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Ethylenediamine-functionalized cubic ZIF-8 for arsenic adsorption from aqueous solution: Modeling, isotherms, kinetics and thermodynamics

Mohamadreza Massoudinejad [a,b], Mansour Ghaderpoori [c,d,*], Abbas Shahsavani [b,e], Ali Jafari [d], Bahram Kamarehie [d], Afshin Ghaderpoury [f], Mostafa M. Amini [g]

[a] *Safety Promotion and Injury Prevention Research Center, Environmental Health Engineering, School of Public Health, Shahid Beheshti University of Medical Sciences, Tehran, Iran*
[b] *Department of Environmental Health Engineering, School of Public Health, Shahid Beheshti University of Medical Science, Tehran, Iran*
[c] *Department of Environmental Health Engineering, School of Health and Nutrition, Lorestan University of Medical Sciences, Khorramabad, Iran*
[d] *Nutritional Health Research Center, Lorestan University of Medical Sciences, Khorramabad, Iran*
[e] *Environmental and Occupational Hazards Control Research Center, Shahid Beheshti University of Medical Science, Tehran, Iran*
[f] *Students Research Committee, Shahid Beheshti University of Medical Sciences, Tehran, Iran*
[g] *Department of Chemistry, Shahid Beheshti University, G.C., Tehran 1983963113, Iran*

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ABSTRACT

Metal-organic frameworks are novel adsorbents that have been widely used in recent years. The main properties of this emerging adsorbent include high specific surface area, significant porosity, regular structure and adjustable cavities. The main objective of this study was ZIF-8 synthesis with cubic structure, functionalization with ethylenediamine and evaluation for arsenic adsorption from aqueous environments. All the experiments were carried out in batch conditions. Main variables including pH, initial arsenic concentration, adsorbent dosage and contact time were evaluated in this work. In this study, Design of Experiment software was used for experimental runs order arrangement and better understanding of the variables effects on the process. The BET results showed that surface area of the synthesized ZIF-8 and ED-ZIF-8 was 910 m^2/g and 850 m^2/g, respectively. It was also revealed that before and after functionalization with ethylenediamine, ZIF-8 adsorbent was aminined at about 3 mmol/g. In addition, the findings showed that the arsenic adsorption capacity increased from 72 mg/g to 83.5 mg/g. The best pH for arsenic adsorption was neutral (pH $\approx$ 7). The co-existing of anions effect showed that bicarbonate, sulfate and chloride had the least inhibition effects on arsenic adsorption. This study showed that application of these class of adsorbents can be seriously addressed in solving some environmental challenges.

1. Introduction

Metal-organic frameworks are emerging class of absorbents that have been used extensively. These regular and porous compounds are composed of two main parts. These parts are metal cations and organic linkers [1]. Combining of these two parts in different conditions, can create an amazing compound useable for various fields. Some of their features have high specific surface, significant porosity, regular structure, adjustable cavities [2]. Metal-organic frameworks (MOFs) have various applications such as storage and separation of various gases, transferring of medicine in the patient's body, use as catalyst and adsorbent. One of the most important applications of this new class of materials is their use for pollutants removal from the environment. Of course, in past there application for environmental purification aspects were low, but in last few years, it has significantly increased. These compounds are used to remove some pollutants such as fluoride [2,3], malachite green [4], phosphate [5,6], Pb and Cd [7], Cr(VI) [8] and Hg [9].

One of the most important classes of MOFs is zeolitic imidazole frameworks (ZIFs) [10]. Accordingly, different types of ZIFs adsorbents have been synthesized such as ZIF-8 [11], ZIF-67 [12,13], ZIF-69 [14], ZIF-11 and ZIF-20 [15], ZIF-22 [16], ZIF-90 [17], ZIF-93 [18] and ZIF-95 [19]. ZIF-8 may have different morphologies depending on its synthesis method. So far, different morphologies have been reported for ZIFs including dodecahedral, cubic, and leaf-shaped [20].

There are many pollutants in environment with natural or manmade sources. In the past due to the abundance of water resources, polluted resources were easily ignored. But now, due to some reasons such as climate change and decrease of atmospheric precipitation as well as increase of population growth, this procedure is not reasonable. Therefore, polluted resources should be purified properly. Among various pollutants, arsenic has been considered as a problematic pollutant in some regions [21]. Arsenic is found in both natural and artificial origin in

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[*] Corresponding author at: Department of Environmental Health Engineering, School of Health and Nutrition, Lorestan University of Medical Sciences, Khorramabad, Iran.
E-mail address: ghaderpoori.m@lums.ac.ir (M. Ghaderpoori).

