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Evaluation of adsorption efficiency of raw and modified pistachio hard skin in removal of Ni(II) from the contaminated solution

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ABSTRACT

The nickel(II) adsorption efficiency by raw and acid modified pistachio hard skin an eco-friendly, low-cost, abundantly available sorbent is evaluated in the present study. The effects of different factors such as contact time, pH, initial nickel concentration, and adsorbent dose were investigated in the batch experiments. The characteristic of raw and acid modified sorbents were confirmed by FTIR, SEM study showing the change in surface properties. Of process factors, pH has important role in the removal efficiency and the highest removal (83%) was obtained at pH 7.0 due to the contribution of electrostatic interactions. The experimental batch adsorption data were fitted by Langmuir isotherm model and maximum adsorption capacity was obtained 19.37 mg/g. Results of the isotherm model study indicated the adsorption process was favorable at optimum condition. Present investigation concluded that acid modified pistachio skin may be applied as a low-cost attractive adsorbent for removal of metal ions from aqueous solution

Keywords: Pistachio hard skin; Acid modification; Adsorption isotherm; Nickel removal; FTIR study

1. Introduction

The presence of some heavy metals in the environment, even at very low concentrations due to the nature of toxicity and the desire to bio-accumulation in the food chain, is an important issue for human societies [1]. Among heavy metals, nickel has one of the most use in the industries of stainless steel, alloys, batteries, and so on. Some nickel compounds such as carbonyl are carcinogenic and easily absorbed by body [2]. Acute intoxication with nickel causes headache, dizziness, nausea and vomiting, chest pain, chest tightness, nausea and shortness of breath, rapid respiration, cyanosis and severe weakness [3]. Lung, nose, and bone marrow cancer is a poisoning complication of exposure to high concentrations of nickel [4]. The maximum permissible amount of nickel concentration in wastewater for discharging to environment is 2 $mg·L^{-1}$ and its permissible limit in drinking water is 0.5 $mg.L^{-1}$ by the WHO. To achieve these standards, wastewater from the industrial activities must be treatment before be discharged to the environment. Different methods are used to remove metal ions from aqueous media such as filtration, chemical precipitation, ion exchange, electrochemical purification, sedimentation and adsorption [5]. The adsorption process is one of the common methods to remove heavy metal ions due to its simplicity, convenience and high removal efficiency. Also, the development of adsorbents with high adsorption capacity, the fast adsorption kinetics, the separation and easy reconstruction are necessary for wastewater treatment [6]. For

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this reason, the use of cheap materials has been considered as adsorbent for the removal of heavy metals form aquatic environment.

In recent years, agricultural by-products, due to include different groups on the surface, have been widely used as adsorbents for the removal of heavy metals from the polluted water. These adsorbents include plant wastes, wood, pine bark, banana stalk, rice bran, soybeans cotton seeds, peanuts, peanut butter, hazelnut, rice peel, sawdust, orange peel, coconut shell, tea leaf, chitosan, corn, seaweed, etc. [4,5,7]. One of the agricultural wastes is pistachio hard skin that is abundance in Iran. Iran Almost 86 % of the world's pistachio production and 47% of world pistachio exports [7,8]. Therefore, using by-product of pistachio as adsorbents diminishes environmental pollution and is an eco-friendly process.

Necessity to use the economical, efficient and environmentally friendly methods for removal of pollutant, this study aimed to improve the surface properties of pistachio hard skin by simple chemical modification, to evaluate its removal efficiency for Ni(II) from aqueous solutions, to determine the adsorption isotherm model as one of the important factors in the design of adsorption systems for determining the maximum adsorption capacity and to optimize the effective factors on the adsorption properties.

2. Method

2.1. Materials and instruments

In this research, chemical substances including nitric acid (HNO_3), nickel nitrate six hydrates ($Ni(NO_3)_2·6H_2O$), sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) with high purity were used to prepare the desired solutions. To adjust the pH, 0.1 N sodium hydroxide or sulfuric acid solution were used. The main solution of Ni(II) with a concentration of 1000 ppm was prepared by dissolving of 4.954 g of nickel nitrate six hydrates with distilled water. The desired concentration of the standard and working solutions were prepared from the main solution to carry out adsorption experiments.

