

Application of Modified LECA in Removing Nickel from Wastewater

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Abstract

In the present study, lightweight expanded clay aggregate (LECA) coated with iron oxide was investigated as a low cost sorbent to remove Ni²⁺ from wastewater. Iron oxide coated LECA (IOCL) as a new sorbent was tested for its efficiency as Ni²⁺ sorbent using operational parameters such as contact time, the initial pH of the solutions, and sorbent dosage concentration in batch systems. The adsorption characteristics of the natural LECA and IOCL were investigated through scanning electron microscopy (SEM), X-ray fluorescence spectroscopy (XRF), and X-ray diffraction (XRD) analysis. The maximum sorption efficiency was found to be 96% for IOCL at an initial pH of 6.0 and a sorbent dosage of 5.0 g.l⁻¹. The results revealed that the Ni²⁺ sorption kinetic data were well fitted to a pseudo second-order kinetic model. The experimental data showed that Ni²⁺ sorption was considerably high on IOCL and the new sorbent is an economical method for Ni (II) removal from effluents and aqueous media.

Keywords: Nickel Adsorption, Water Treatment, LECA

1. Introduction

Heavy metals pollution is a growing ecological crisis and major concern today (Ashouri, 2014; Poornaseri et al., 2013). Heavy metals have a great tendency to bio-accumulate and end up as perennial additions to the natural ecosystems (Krishna and Swamy, 2011). Therefore, the problem of removing heavy metals from effluents and contaminated water is an important process and is becoming more important with the increase in the industrial activities (Rađenović et al., 2011).

Nickel is a toxic heavy metal found in the environment as a result of various natural and industrial activities such as silver refineries, galvanization and smelting, mining, electroplating, storage batteries, and zinc base casting (Krishna and Swamy, 2011; Rani et al., 2012; Kadirvelu et al., 2001). As nickel resists corrosion even at high temperatures, it can also be used in gas turbines, rocket engines, and desalination plants (Krishna and Swamy, 2011). The most common oxidation state of nickel is +2; hence nickel (II) forms compounds with all common anions and nickel is available in

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water as Ni^{2+} (aqueous) (Cempel and Nikel, 2006). The higher concentration of Ni causes numerous severe damages such as lung cancer, low blood pressure, muscle tremors, heart attacks tightness, chest pain, cyanosis, and extreme weakness (Varma et al., 2013). Skin contact with nickel causes a deadly sore disease called "Nickel itch." Due to the high toxicity of nickel, the World Health Organization (WHO) established 0.1 mg/l and 3.0 mg/l as the permissible toxic limits for Ni concentrations in drinking water and industrial effluents respectively (Kinhikar, 2012). Therefore, it is essential to remove Ni (II) from industrial effluents before it is released into streams. For this reason, the most widely used methods for Ni (II) removal from the various industrial effluents are advanced treatment processes such as chemical oxidation or reduction, chemical precipitation, ion exchange, electrochemical treatment, reverse osmosis, filtration and membrane separation, etc. (Hasar, 2003). Most current technologies have high maintenance cost and operational difficulties and require relatively expensive mineral adsorbents, which offsets performance and efficiency advantages (Amiri, et al. 2011). Adsorption has received more attention due to its high application efficiency, cost effectiveness, and being easy to operate among the physicochemical treatment processes (Haque et al., 2008; Behnood et al., 2013; Jamei et al., 2013). Numerous materials have been examined as adsorbents for Ni (II) removal from wastewater including activated carbon, silica, ion-exchange resins, rock materials, activated slag, agricultural wastes, microbial- and plant-derived biomass, and chitin (Varma et al., 2013). However, it is necessary to explore a low-cost, efficient, high adsorption capacity sorbent for Ni (II) treatment from contaminated water and effluents (Babel and Kurniawan, 2003).

Based on published works to date, iron or iron-coated materials such as sands, alumina, zeolite, red mud, etc. are widely used. Fe (III) creates favorable adsorption sites for the removal of heavy metals, is an effective and high efficient adsorbent for the removal of heavy metal ions from the contaminated water. Therefore, the development of new iron-coated materials for Ni (II) removal from contaminated water is a priority in contaminated regions to unauthorized Ni (II) concentrations.