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water resources [22]. According to the WHO guidelines, the maximum allowable concentration of arsenic is 10 μg/l in drinking water [23,24]. The main objectives of this study were to: First, ZIF-8 synthesis with cubic morphology and its functionalization with ethylenediamine. Second, the evaluation of cubic ZIF-8 and ethylenediamine (ED) cubic ZIF-8 in adsorbing arsenic from aquatic environment.

2. Materials and methods

2.1. Materials

Zinc nitrate hexahydrate, ethylenediamine solution, 2-methylimidazole, ammonium hydroxide, and methanol were supplied by Sigma-Aldrich. All the chemicals used in the experiments applied without further purification.

2.2. Synthesis and post synthetic modification of ZIF-8

Cubic ZIF-8 was synthesized according to previous studies. The solvent used for the preparation of adsorbent in this work was distilled water, which has no harm to the environment [6]. In brief, 0.594 g zinc nitrate hexahydrate and 0.328 g 2-methylimidazole were mixed separately in 3 ml deionized water and 3.76 g ammonium hydroxide solution. After thorough mixing, zinc nitrate hexahydrate solution was slowly added to 2-methylimidazole solution and was stirred for 10 min. After a specified time, a white powder was formed which was separated using a centrifuge. In the next step, after cubic ZIF-8 structure confirmation by XRD and FE-SEM, functionalization process was performed. Before functionalization process, cubic ZIF-8 produced from the previous stage was dried for 24 h at 383 °C. For functionalization, a solution of 30% ethylenediamine was first prepared. Cubic ZIF-8 was placed in a Teflon bottle containing ethylenediamine solution (30%) and put in the oven. The autoclave was heated in an oven at 144 °C for 1 h and then 108 °C for 6 h. Finally, A light-yellow powder was formed which then was separated with a centrifuge. The final powder was washed with distilled water several times. Before the final use the light-yellow powder was dried at of 110 °C for 1 day for activation [25].

2.3. Ethylenediamine-functionalized cubic ZIF-8 nanoparticles

2.3.1. General characterization

The ZIF-8 and ED-ZIF-8 adsorbents characteristics were determined by Fourier Transform Infrared Spectroscopy (Spectrum Two model, PerkinElmer Company), X-ray Diffraction (X' Pert Pro model, Panalytical Company), Energy Dispersive X-ray Spectroscopy, Field Emission Scanning Electron Microscopy (SIGMA VP-500 model, ZEISS Company), and BET surface area and total pore volumes of the samples (BElSORP Mini model, Microtrac Bel Corp.) were determined from nitrogen adsorption isotherms at 77 K.

2.3.2. Adsorption studies

All adsorption experiments were carried out in batch conditions. Initially, the most important variables affecting the adsorption of arsenic were determined through literature review (Table ST-1, Supplementary data). These variables were pH (3—11), initial concentration (1–10 mg/l), adsorbent dosage (1–5 g/l), and contact time (5–60 min). The Design of Experiment software was used to design the experiments. Among the different methods, the Central Composite Design (CCD) method was chosen. According to this method 5 levels were investigated for each variable. The runs and levels of each variable are shown in Table ST (Table ST-2, Supplementary data). All experiments were carried out at the laboratory temperature. In order to provide constant conditions for experiments, the sample volume was considered to be 150 ml for all the experiments. The stirring speed was kept 300 rpm for the experiments. To measure the initial and final concentrations of arsenic, ICP-OES (Instrument Model: Varian VISTA-MPX) was used. Eqs. (1) and (2) were also used to determine the adsorbent arsenic and removal efficiency, respectively [26–28]:

$$q_e = \frac{V(C_o - C_e)}{M} \tag{1}$$

$$R,\% = \frac{(C_0 - C_t)}{C_0} \tag{2}$$

where, q_e (mg/g) is the equilibrium adsorption capacity, C_e is arsenic concentration at equilibrium (mg/l), V is the volume of solution (l) and M is the weight of adsorbent (g). Also, C_0 and C_e are the initial and equilibrium concentrations of arsenic in solution (mg/l), respectively. After obtaining optimum adsorption conditions, isotherms and adsorption patterns were calculated. In this work, Langmuir and Freundlich models were also used to determine the best isotherm model. In addition, pseudo-first-order and pseudo-second-order models were used to determine the best kinetic model [29,30].