The 20AA atomic absorption spectrophotometer (AAS) was used to measure residual nickel ions. A digital laboratory scale were used to weigh the material and a pH meter (Sheng) were used to measurement and adjustment the pH solutions. Investigation of the properties of adsorbent surface structure was performed by scanning electron microscope (SEM, Cam Scan, MV 2300). To determine the functional surface groups participating in the adsorption process used a fourier transform infrared spectrophotometer (Bruker CO. Tensor 27, Germany) at a range of 400–4000 cm^{-1} and a comparison was made between pistachio peanut granules before and after modification. For well contact of adsorbent and nickel ions in the suspension, a programmable shaker was used with a suitable rate mixing (150 rpm) at different times.

2.2. Preparation of adsorbent

Pistachio hard skin was prepared from a pistachio processing unit in Kerman, Iran. In order to prepare the adsorbent, first, the pistachio hard skin was crushed by electric mill, sized between 40–18 mesh, and then granules with the sizes of 0.20–0.25 mm were selected for testing. Then, granules were washed with distilled water several times to remove impurities and dried in a furnace at 70°C for 24 h. To modify pistachio skin, 30 g of granules were refluxed in 100 ml of nitric acid (2 M) for 1 h at 70°C and washed several times with distilled water. To remove the remaining nitric acid, the granules were soaked in sodium bicarbonate for 12 h, washed with distilled water, and then dried at 70°C for 24 h.

2.3. Determination of sorbent zero point charge pH (pH_{zpc})

The pH zero charge point of sorbent was determined to evaluate the adsorbent surface property. For this purpose, a value of 50 ml of 0.01 M sodium chloride solution was added to the 6 Erlenmeyer, and the initial pH for each of the solutions was adjusted by 0.1 N or 1 N sulfuric acid or sodium hydroxide to adjust pH 2 to 12. Then 0.2 g of the adsorbent was added to each of Erlenmeyer. After 24 h, the final pH of the solution was measured with pH meter. Finally, pH_{zpc} was determined by plotting the final pH via the initial pH value [9].

2.4. Batch adsorption experiments

Adsorption experiments were carried out in a batch system using 100 ml Erlenmeyer as an adsorption reactor. Effective factors in the adsorption process (pH, contact time, adsorbent dose and initial nickel concentration) were changed and optimized by single-variable method. The effect of initial pH was evaluated on adsorbent at initial pH 3 to 8, the effect of contact time on nickel ion adsorption with different contact times (5–60 min), the effect of adsorbent dose with different adsorbent amounts (0.1–1 g), and the effect of initial nickel concentrations with different concentrations of nickel ion (10–100 mg /L) were tested by 21 batch experiments, separately. The detail of tests conditions are presented in Table 1.

All experiments were carried out in a constant volume of 50 ml and laboratory temperature (25 ± 2°C). At the end time of the each experiment, suspension was taken from the shaker and filtrated with a cellulose filter paper (0.45 µm) for all of experiment. Then, after the calibration AAS, the residual nickel ion concentration was measured in the filtrated solutions by atomic absorption spectrophotometer at 352.5 nm. To ensure results, the absorption of each sample was read three times with the atomic absorption spectrophotometer and the mean concentration was used to calcu-

Table 1
Detail of tests conditions

Parameter	Values
Solution pH	3, 4, 5, 6, 7, 8
Contact time, min	5, 10, 15, 30, 60
Adsorbent dose, g/100 ml	0.1, 0.2, 0.4, 0.8, 1
Initial Ni concentration, mg/L	10, 20, 40, 80, 100

late the percent of nickel ion removal (R %) and the amount of the adsorbed nickel in equilibrium (q_e) as the Eqns. (1) and (2), respectively [10]:

$$R\% = \frac{\left(C_i - C_e\right)}{C_i} \times 100 \tag{1}$$

$$q_e = \frac{\left(C_i - C_e\right)}{m} V \tag{2}$$

In these equations C_i and C_e are the initial and final concentrations of Ni(II) (mg·L^{-1}), q_e is the amount of the adsorbed nickel ions on the unit of adsorbent (mg/g), m is the amount of adsorbent (g), and V is the volume of the Ni(II) solution (L).