Light weight expanded clay aggregate (LECA) as a new material is an environment-friendly, non-biodegradable, and chemically natural product produced by firing plastic clay in a rotary kiln at around 1200 °C (Shojaimehr et al., 2013; Malakootian et al., 2009). A high temperature (1200 °C) burns organic compounds and the expanding clay forms pellets with honeycomb structure (Yaghi and Hartikainen, 2013). The outside surface of each granule melts and becomes sintered (Yaghi and Hartikainen, 2013). The resulting lightweight aggregates are porous, moisture impermeable, inert with a natural pH value, are capable of insulating against high temperature and sound, and will not break down in water (Yaghi and Hartikainen, 2013). In addition, LECA in natural life is employed in agriculture as a growing medium in hydroponics systems and blended with soil to improve drainage to keep moisture in the soil; in construction industrial, it is used for the production of lightweight concrete blocks and, in water treatment facilities, it is utilized for the filtration and purification of effluents and drinking water (Yaghi, 2007).

LECA and modified LECA are considered as low cost adsorbents (Crini, 2006; Malakootian et al., 2009). The cost of LECA is around 0.15 US\$/kg (Nkansah et al., 2012) compared to that of conventional activated carbon, which is about 20 US\$/kg (Crini, 2006). LECA and modified LECA have been successfully used in the removal of poly aromatic hydrocarbons (Nkansah et al., 2012), and some heavy metal ions from water (Haque et al., 2008; Kalhori et al., 2013; Yaghi and Hartikainen, 2013). However, the impact of Fe oxide coating on Ni (II) sorption by LECA has not been investigated yet. To fill the gap, in this study, iron-oxide-coated LECA (IOCL) as a new sorbent is employed to investigate the removal efficiency and sorption properties of IOCL for the removal of Ni

(II) from contaminated water. Consequently, in this paper, it is aimed to (1) develop a new and low cost adsorbent from LECA using iron oxide, (2) characterize the NL and IOCL samples using appropriate techniques, (3) determine the effects of contact time, the initial pH of Ni (II) solution, and the sorbent dosage on the Ni (II) removal efficiency of the sorbent, and (4) study the sorption kinetics.

2. Materials and methods

2.1. Chemicals

All the chemicals (materials) used in this work were analytical grade and all the stock solutions were prepared with distilled water. Analytical grade $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (molecular weight = 237.7 g/mol, Merck) was used for the preparation of nickel (II) stock solution by dissolving an appropriate amount in distilled water. $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (molecular weight = 404 g/mol, Merck) was used for the preparation of iron solutions and LECA modification. The solution pH was adjusted and regulated using 0.1 N HNO_3 or NaOH .

2.2. Apparatus and characterization of adsorbent

The morphological characterization of natural LECA (NL) and IOCL were determined using an XL-30 Philips (The Netherlands) scanning electron microscope (SEM). The Ni (II) concentration in the filtered solution was determined by a Varrian 220 SS atomic adsorption spectrometer. In addition, a pH meter (Jenway model 3510, UK) and a platform shaker (yellow line-MSEB model, Germany) were used to conduct the batch experiments. The XRD patterns of the NL and IOCL were collected by means of a Philips PW1400 (The Netherlands). X-ray fluorescence analysis (Philips WDXRF series PW1404, The Netherlands) was used to characterize the chemical compositions of the sorbent. Moreover, Fourier transform infrared (FTIR) spectroscopy was used as a complementary technique of XRD in order to obtain a qualitative characterization of the samples. The infrared (IR) spectra of the samples were obtained in the range of 400–4000 cm^{-1} wave number by means of an FTIR vertex 70 Bruker (Germany).

2.3. Adsorbent preparation

LECA was provided from LECA Company, Tehran, Iran. LECA was crushed by hand, sieved through a 50 mesh size screen and then the particle-size fraction of 0.25–0.50 mm was chosen for the coating procedure. One hundred grams of the sieved LECA was soaked in an acid solution (0.01 M HNO_3) for 48 hrs to remove any contaminants and then LECA was rinsed eight times with distilled water and oven-dried at 105 °C for 24 hrs. IOCL was prepared by modifying the methods of Edwards and Benjamin (Edwards and Benjamin, 1989). Briefly, for coating the LECA with ferric nitrate, 0.5 M Fe (III) was prepared by dissolving $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in distilled water and the pH was set at 11.5 ± 0.5 by adding concentrated NaOH drop wise during stirring. Then, the solution was poured over the LECA at a LECA to ferric nitrate weight ratio of 2:1 (100 g LECA / 200 ml $\text{Fe}(\text{NO}_3)_3$ solution) and the mixture was heated in an oven at 110 °C for 24 hrs with stirring during the first 4 hrs. After that, the coated LECA was washed with distilled water several times so that then run-off water became clear. Finally, the washed materials were oven-dried at 105 °C for 24 hrs and the coated LECA was saved in a glass bottle for usage.