3. Results and discussion

3.1. Characterization of as-synthesis ZIF-8 and ED-ZIF-8

The various experiments were carried out to determine the characteristics of the synthesized adsorbents. The XRD shows the crystalline structure of ZIF-8 and ED-ZIF-8. Fig. 1 shows the results of XRD. As shown in Fig. 1, the main peaks of ZIF-8 were very clear and similar to the original sample. The presence of strong peaks indicated good crystallization of the adsorbents. The XRD patterns of ZIF-8 and ED-ZIF-8 were very also similar to each other and have no particular difference. Actually, functionalization has no effect on the crystalline structure of ZIF-8 [25]. the XRD spectra showed that the main peaks were at angles of 2θ between 7, 10.33, 12.8, 14.64, 16.4 and 17.98. Fig. 2 shows the morphology of the synthesized ZIF-8 and ED-ZIF-8. As can be understood from the Fig. 2, ZIF-8 was synthesized into a cubic form. Depending on the synthesis conditions, the ZIF-8 can have different shapes like dodecahedral, cubic, and leaf-shaped [20]. The findings from various studies indicate that the surface area of adsorbents can vary depending on morphology, quality of raw materials and synthesis conditions. Furthermore, the results of BET showed that the surface area of ZIF-8 and ED-ZIF-8 were 910 and 850 m^2/g, respectively. In the Liu et al. [20] surface area of dodecahedral, cubic, and leaf-shaped were 1151.2, 958.4, and 12.7 m^2/g, respectively. After functionalization, the total pore volume was improved. The total pore volume of ZIF-8 and ED-ZIF-8 were 0.57 and 0.65 m^3/g, respectively. The reason for this increase is attributed to the opening of blocked spaces [31]. To analyze the functional groups on ED-ZIF-8, FTIR was used. Fig. 3 shows FTIR results of ZIF-8 and ED-ZIF-8. As shown, the FTIR spectrum of ZIF-8 and ED-ZIF-8 are almost identical, and only in 3381 band a new peak observed which was associated with the N—H group in the ED-ZIF-8. This pattern is consistent with the reference pattern [25]. The results of EDX showed that the percentage of C, N, Zn and O in the ZIF-8 was 49, 22, 27.7, and 1.54, respectively. The percentage of C, N, Zn, and O in the ED-ZIF-8 was 42.54, 30.24, 24, and 3.12, respectively. Also, The results before and after functionalization of ZIF-8 with ethylenediamine showed that the adsorbent was aminined at about 3 mmol per gram of adsorbent. Finally, the arsenic adsorption capacity increased from 72 to 83.5 mg/g of adsorbent (approximately 14% increase in adsorption capacity).

3.2. Statistical analysis and model fitting

The lack of fit (Table ST-3, Supplementary data) showed that the empirical data obtained from arsenic adsorption via ED-ZIF-8 could be described with a polynomial model. As ANOVA results showed (Table ST-4, Supplementary data), the model F value of 41.37 implied that the model was significant. There was only a 0.01% chance for F model