2.5. Adsorption isotherms

The parameters obtained from various adsorption isotherm models provide important information on the adsorption mechanism, the surface properties of the adsorbent and internal bonds [10]. In this study, three main isotherms of Langmuir, Freundlich, and Temkin were selected to describe the adsorption isotherm [11].

The Langmuir model assumed adsorption on the homogeneous adsorbent sites with single-layer adsorption. The linear form of Langmuir isotherm is expressed by Eq. (3):

$$\frac{C_e}{q_e} = \left(\frac{1}{Q_m b}\right) + \left(\frac{1}{Q_m}\right) C_e \tag{3}$$

C_e (mg·L^{-1}) is the equilibrium concentration of the residual metal ion in aqueous solution and q_e (mg·g^{-1}) is the amount of ion adsorbed on the solid phase. Q_m (mg·g^{-1}) is the maximum single-layer adsorption capacity by adsorbent and b is the Langmuir adsorption constant [12].

Freundlich isotherm is usually used to describe the adsorption properties for heterogeneous surfaces. The linear Freundlich model is according to Eq. (4):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{4}$$

The constant K_F (mg·g^{-1}) is the Freundlich isotherm, n is the adsorption intensity, C_e (mg·L^{-1}) is the equilibrium concentration in solution, and q_e (mg·g^{-1}) is the adsorbate amount in one gram of adsorbent at equilibrium. The constant K_F is an approximation of the adsorption capacity, and $1/n$ represents the adsorption power for the adsorption process [13].

Temkin's adsorption isotherm model considers the interaction between adsorbent-adsorbate. The linear form of the Temkin isotherm is expressed in Eq. (5):

$$q_e = B_T \ln K_T + B_T \ln C_e \tag{5}$$

where $B_T = RT/b$ and k_T are constant. R (8.31 J·mol^{-1}·K^{-1}) is the gas general constant and T (K) absolute temperature, K_T (L·mg^{-1}) is equilibrium bond with maximum transition energy and constant B_T is the adsorption heat.

3. Results and discussion

The raw and modified pistachio hard skin as granule are shown in Figs. 1a and b. The comparison of two images indicates that the acid modification cause the modified granules changes to bright red color.

3.1. Adsorbent properties

The scanning electron microscope (SEM) image of the surface structure of the raw and nitric acid-modified pistachio granules are presented in Figs. 2a and 2b, respectively. As shown in Fig. 2b, chemical modification with HNO_3 has led to change the surface and opening pores on the adsorbent. This proved that HNO_3 had the potential to modify the surface structure of adsorbent by using its chemical properties in the degradation of pistachio skin surface and increasing porosity of the adsorbent surface [14]. On the other hand, acid modification can dissolve the minerals in the adsorbent pores, so the pore size and adsorbent surface increase after modification. As a result, the hot acid modification of pistachio granules can be presented the high adsorption capacity compared to the raw pistachio granules.

The pattern of metal adsorption on natural adsorbents is attributed to active functional groups at the adsorbent surface

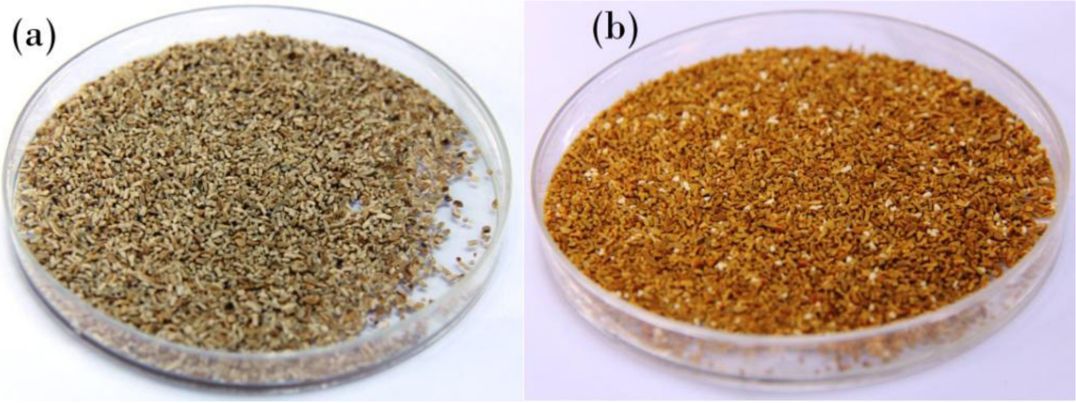


Fig. 1. (a) and (b) raw and nitric acid modified adsorbent granules.

