pollutant was separated by an external magnetic field which was a horseshoe-shaped magnet. The particles were washed by sulfuric acid, and chromium(III)

was put in the recyclable liquid. Figure 1 shows the different stages of the test. For the purpose of neutralization, magnetic nanoparticles were reduced by sodium hydroxide and washed by distilled water.

Considering the amount of pH discharge into the environment and in order to determine the optimal pH, it was decided to set the value of pH within the range of 3–9. This was adjusted by sulfuric acid and sodium hydroxide.

Results and discussion

In this method, wastewater was touched by nanoadsorbent, the surface of which was to adsorb the pollutants. This way, the pollutant was removed from the environment and adsorbed by the adsorbent. Adsorption time, pollutant's concentration, amount of nanoparticles, and pH proved to be the important parameters in pollutant removal. The results of adsorption test and the percentages of chromium removal with the initial concentration of 500 mg/L in different pH values are shown in Fig. 2.

To determine the optimal pH, test temperature was 25 °C, magnet rotation speed was 300 rpm, test time was 60 min, and amount of nanoparticle was 1 g.

Table 1 shows the amount of initial pH as well as final pH versus the amount of chromium removal after adsorption of the pollutant onto the Fe_3O_4 magnetic nanoparticles.

As Table 1 indicates, adsorption of chromium(III) occurs in the high pH. This attributes to the fact that in high pH, hydroxide ions are adsorbed onto the adsorbent surface, resulting in electrostatic attraction between the ions of hydroxide and chromium. In case of pH reduction, hydroxide surface weakens and reduces the adsorption of chromium ions. Chromium adsorption onto Fe_3O_4 occurs in a pH of approximately 6. Results showed that in higher pH, the main cause of chromium removal is the formation of hydroxide deposit. Studies carried out by Nassar (2010) confirm the obtained results as well.



Fig. 2 Comparison of chromium removal percentages in different pH values after adsorption process

Table 1 Amount of initial and final chromium concentrations and pH values before and after adsorption process

Initial chromium (mg/L)	Final chromium (mg/L)	Initial pH	Final pH
500	336.42	3	2.99
500	55.05	5.5	5.13
500	3.95	7.5	6.41
500	0.6	9	6.95

The factors investigated in this study are amount of iron oxide nanoparticles, optimal time of the test, magnet rotation speed, and the test temperature in the initial chromium concentration of 500 mg/L. The impact of initial concentration of chromium on the amount of adsorption was studied as well. The analysis of these factors has been given further in the present study. To obtain the amount of nanoparticles, the tests were carried out using 0.25, 0.5, 0.75, 1, and 1.5 g of Fe₃O₄ magnetic nanoparticles, with the pH of 5.5. The obtained results have been indicated in



Fig. 1 Stages of chromium removal from wastewater by Fe_3O_4 nanoparticles, and its separation and reduction. **a** Wastewater comprising chromium(III); **b** encompassing chromium(III) by magnetic

nanoparticles; c chromium(III) removal by magnetic separation; d reduction of chromium(III); and reuse of magnetic nanoparticles





Fig. 3 Comparison of chromium removal percentages with different amounts of Fe_3O_4 nanoparticles after adsorption process

Table 2 Initial and final chromium and pH before and after adsorption process with different amounts of nanoparticles

Amount of nanoparticle (g)	Initial chromium (mg/L)	Final chromium (mg/L)	Initial pH	Final pH
0.25	500	215.44	5.46	4.47
0.5	500	128.46	5.64	4.53
0.75	500	58.6	5.58	4.48
1	500	55.05	5.75	4.60
1.5	500	7.64	5.69	4.71

Fig. 3 and Table 2. As it can be seen, higher amount of nanoparticles leads to higher removal percentage. The results of adsorption using 0.75 and 1 g of nanoparticles were found to be almost the same. Therefore, to obtain the optimal values of other parameters, the experiments went on using 0.75 g of nanoparticles. With an increase in the amount of nanoparticles, adsorption rate was increased due to the increase in adsorption sites. Previous studies have also highlighted this point (Nassar 2010; Shen et al. 2009).