2.4. Batch adsorption studies

All the experiments were conducted in batch mode in 100 ml glass beakers at a constant agitation speed (200 rpm) and room temperature (25 °C). Several operating parameters, including contact time (30 mins–240 mins), pH (3–9), and adsorbent dosage (1-5 g/l) were studied. The effect of adsorption time on Ni (II) adsorption by IOCL was first studied by varying the contact time at room temperature, a pH of 6.0, and the adsorbent dosage of 5 g/l. To do this, 0.25 of the adsorbent was added to 50 ml of the solution in a glass beaker containing Ni (II) ions at an initial concentration of 50 mg/l. The pH of the solutions was adjusted by adding 0.1 N nitric acid or 0.1 N sodium hydroxide solutions. The mixture solutions were shaken at 200 rpm by the platform shaker. The samples were taken at predetermined time intervals, filtered (0.45 μ millipore membrane), and the nickel concentration in the filtrate solution was then determined using flame atomic adsorption spectrophotometer. The removal efficiency (RE) was calculated by means of the following equation:

$$RE = \frac{100(C_0 - C_t)}{C_0} \quad (1)$$

where, RE is the percentage of Ni (II) removed at time t ; C_0 and C_t represent the initial and residual concentrations of nickel (mg/l) after time t in the solutions respectively.

2.5. Kinetic experiments

In this experiment, 500 ml of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ with a nickel (II) concentration of 50 mg/l was prepared and its pH was set at 6.0 by adding 0.1 N nitric acid or 0.1 N sodium hydroxide drop wise. Next, 50 ml of this nickel (II) solution was delivered into a 100 ml glass beaker, to which 0.25 g of IOCL, as the sorbent, was added. The mixture solutions were stirred at 200 rpm at room temperature. After the contact times of 30, 60, 120, 240, 360, and 480 mins, the mixture solutions were allowed to settle for 5 mins. Next, the mixture was filtered through a 0.45 μ millipore membrane and the amount of nickel ions in the residual solutions was analyzed using a Varrian 220 SS atomic adsorption spectrometer.

The linearized form of the pseudo first-order equation is given by the following equation (Largergren, 1898):

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \quad (2)$$

where, q_e and q_t are the amount of metal ions adsorbed (mg/g) at equilibrium and at time t respectively; K_1 is the rate constant of pseudo–first–order adsorption process (1/min). The linearized form of the pseudo second–order equation given by Largergren is defined by (Ho and McKay, 1999; Seyedi et al., 2013; Popuri et al., 2009):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (3)$$

As the quantity q/t approaches zero (mg/g min), the initial adsorption rate h_0 is obtained by the following expression (Kalhori et al., 2013):

$$h_0 = k_2 q_e^2 \quad (4)$$

3. Results and discussion

3.1. Characteristics of the adsorbent

Figure 1 reports the morphology of NL and IOCL, in which holes and cave type openings on the surface state high porosity and a favorable surface area for adsorption process. The surface of LECA did not change significantly after the modification. This result indicated that an improvement in IOCL adsorption efficiency was not related to the increased surface area.

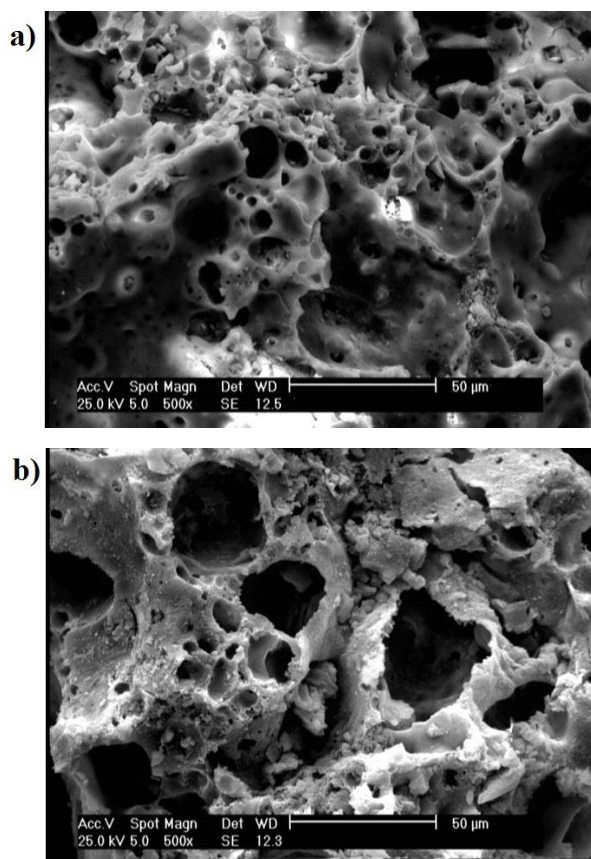


Figure 1
SEM images of (a) the natural LECA and (b) iron-oxide-coated LECA.