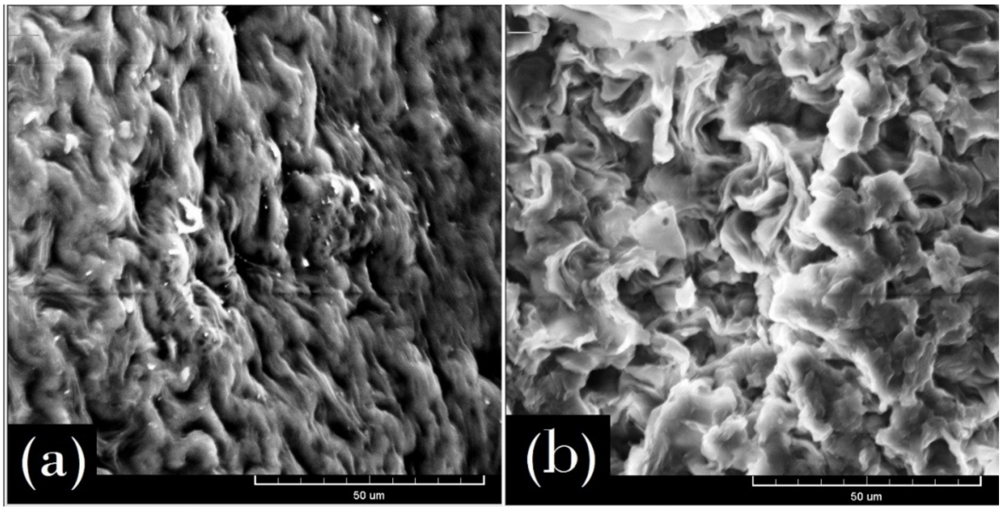


Fig. 2. SEM image raw pistachio granules (a) nitric acid-modified granules (b).

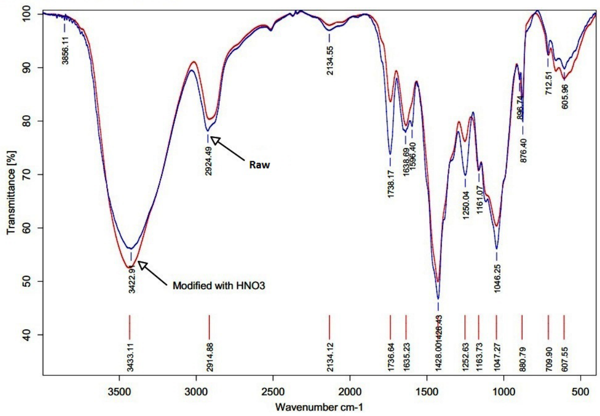


Fig. 3. FTIR spectra of raw and nitric acid-modified pistachio granules.

[15]. The FTIR analysis was used to study the main groups responsible to adsorb ions. Fig. 3 shows the FTIR spectrum of raw and nitric acid-modified granules. Major functional groups in raw and acid modified pistachio granules were identified and reported in Table 2. The peak of 3422 cm^{-1} is due to inter or intra molecular hydrogen bond of the O-H and N-H group. The stretching vibrations of O-H indicate the presence of free hydroxyl groups attached to carboxylic acid which treatment with HNO_3 released some hydroxyl and carboxylic groups bonding with metal ions [16]. The peaks at wave numbers of 1250 and 1046 cm^{-1} are attributable to the stretching vibration of C-O and C-N from the phenyl alkyl ether and amine groups. The peaks observed at a wave number of 1161 cm^{-1} are related to the stretching vibrations of the C-O from the alcohol group. These results showed that the hydroxyl groups, ester, ether, amines and alcohol are the main groups of oxygen in the pistachio skin. The studied FTIR spectrum is approximately the same as the results of the study entitled effect of thermal activation on the textural and chemical properties of potassium hydroxide activated carbon prepared from pistachio nut shell.