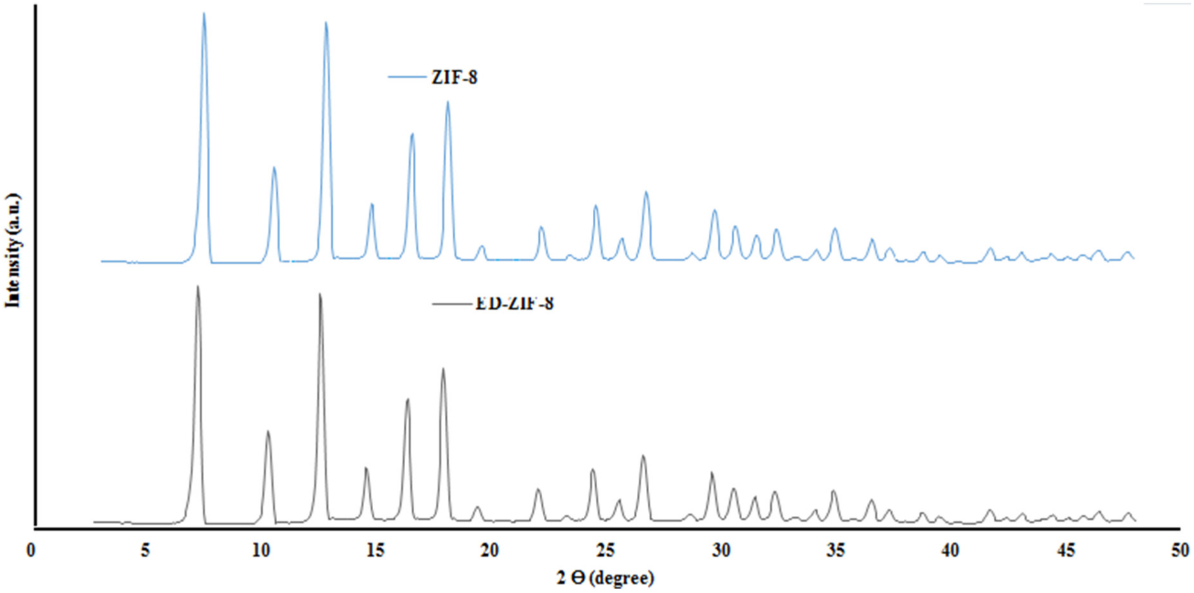

Fig. 1. XRD spectra of as-synthesis of ZIF-8 and Ed-ZIF-8.

value (this large could occur due to noise). The lack of fit F value of 2.39 implied the lack of fit is not significant relative to the pure error. There was a 17.32% chance for a lack of fit F value (large could occur due to noise). Non-significant lack of fit is good. The pred-R^2 of 0.8895 was in reasonable agreement with the adj-R^2 of 0.9476 (Table ST-5, Supplementary data). Adequate precision measures the signal to noise ratio. A ratio >4 is desirable. In this model, ratio of 23.566 indicated an adequate signal. This model can be used to navigate the design space. To obtain the optimum conditions of the arsenic adsorption on ED-ZIF-8, the main variables were selected. According to the selected model, the best adsorption conditions were arsenic concentration of 2.88 mg/l, pH 5, ED-ZIF-8 dosage of 0.2 g/l and contact time of 31.67 min. In optimum conditions, the efficiency of the adsorption process was 97% with desirability of 0.997. At the end of the modeling, validation should be done. To validate the results, the optimal conditions of the software were repeated. Validation results showed that the obtained results