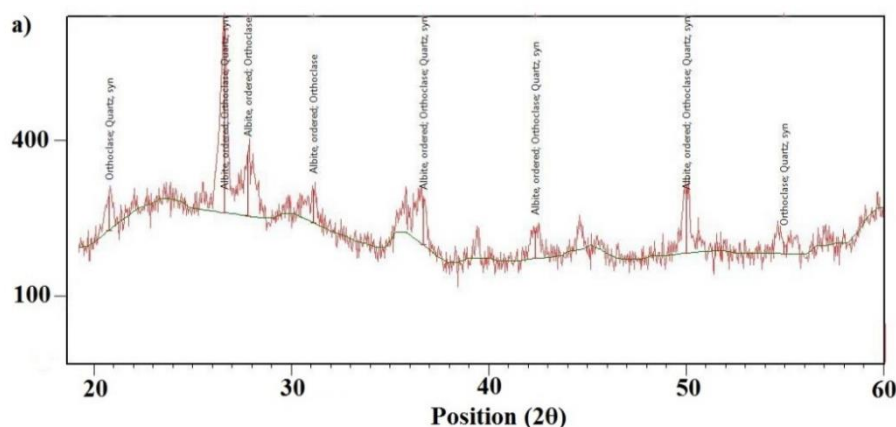
The results of XRF analysis for the determination of LECA characterization are listed in Table 1. From the data tabulated in Table 1, NL and IOCL contained a significant amount of SiO_2 , Al_2O_3 , and Fe_2O_3 , while the other metal oxides were present as a trace mineral. According to the results, the natural LECA sample is a typical aluminosilicate mineral. The chemical composition was significantly affected by iron oxide modification. The data indicate that the mass fraction of iron oxide (Fe_2O_3) increased from 10.44 to 37.26% (w/w). However, the mass fraction of quartz (SiO_2) and aluminum decreased from 13.84 to 9.01% and from 56.89 to 36.72% (w/w) respectively. Therefore, the increase in the mass fraction of Fe_2O_3 in IOCL is probably the main contributing factor in the modified LECA.

The chemical composition and the Si/Al ratio of the NL sample are similar to what is cited in literature (Kalhori et al., 2013). In addition, the research of Kalhori et al. (2013) confirmed the fact that the mass fraction of iron oxide (Fe_2O_3) is significantly increased in modification with an iron solution.

As seen in Figure 2, the XRD analysis confirms the mineralogical composition of the samples. The X high background and a very broad peak clearly indicate the presence of amorphous phase in the samples. Apart from the amorphous phase, the characteristic peaks of quartz and albite can be seen among the crystalline phases presented in the NL and IOCL samples. Albite is the sodium end member of the plagioclase solid solution series with the formula of $\text{NaAlSi}_3\text{O}_8$. In addition, orthoclase, a common alkali feldspar mineral, is an important tectosilicate mineral, which forms igneous rock with the formula of KAlSi_3O_8 . As can be seen, the XRD pattern of IOCL remains almost intact, which indicates that the coating process does not influence the overall crystalline phases.

Table 1
The sorbent characterization.

Constituent	Chemical composition (wt.%)	
	NL	IOCL
SiO ₂	56.889	36.724
Al ₂ O ₃	13.841	9.010
Fe ₂ O ₃	10.444	37.260
P ₂ O ₅	0.259	0.153
SO ₃	0.027	0.002
CaO	2.944	2.081
MgO	4.730	3.315
MnO	0.102	0.082
K ₂ O	2.937	1.726
TiO ₂	0.696	0.497
Na ₂ O	3.256	6.162
LOI	1.4	0.970