Comparing of the FTIR spectrum results before and after treatment with acid showed that the intensity of all absorption frequencies has changed after modification as are presented in Table 2. Therefore, it seems that the adsorbent was well affected by nitric acid in modification process without to be significantly changed the adsorbent chemical structure.

Determining the zero point charge pH of an adsorbent in solution is essential and helps to better understand the adsorption mechanism. As seen from Fig. 4, the zero point charge pH of the acid modified granule was obtained 6.46 which is suitable pH to adsorb cations.

According to Fig. 4, at pH < pH_{zpc} the adsorbent surface has a positive charge due to high concentration of H^+ ions. At this state, electrostatic repulsion occurs between positive metal ions and surface positive charge of adsorbent which decreases the metal ion adsorption from solution. When the solution pH is higher than the pH_{zpc} (pH > 6.35), the adsorbent surface has the negative charge. At this condition, electrostatic interaction increases between positive ions and negative surface charge of adsorbent and so, an increase is observed in the adsorption of cations from the solution. Therefore, at pH higher than pH_{zpc}, nickel ion adsorption is faster than other pHs. Therefore, the batch experiments were done at the pH of 7, further.

3.2. Effect of pH on the nickel removal efficiency

As mentioned above, the initial pH of the solution is one of the most important factors in adsorbing metal ions on the adsorbent. It affects the ionization degree of metal ions, adsorbent surface functional groups and determines the adsorbent surface charge in the solution. Proper pH selection optimizes adsorption parameters and increases the efficiency of the adsorption process. The effect of pH on the removal efficiency of nickel ions by the raw and acid modified pistachio granules are presented in Fig. 5.

The results showed that increasing the pH from 3 to 8, the removal efficiency of raw adsorbent changed from 2.1% to 69.4% while for acid modified was higher than raw at all pHs. This is also predictable with respect to Eqs. (1)–(3).

Table 2
Assignment of FTIR spectra to the functional groups in raw and nitric acid-modified pistachio granules

Wave number (cm^{-1})			Functional groups
Raw granules	Modified granules	Changes	
3422.91	3433.11	+10.20	N-H and O–H stretching
2924.49	2914.88	–9.61	C–H stretching
2134.55	2134.12	–0.43	C≡C stretching
1738.17	1736.64	–1.43	C=O stretching
1638.69	1635.23	–3.46	C=C stretching
1426.43	1428.01	+1.58	CH_3 and CH_2 bending
1250.04	1252.63	2.59	C-N and C–O stretching
1161.07	1163.73	2.66	C–O stretching
1046.25	1047.27	1.02	C–O stretching
876.40	880.79	4.39	=C–H out-of-plane bending
712.51	709.9	–2.61	=C–H out-of-plane bending

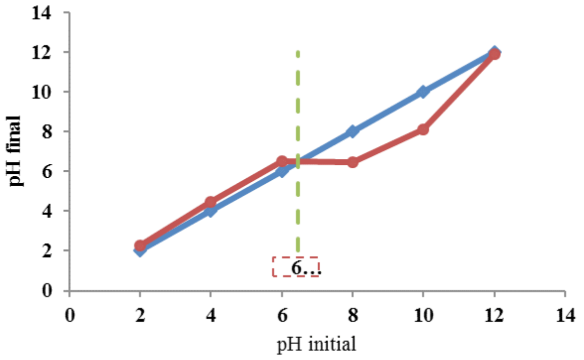


Fig. 4. The pH_{zpc} of nitric acid modified pistachio granules.