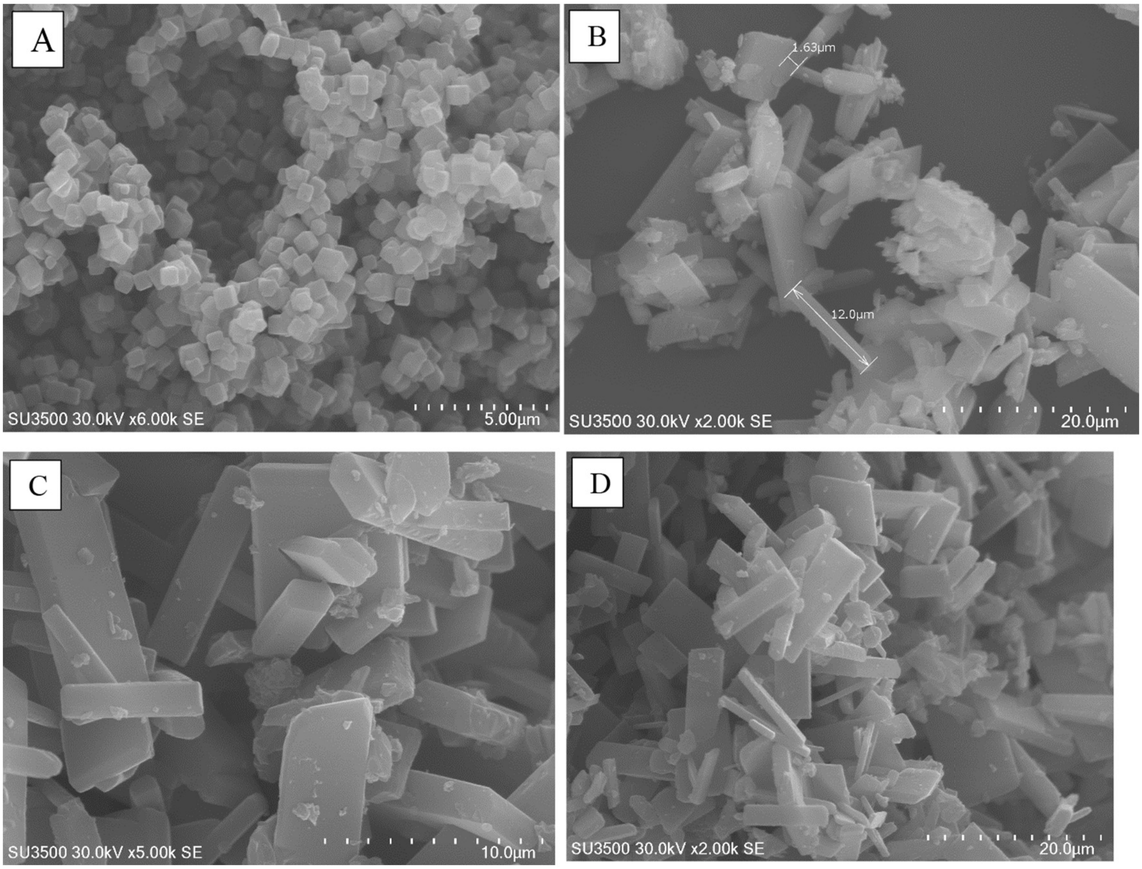

Fig. 2. FE-SEM image of ZIF-8 and ED-ZIF-8.

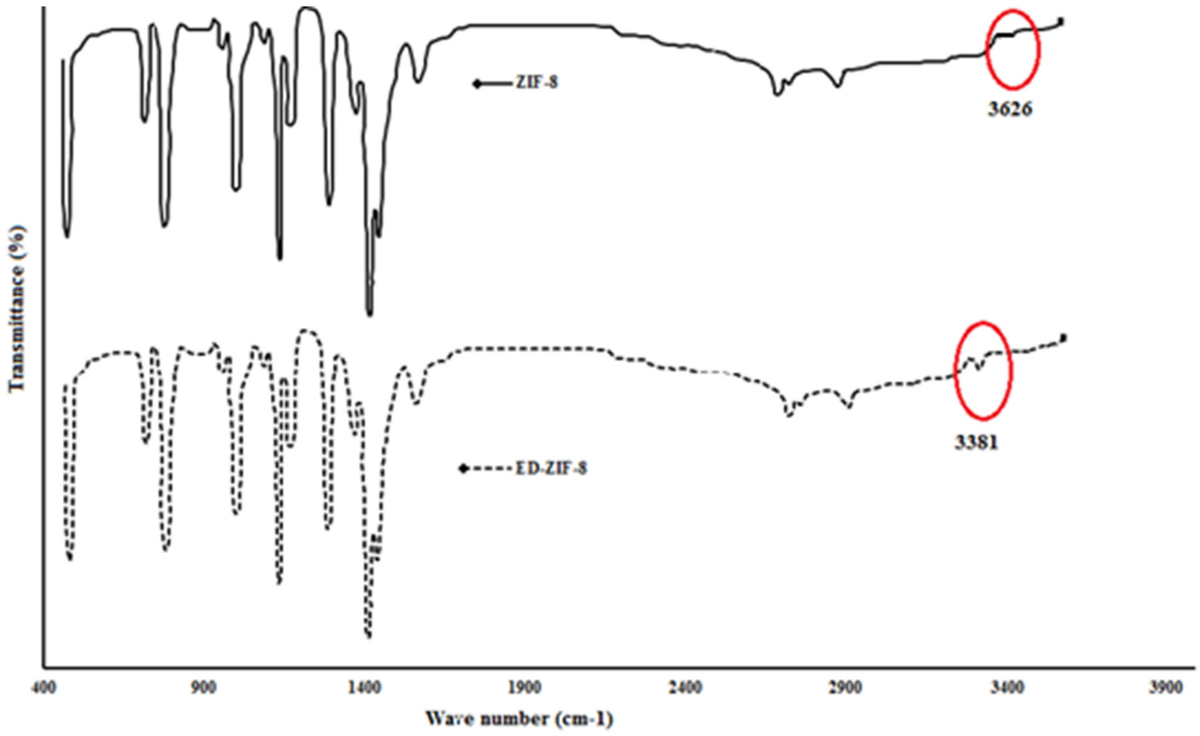

Fig. 3. FT-IR spectra of ZIF-8 and ED-ZIF-8.

were not significantly different from the software output. The final formula for the arsenic adsorption is presented in Supplementary data (Table ST-6).