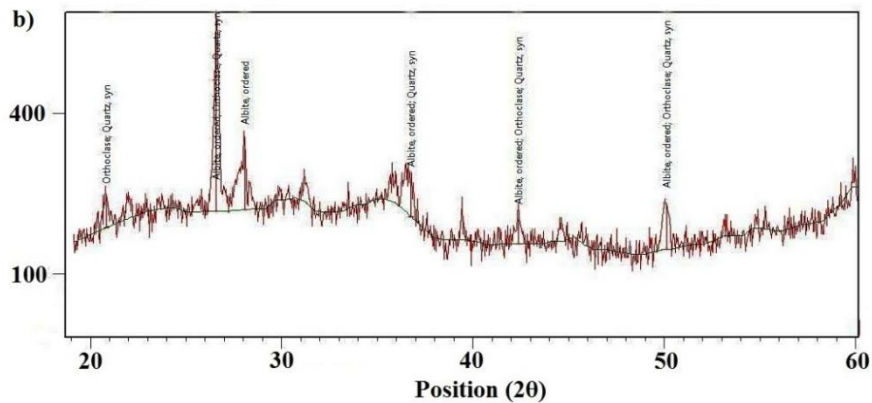


Figure 2
XRD patterns: (a) natural LECA and (b) iron-oxide-coated LECA.

3.2. Effect of contact time on sorption

Figure 3 shows the effect of contact time on Ni (II) removal from water at a pH of 6.0 and room temperature (25 °C) by IOCL. It can be concluded that the sorption rate rises by increasing the contact time. The results show that nickel is removed at a rapid rate during the first 30 minutes due to the availability of large number of binding sites and surface area on the IOCL surface at the initial stage. After 2 hrs, which approximately 92.5% of Ni (II) had been removed, the removal efficiency decreased gently as sorption increased until it reached a maximum at 4 hrs. After this equilibrium period, the removal efficiency versus contact time curve is smooth and, at this time, 96% of nickel has been removed. Therefore, 2 hrs and 4 hrs are selected as the optimum and approximate equilibrium times respectively.

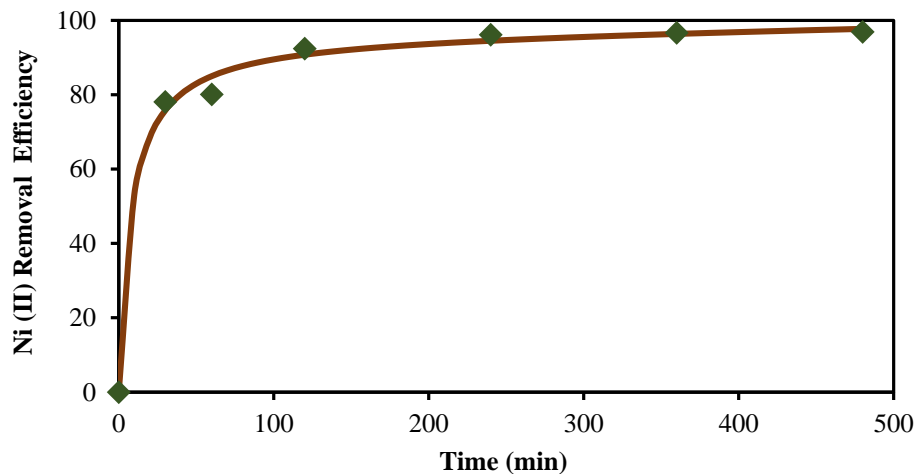


Figure 3
Effect of contact time on the adsorption of Ni (II): IOCL adsorbent= 5 g/l, initial Ni (II) concentration=50 mg/l, pH = 6, $T=25$ °C, and agitation rate= 200 rpm.

3.3. Effect of pH

The pH of the aqueous solution determines the surface charge of the adsorbent and therefore is an important parameter which affects the adsorption process (Amiri et al., 2011). To determine the optimal pH value for the maximum removal of Ni (II), some batch sorption experiments were carried out over a pH range of 3 to 9. Figure 4 indicates that nickel binding to IOCL is pH-dependent since

the percentage of nickel removal from water is increased to a maximum value at an initial pH of around 6 and is then decreased. At a low pH, the number of available H^+ ions is high and H^+ ions hinder Ni (II) ions from reaching the sorption sites because of the repulsive forces. Therefore, the sorption of Ni (II) on IOCL is increased with an increase in the initial pH from 3 to 6 since the repulsive forces and the competition of Ni (II) and H^+ decrease. At higher initial pH values ($pH > 6$), the Ni (II) ions become precipitated due to the hydroxide anions forming a nickel hydroxide precipitate. Therefore, a pH value of 6 was considered as the optimal pH value.