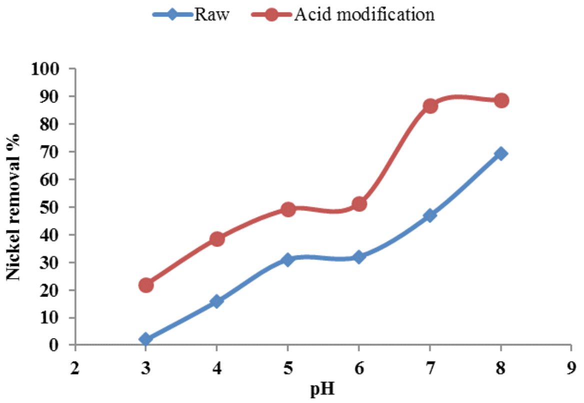
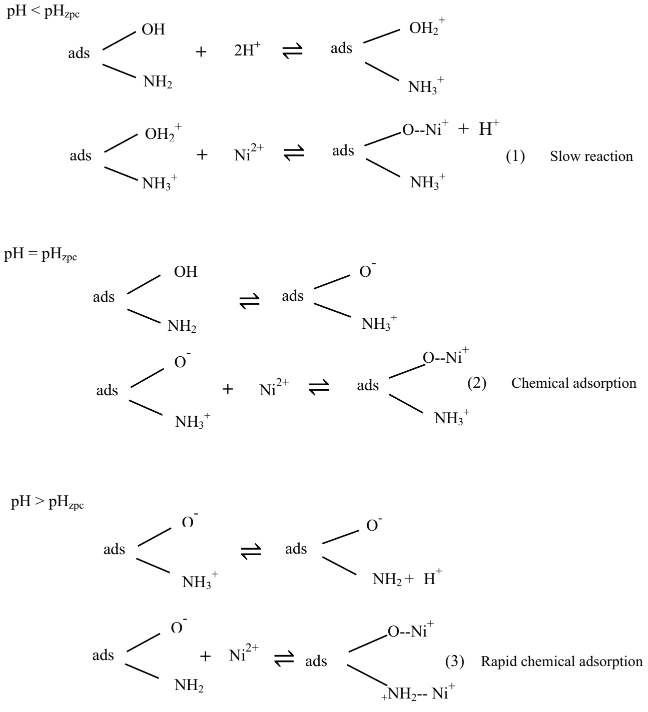


Fig. 5. The effect of solution pH on the nickel removal efficiency.

At low pH, high concentration of hydrogen ions in aqueous medium with a positive charge of metal ion is competing for binding to adsorbent sites which lead to a decrease in the removal of metal ion (reaction 1). In other word, in acid conditions, metal ions and adsorbents have a positive charge that causes electrostatic repulsion between them [17]. By increasing the initial pH, the concentration of H^+ ions decreases and more nickel ions can react with the free active sites of the adsorbent. The increase in the nickel uptake capacity with increasing pH is due to a decrease in the concentration of hydrogen ion (reaction 2). The highest removal efficiency was obtained at pH higher than 6.46 (pH 7), which can interpret according to reaction 3, indicates the high adsorption tendency to adsorb the nickel ions. Therefore, the optimum pH solution for nickel removal was in the range of 7, and other tests were performed at this pH. Similar results were obtained by a study of Ghasemi et al. for investigation the ability of treated peganum harmala-L seed to remove nickel ion from aqueous solution. Despite the removal efficiency increased at pH 8, 9, 10, due to hydroxide precipitation at high pHs, optimal pH was considered to be 7 [18].



3.3. The effect of adsorbent mass on nickel removal efficiency

Fig. 6 shows the effect of adsorbent mass on the Ni(II) removal efficiency from aqueous solutions at 0 until 60 min contact times. As shown in Fig. 6, by increasing the amount of adsorbent in the range of 0.1 g to 1 g in 50 ml solution, the removal efficiency increased from 9% to 83% at the contact time of 60 min. Also, the results indicated that by increasing the amount of adsorbent mass, due to the greater surface area, the number of active sites increases for the complexation of ions. When the amount of adsorbent reaches its maximum efficiency (1 g in 50 ml), the number of active sites available on the adsorbent surface is sufficient to adsorb the number of ions in the solution at equilibrium adsorption process. Therefore, the optimum amount for adsorbent was considered 1 g/50 ml with 83% removal efficiency.

When the adsorbent dose is constant in a batch adsorption system, the number of active sites will remain constant [19]. The high initially rate adsorption is due to the availability of active sites at the adsorbing on the surface. Adsorption occurs quickly at the first times and is typically controlled by the diffusion process from solution to adsorbent surface. In the next step, low change in adsorption is probably due to the decrease of active sites available at the adsorbent surface [20]. Also, since the number of active adsorption sites in a batch system is constant, and one active site can adsorb only one ion in a single layer, adsorption of ions on the adsorbent surface will be rapid at first of process and when the residual competition ion in the solution intensifies to access the active sites, the adsorption of the metal ions are slowed down [21].