3.3. Interaction effects of pH and initial concentration changes on arsenic adsorption

pH variations not only can affect the charge distribution on adsorbent and solution but also the adsorption of arsenic. The interaction results of pH and initial concentration are given in Fig. 4. To better understand the effect of pH on adsorption process, pH of the isoelectric point (pH_{IEP}) is mainly obtained. The results showed that pH_{IEP} of the adsorbent was about 9.5, it means that at values higher and lower than this point the adsorbent charge is negative and positive, respectively [2]. As shown in Fig. 4, at a constant concentration, with decreasing pH from 9, the capacity of arsenic adsorption increased. The ion charge on the arsenic (as arsenate) is negative, as the pH drops (pH < 9), the concentration of hydrogen ion increases, which ultimately leads to an increase in arsenic adsorption. At pH above 9, concentrations of ions such as OH increased which these ions can compete with arsenic

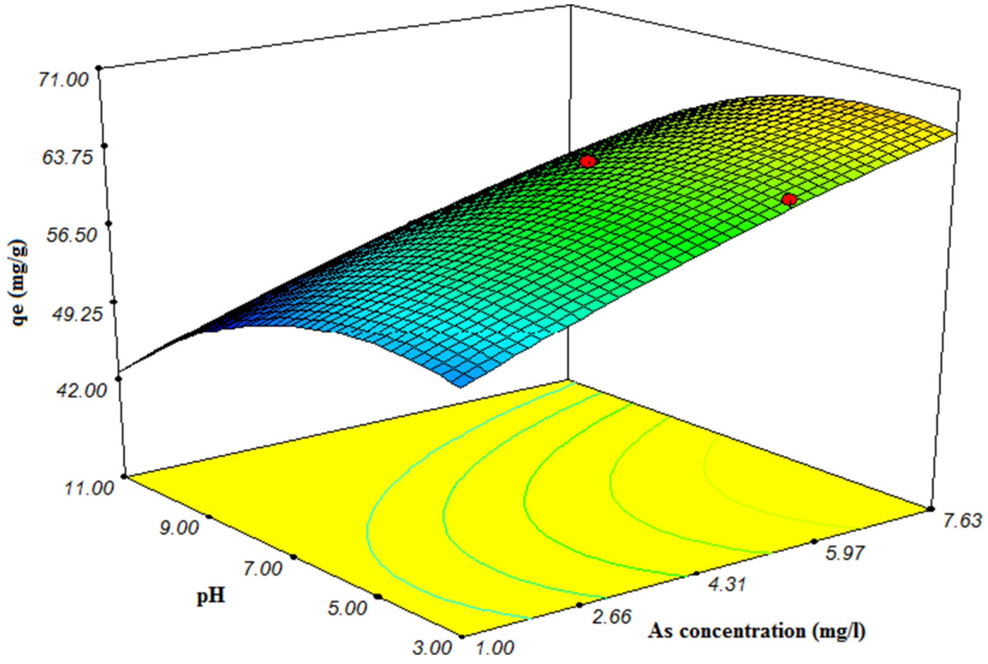

Fig. 4. Interaction effects of pH and initial concentration changes on arsenic adsorption.

ions (which has a negative charge). According to Zhang et al. study, the adsorption capacity decreases with increasing of pH due to the electrostatic force between the adsorbent and arsenate [23]. In this study the best pH for arsenic adsorption was neutral (pH ≈ 7). At constant pH (for example 7), with increasing the initial concentration, arsenic adsorption increased due to increasing the concentrations of target pollution (arsenic) results in a more intense competition for adsorption on the adsorbent which ultimately can increase the adsorbent capacity.