These results are supported by similar studies as will be explained in the following. Hasar (2003) and Krishna and Swamy (2011) studies showed that, at lower pH values, nickel removal was inhibited possibly because of the competition between hydrogen and nickel ions in the sorption site. The apparent dominance of hydrogen ions restricts the approach of metal cations as a consequence of the repulsive force. At a pH greater than 6, Ni (II) ions start precipitating as a result of hydroxide anions forming nickel hydroxide precipitate.

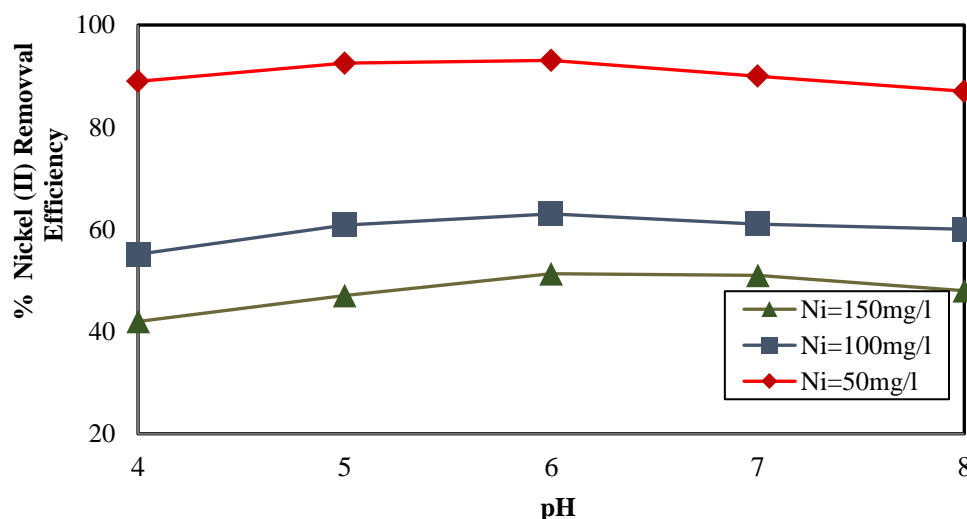


Figure 4

Effect of pH on the adsorption of Ni (II): IOCL adsorbent= 5 g/l, initial Ni (II) concentrations= 50, 100, and 150 mg/l, contact time= 120 mins, $T= 25\text{ }^{\circ}\text{C}$, and agitation rate= 200 rpm.

3.4. Effect of adsorbent dosage on Ni (II) removal

Figure 5 shows the percentage removal of nickel at an initial pH of 6 and different initial Ni (II) concentrations as a function of LECA adsorbent load. It is evident from this figure that the nickel removal from the solution takes place more rapidly by increasing IOCL doses because more surface area becomes available for nickel (II) ions and therefore more binding sites are available for Ni (II) adsorption.

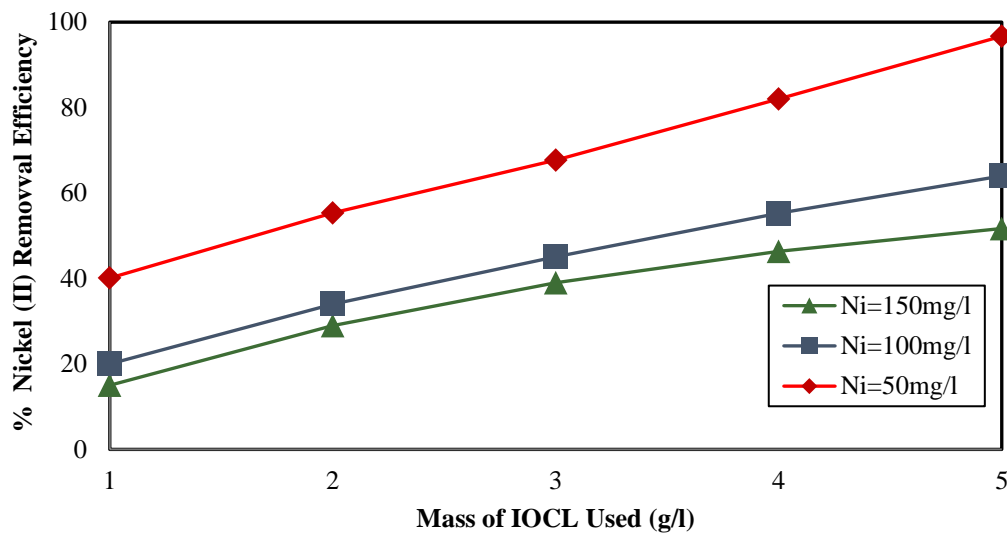


Figure 5

Ni (II) sorbed versus the dose of IOCL adsorbent: pH= 6, initial Ni (II) concentrations= 50, 100, and 150 mg/l, contact time= 120 mins, $T= 25^{\circ}\text{C}$, and agitation rate= 200 rpm.