As contact time increases, removal efficiency increases until the equilibrium occurs between adsorption and desorption process. Equilibrium time is one of the most important parameters in the design of treatment systems. Fig. 6 shows the removal of nickel ion was reached to maximum at 60 min and indicates the rate of nickel ion removal was rapid at the beginning of adsorption process. It was gradually decreased to a time of equilibrium and then no significant increase was observed in the adsorption rate after 30 min at all of the adsorbent mass. Hence, the 60 min contact time was chosen as optimal time for adsorption of nickel. The observed fast adsorption is probably due to the high availability of active sites on the adsorbent surface. These sites are gradually occupied by the adsorbate and the surface adsorption efficiency decreases. At equilibrium, active sites are saturated and the adsorption process continues through diffusion [22]. Lam et al. used Lunsium domesticum peel as a biosorbent in removing Ni(II) from aqueous solution. Their result showed that the optimum pH for adsorbing Ni(II) ions was 6 and the appropriate contact time was 30 min to achieve equilibrium [23]. Another research was conducted in 2009 by Kumar et al. to investigate the adsorption behavior of Ni(II) on cashew nut shell. Their results showed that optimum pH was 5 and the adsorption rate was reached to equilibrium after 30 min. An increasing the contact time after 30 min had little effect on the nickel (II) adsorption [24].

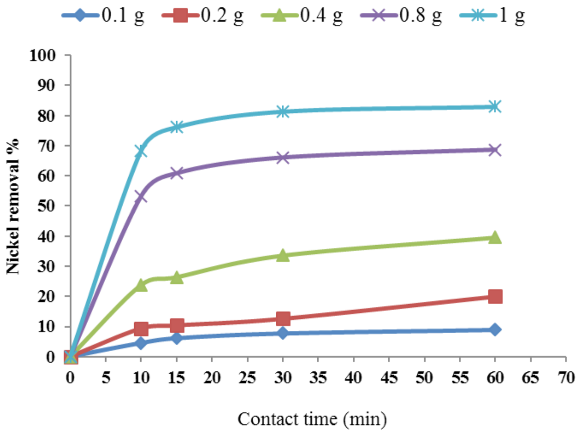


Fig. 6. The effect of adsorbent mass on the nickel removal efficiency.

3.4. Effects of the initial nickel concentration on the nickel removal efficiency

In a batch adsorption process, the initial contaminant concentration in solution provides the necessary forces to overcome the mass transfer resistance between the soluble and solid phases [25]. To achieve this purpose, the effect of the initial nickel concentration was investigated on removal efficiency of raw and nitric acid modified pistachio hard skin that are presented in Fig. 7. As shown in Fig. 7, nickel removal decreases with increasing nickel ion concentration. At low concentrations of 10 and 20 mg·L^{-1} of nickel ion, about 100% nickel ion was eliminated at optimal contact time of 30 min. By doubling the concentration of nickel from 40 to 80 mg·L^{-1}, the removal efficiency decreased from 82.6% to 33.5%. This is due to the fact that adsorbents have a limit adsorption sites and so an increasing the concentration of pollutant causes their capacity is quickly saturated and the removal efficiency decrease [26]. According to Fig. 7, the equilibrium time in all states is 30 min and the plots are almost similar. This means that the equilibrium time is not influenced by the initial concentration which reflects the fact that the Ni(II) ions form a single layer at the external surface of the adsorbent and the adsorbent surface has a number of stable active sites.

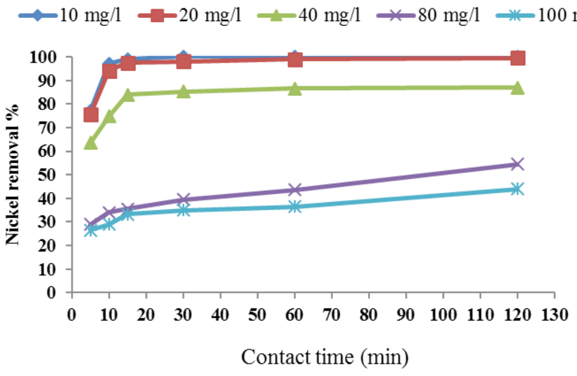


Fig. 7. The effect of initial concentration on the nickel removal efficiency.