To obtain optimal time, the tests were carried out in various time durations of 15, 25, 30, 45, 60, and 90 min. The obtained results have been given in Fig. 4 and Table 3. As can be seen, the optimal time was considered to be 45 min and an increase in the test time increased the removal percentage. The increase was so extensive that the adsorbent surfaces become saturated. Afterward, no adsorption increase was observed. In these experiments, the amount of nanoparticles was 0.75 g, the temperature was 25 °C, magnet rotation speed was 300 rpm, and the initial pH was 5.5. The equilibrium time obtained in this study revealed to be consistent with that obtained in the studies carried out by Nassar (2010).



Fig. 4 Comparison of the final adsorbed chromium in various test time durations after adsorption process

Table 3 Comparison of initial and final chromium after adsorption

 process in different time durations

Time of test (min)	Initial chromium (mg/L)	Final chromium (mg/L)
15	500	144.05
25	500	95.8
30	500	73.69
45	500	60.76
60	500	58.6
90	500	56.46

To determine the appropriate magnet rotation speed, the tests were carried out using 0.75 g of nanoparticles, at the temperature of 25 °C, the test time of 45 min, and initial pH of 5.5. The appropriate rotation speed was 200 rpm (Fig. 5). The amount of pH in Table 4 indicates that after the adsorption of hydroxide ions by nanoparticles, chromium was adsorbed onto the particles. This mechanism plays an important role in the process of removing the pollutants. Therefore, higher rotation speed would break the bonds and release the pollutants which lead to a decrease in chromium removal efficiency.

To determine the optimal temperature of the test and the effect of temperature on adsorption process during the chromium removal, several experiments were carried out in different temperatures of 15, 20, 25, and 30 °C. In the series of experiments, test duration was 45 min with 200 rpm rotation speed and 0.75 g of nanoparticles with the initial pH of 5.5 was used. The amount of chromium removal was affected by the initial temperature shown in Fig. 6.

As Table 5 shows, the existing reactions in the adsorption process are endothermic and any increase in temperature within a special time also increases the amount of chromium removal. Since the increase in temperature led to the increase in the amount of energy consumption, the





Fig. 5 Comparison of chromium removal percentages in different rotation speeds of magnetic stirrer after adsorption process

Table 4 Comparison of initial and final chromium and pH values after adsorption process in different rotation speeds

Round of stirrer (mg/L)	Initial chromium (mg/L)	Final chromium (mg/L)	Initial pH	Final pH
200	500	51.29	5.53	4.41
300	500	60.76	5.49	4.54
400	500	66.93	5.48	4.56
600	500	89.47	5.68	4.77

optimal temperature was decided to be 25 °C in these experiments. The results achieved at 25 and 30 °C were almost the same. Researches by Shen et al. (2009) and Nassar (2010) imply that the increase in temperature increases ions mobility and more ions are therefore adsorbed onto the active sites of adsorbent. Therefore, absorption process leads to the increase in the removal efficiency of metal ions.

The results of adsorption test and percentage of chromium removal with different concentrations of chromium have been given in Table 6 and Fig. 7. Test temperature was 25 °C with magnet rotation speed of 200 rpm, and test time was 45 min with 1 g of nanoparticles and initial pH of 6. The reason for increase in the amount of nanoparticles was an increase in the subject of test which required enough surfaces for the adsorption.

The adsorption efficiency decrease was due to an increase in concentration. Studies by Iram et al. (2010) show that the increase in concentration leads to the decrease in removal efficiency.