3.5. Sorption kinetics

This experiment was organized to determine the equilibrium time for Ni (II) binding to the modified LECA. Figure 6 shows the rate of Ni (II) sorption on IOCL as a function of time at an initial pH of 6, an initial Ni (II) concentration of 50 mg/l, a sorbent dosage of 5 g/l, and a temperature of 25 °C. It indicates that the rate of Ni (II) sorption on IOCL decreases with increasing reaction time. In addition, this figure discloses that equilibrium is achieved approximately after 4 hrs. The rate of changes happened within the first hour is the highest followed by small rate of changes and leveling off in later times. Figure 6 demonstrates that more than 85% of nickel ions are adsorbed on IOCL within one hour. The rapid binding of the Ni (II) to IOCL may indicate that nickel is being adsorbed onto the surface of the sorbent. Equilibrium is slowly approached within the next 3 hours.

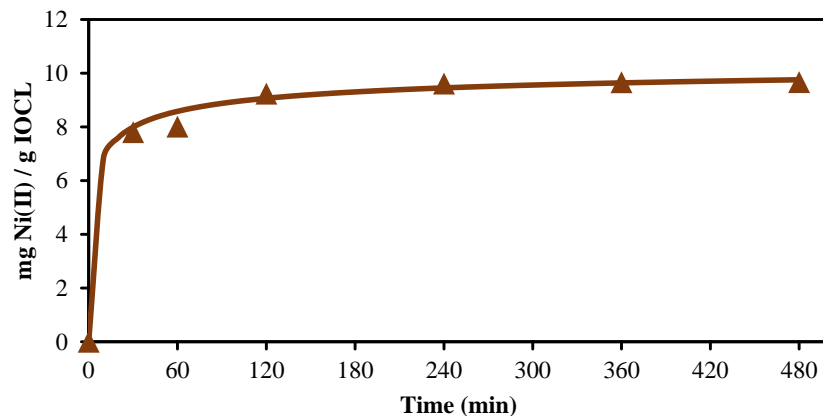


Figure 6

Nickel sorption by IOCL as a function of reaction time: Initial concentration= 50 mg/l, pH = 6.0, sorbent dose= 5.0 g/l, agitation speed= 200 rpm, and $T= 25^{\circ}\text{C}$.