3.5. Adsorption isotherm study

Adsorption isotherm is an important factor in design of adsorption systems. In fact, the isotherm are distinguished that how interactions are occurred between adsorbent and adsorbed, hence it is always considered as an essential factor in determining the adsorption capacity. The empirical adsorption data were analyzed with linearized three Langmuir, Freundlich and Temkin isotherms. The parameters of each model with its correlation coefficient are listed in Table 3. The higher correlation coefficient ($R^2 = 0.994$) of the Langmuir isotherm model indicate the higher applicability of this model in describing for nickel removal by the modified adsorbent. Also, it indicates that the adsorption process occurs as monolayer onto the surface of adsorbent [17].

The main parameter of the Langmuir isotherm is a dimensionless Langmuir constant (R_L) called the equilibrium parameter, defined by the Eq. (6).

$$R_L = \frac{1}{\left(1 + bC_0\right)} \quad (6)$$

where C_0 is the initial nickel concentration and b is the Langmuir isotherm constant. The process feasibility is considered by the R_L values as: $0 < R_L < 1$ for the desirable process; while $R_L = 0$ for irreversible condition, $R_L = 1$ for the linear state, $R_L > 1$ for the undesirable adsorption process. The R_L for adsorption of Ni(II) by acid modified pistachio skin was obtained 0.08 in optimum condition that indicates the adsorption process is favorable for this condition.

Since the correlation coefficient of Freundlich model is 0.915, so the Freundlich model relatively fitted with experimental results for adsorption of nickel by acid modified pistachio skin. In this model, the value of n in Freundlich adsorption isotherm shows a deviation from the linearity. If $n = 1$, adsorption is homogeneous and there is no interaction between adsorbed species. If $1/n < 1$, adsorption is desirable and new adsorption positions are generated. If $1/n > 1$, adsorption is undesirable, the bonding is weak and the adsorption capacity decreases. According to Table 3, the $1/n$ was 0.19 and so the adsorption of Ni(II) by acid modified pistachio skin was desirable in optimum condition.

Considering, the high value of R^2 (0.985), the data of nickel adsorption onto pistachio skin had well fitted with Temkin isotherm and the equilibrium adsorption properties can also be described by the Temkin isotherm. In addition, Temkin adsorption isotherm was chosen to. The liner plots of q_e versus lnC_e determine the B_T and K_T constants from the slope and intercept, respectively. The parameters K_T and B_T of the Temkin equation were calculated for nickel adsorption (Table 3). It can be seen that the modified pistachio skin adsorbent is energetically suitable for removal of cationic heavy metals such as Ni(II) with a relatively large B_T.

4. Conclusion

The results presented in this study indicated that raw and modified pistachio shell can be successfully used as biosorbent for the removal of heavy metals Ni(II) from aqueous solution. The modification of raw pistachio skin with nitric acid increased its porosity and pH_{zpc}. This modification resulted in higher adsorption of nickel as compared to the raw sorbent. The IR study indicated that the most important functional groups in removal of nickel were carbocyclic, amine, and hydroxyl groups. The Langmuir isotherm model was the most adequate method in describing the distinguishing of the biosorption process. Use of this biosorbent in the nickel removal process will be helpful for environmental protection which is important in the conception of sustainable development strategies.

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Table 3
Linear equation and adsorption isotherm parameters for nickel removal by acid modified pistachio adsorbent

Isotherm	Langmuir			Freundlich			Temkin		
Linear equation	$\frac{C_e}{q_e} = \left(\frac{1}{Q_m b}\right) + \left(\frac{1}{Q_m}\right) C_e$			$\log Q_e = \log K_F + \frac{1}{n}\log C_e$			$q_e = B_T \ln K_T + B_T \ln C_e$		
Parameters	R^2	b	Q_m	R^2	n	K_F	R^2	B_T	K_T
Obtained values	0.994	5.73	19.37	0.915	5.23	9.38	0.984	232.18	0.286

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