Fig. 6 Comparison of chromium removal percentages in different temperatures after adsorption process using Fe_3O_4 magnetic nanoparticles

 Table 5
 Initial and final chromium and pH values in different temperatures before and after adsorption process

Initial temperature (°C)	Initial chromium (mg/ L)	Final chromium (mg/ L)	Initial pH	Final pH
15	500	146.32	5.70	4.67
20	500	74.87	5.71	4.62
25	500	43.23	5.59	4.48
30	500	35.55	5.63	4.46

Table 6 Comparison of initial and final chromium and its removal percentages after adsorption process in different initial concentrations

Initial chromium (mg/L)	Final chromium (mg/L)	Removal %
1,000	30.37	96.96
750	16.83	97.76
500	9.6	98.08
250	2.25	99.1

To investigate the mechanism of adsorption of trivalent chromium ion by Fe_3O_4 magnetic nanoparticle, Langmuir and Freundlich isotherm constants were adjusted with the data. Using Langmuir and Freundlich isotherms, the ability and capacity of the adsorbent were determined. Langmuir isotherm is based on single-layer adsorption and lack of movement of adsorbed material, and Langmuir model describes the adsorption occurring on homogeneous





Fig. 7 Comparison of initial and final chromium with different concentrations throughout the adsorption process

surfaces. Linear form of Langmuir equation for adsorption is as follows:

$$\frac{C_e}{q_e} = \frac{1}{b \cdot q_m} + \frac{C_e}{q_m}$$

Drawing C_e/q_e versus C_e , a straight line can be obtained, where q_e (mg/g) is absorption capacity, C_e (mg/L) is equilibrium adsorbate concentration, q_m is maximum value of q_e with increasing C_e , and b is a coefficient associated with adsorption energy and related to adsorption bond strength.

Freundlich model describes the adsorption occurring on adsorbent heterogeneous surfaces. Freundlich equation is as follows:

$$\operatorname{Log} q_e = \log K + \frac{1}{n} \log C_e$$

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where q_e is the amount of adsorbate per unit of adsorbent at equilibrium condition (mg/g), C_e is equilibrium



Fig. 8 Freundlich isotherm of Cr(III) adsorption onto Fe₃O₄ magnetic nanoparticles

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Fig. 9 Langmuir isotherm of Cr(III) adsorption onto Fe₃O₄ magnetic nanoparticles

concentration in solution (mg/L), and K and n are Freundlich constants (Absalan et al. 2011; Nassar 2010). Adsorption isotherms were determined at the temperatures of 25 °C.

As it is evident, Langmuir and Freundlich isotherm models can be taken as good models for the adsorption of chromium onto Fe₃O₄ magnetic nanoparticles. According to Figs. 8 and 9, the amount of R^2 proves that Freundlich isotherm is a more suitable model for adsorbing chromium onto the nanoparticles.

Conclusion

The existing pollutant in the environment was touched by nanoparticles with suitable adsorption surfaces. In this way, the pollutant was removed from the environment and adsorbed by the adsorbent. Thereafter, magnetic adsorbent and the pollutant were separated from the liquid by an external magnetic field in order to be prepared for the further adsorption. The mechanism of chromium(III) removal by adsorption process is affected by pH. In this study, the optimal pH was about 6 and hydroxide was adsorbed by the adsorbent. Afterward, electrostatic attraction occurred between hydroxide and chromium ions within the wastewater. In higher pH values, the existence of hydroxide induced the formation of metal hydroxide deposit. The reactions in this process were endothermic; thereby, higher temperature was suitable for the chromium removal. On the other hand, higher temperature would lead to higher energy consumption. Considering the results from the experiments, the optimal temperature of 25 °C was suggested. The longer the test time, the higher amount of pollutant was adsorbed by active levels of the adsorbent. Thus, the adsorbing levels were rapidly saturated which was due to the fact that an increase in time duration of the test would have no effect on the process of adsorption. The results obtained from the test prove that Freundlich

isotherm is a more suitable model for chromium adsorption onto nanoparticles. The results showed that test time reduced by increase in the amount of nanoparticles, temperature, and pH; however, to determine each parameter, the environmental and economical issues must be closely taken into account.

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