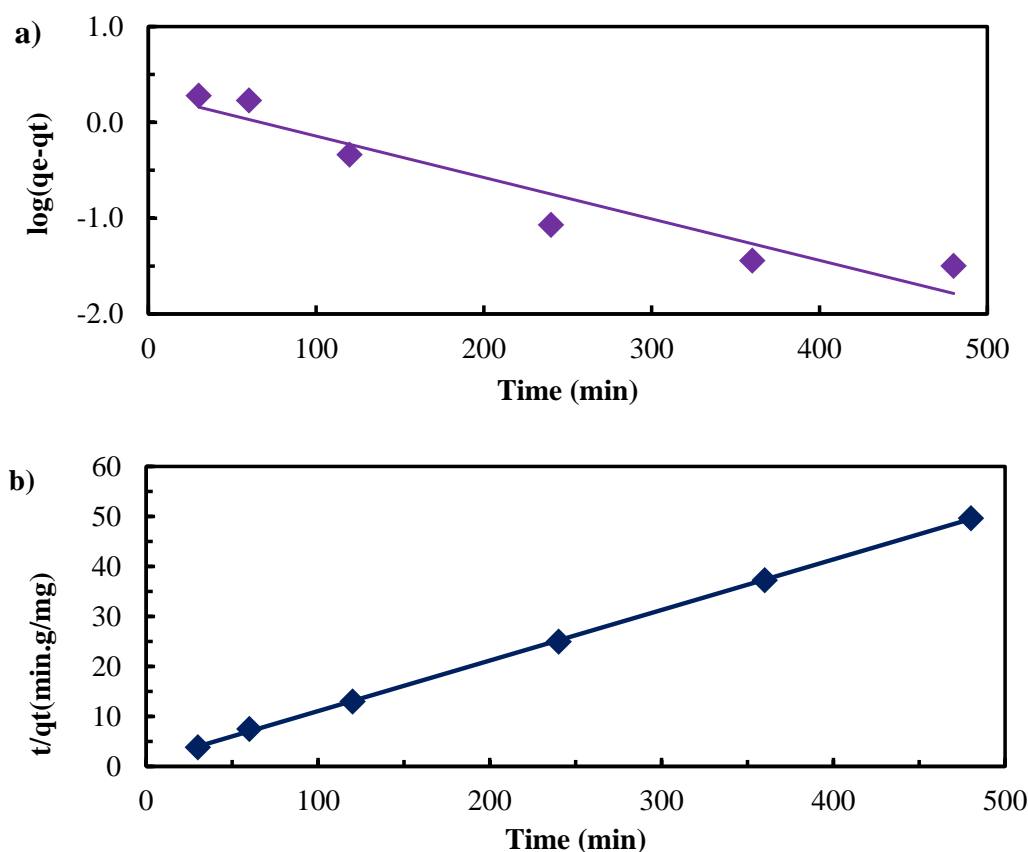


Figure 7
Sorption kinetics of Ni (II) by IOCL adsorbent: a) pseudo first order equation and b) pseudo second order equation.

Figure 7 demonstrates the plots of the kinetic model equations used. The values of rate constant and calculated Ni (II) adsorption capacity ($q_{e,cal}$) of the pseudo first-order kinetic model were obtained from the slope and intercept of the plot of $\log(q_e - q_t)$ versus time and are summarized in Table 2. Based on Table 2, the correlation coefficient of pseudo-first-order model is 0.9131 and the calculated Ni (II) adsorption capacity (1.94 mg/g) is very different from the experimental data (9.70 mg/g). However, the R^2 value of pseudo-second-order kinetic model (0.9998) is higher and this model matches the experimental data much better. In addition, the calculated Ni^{2+} adsorption capacity (9.90 mg/g) agrees very well with the experimental value. Therefore, the results obtained from the kinetic experimental data of Ni (II) sorption on the modified LECA follows a pseudo second-order kinetic model.

Table 2
The kinetic parameters for the adsorption of Ni (II) on IOCL.

Pseudo-first order model					Pseudo-second order kinetic model			
C_0 (mg/l)	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	K_1 (min^{-1})	R^2	$q_{e,cal}$ (mg/g)	K_2 (g/mg.min)	h mg/g.min	R^2
50	9.70	1.94	0.0099	0.9131	9.90	0.0104	1.0214	0.9998

4. Conclusions

This work indicated that the investigated adsorbent was efficient, low cost, and locally available for treating Ni (II) from contaminated water and industrial effluents. Moreover, since the price of LECA compares favorably with other natural sorbents, it can be used as an effective alternative material for treating industrial effluents. The extent of Ni (II) removal efficiency enhanced with increasing contact time and sorbent dosage. Moreover, increasing the initial pH of the solution showed a positive impact on the Ni (II) removal efficiency up to a value of 6. The removal efficiency of 92.5% was found at a contact time of 2 hrs, an initial pH of 6.0, and a sorbent dosage of 5 g/l. However, the removal efficiency of 96% by using IOCL sorbent occurred at a contact time of 4 hrs, which was very close to the equilibrium value at the mentioned pH and dosage. The experimental results obtained from the kinetic investigation were best fitted by a pseudo second-order kinetic model.

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Nomenclature

C_0	: Ni (II) initial concentration
C_t	: Ni (II) residual concentration at time t
FTIR	: Fourier transform Infrared spectroscopy
h_0	: Initial adsorption rate
IOCL	: Iron oxide coated LECA
K_1	: Rate constant of pseudo-first-order adsorption process
K_2	: Rate constant of pseudo-second-order adsorption process
LECA	: Lightweight expanded clay aggregate
LOI	: Loss of ignition
NL	: Natural LICA
q_e	: Ni (II) adsorbed at equilibrium (mg/g adsorbent)
$q_{e,cal}$: Calculated Ni (II) adsorption capacity (mg/g adsorbent)
$q_{e,exp}$: Experimental Ni (II) adsorption capacity (mg/g adsorbent)
q_t	: Ni (II) adsorbed at time t (mg/g adsorbent)
RE	: Removal efficiency
SEM	: Scanning electron microscopy
XRF	: X-ray fluorescence spectroscopy
WHO	: World Health Organization

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