3.4. Interaction effects of ED-ZIF-8 dosage and initial concentration changes on arsenic adsorption

The interaction effects of ED-ZIF-8 dosage and initial arsenic concentration changes are given in Fig. 5. As shown in Fig. 5, at constant dosage, with increasing arsenic concentration, the capacity of the adsorption increased. The optimal dosage of ZIF-8 and ED-ZIF-8 for arsenic adsorption was 0.3 g/l and 0.2 g/l, respectively. As shown in Fig. 5A, at constant concentration, with increasing ED-ZIF-8 dosage from 0.1 and 0.5, the capacity of arsenic adsorption decreased. As a reason, by increasing the adsorbent dosage, the specific surface area and active sites on ED-ZIF-8 significantly increased, but in these situation, there is no arsenic for adsorption (according to Eq. (1)). Therefore, by increasing the adsorbent dosage at a constant concentration of pollutants, q_e for ED-ZIF-8 decreased. So, the changes in removal efficiency (%) and q_e (mg/g) with increasing the dosage at constant concentration were revealed. In other words, by increasing the dose of ED-ZIF-8 at constant arsenic concentration, the removal efficiency increased until the optimal dosage (according to Eq. (1) and Fig. 5B). Accordingly, the optimal concentration of arsenic for adsorption was 2.88 mg/l.

Table 1
Kinetic (A), isotherm, (B) and thermodynamic (C) constants of arsenic adsorption on the ED-ZIF-8.

A. Kinetics					
Pseudo-second-order			Pseudo-first-order		
$q_{e\ (mg/g)}$	K_2	R^2	$q_{e\ (mg/g)}$	K_1	R^2
65.45	0.0072	0.99	77.34	0.0265	0.886
B. Isotherms					
Freundlich			Langmuir		
n	K_f	R^2	$q_{m\ (mg/g)}$	b	R^2
3.55	17.7	0.845	83.7	2.32	0.988
C. Thermodynamics					
Tem. (K)	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})		ΔS° (kJ mol^{-1} k^{-1})	
293.15	−19.7	−35.12		0.047	
303.15	−17.6				
313.15	−15.6				

3.5. Kinetics, isotherms, and thermodynamics of arsenic adsorption on ED-ZIF-8

To better understanding the influencing factors on reaction rate, evaluation of reaction kinetic is necessary. The kinetics analysis are useful for predicting the adsorption rate, designing and modeling the process [32]. Table 1-A shows the results of kinetic models investigated in this study. The basis for choosing the models is coefficient of determination (R^2) value. Among the studied models, the highest coefficient of determination was related to the pseudo-second-order model. The results showed that the rate of arsenic adsorption on ZIF-8 and ED-ZIF-8 was very fast and >90% of the equilibrium adsorption capacity was achieved in first 30 min. The equilibrium time of ZIF-8 and ED-ZIF-8 for arsenic adsorption was approximately 5 and 6 h, respectively. In this study, the optimal times for arsenic adsorption by ZIF-8 and ED-ZIF-8 were 30 and 32 min, respectively. The findings of Liu et al. showed that arsenite (III) concentration in the solution reduced from 200 μg/l to <10 μg/1 after 2 h [20]. According to Jian et al., the arsenate and arsenite adsorption was very rapid. Required time to reach the equilibrium status for arsenate and arsenite adsorption was 7 and 13 h, respectively [33]. Table 1-B shows the results of isotherm models (namely Langmuir and Freundlich models) investigated. Between the studied models, the highest R^2 was related to the Langmuir model. According to this model, the maximum adsorption capacity of arsenic on ZIF-8 and ED-ZIF-8 were 65, 83.7 mg/g, respectively. By placing two amine groups (NH_2-) on ZIF-8, arsenic adsorption capacity was increased by 18.7 mg/g. According to the Langmuir isotherm model, various adsorption capacities have been obtained in various studies with ZIF-8. The maximum adsorption capacity of As (V) in Liu et al. [20], Jian et al. [33], and Li et al. [34] works were 122.6, 60.03, and 76.5 mg/l respectively. These differences can be due to some reasons such as quality of raw materials and the adsorbent properties. To calculate thermodynamic indices, the constants of ΔG° (Gibbs free energy), ΔH° (enthalpy), and ΔS° (entropy) was calculated [35]. Table 1-C shows the results of Thermodynamic models that were investigated. Gibbs free energy (ΔG°) was negative throughout the examined temperature range. It can therefore be concluded that arsenic adsorption on ED-ZIF-8 could be spontaneous. As the temperature raised, the negative Gibbs free energy decreased, which indicated that it was less favorable at high temperatures. The results indicated that enthalpy was negative. Negative values indicated the adsorption process was exothermic, which means

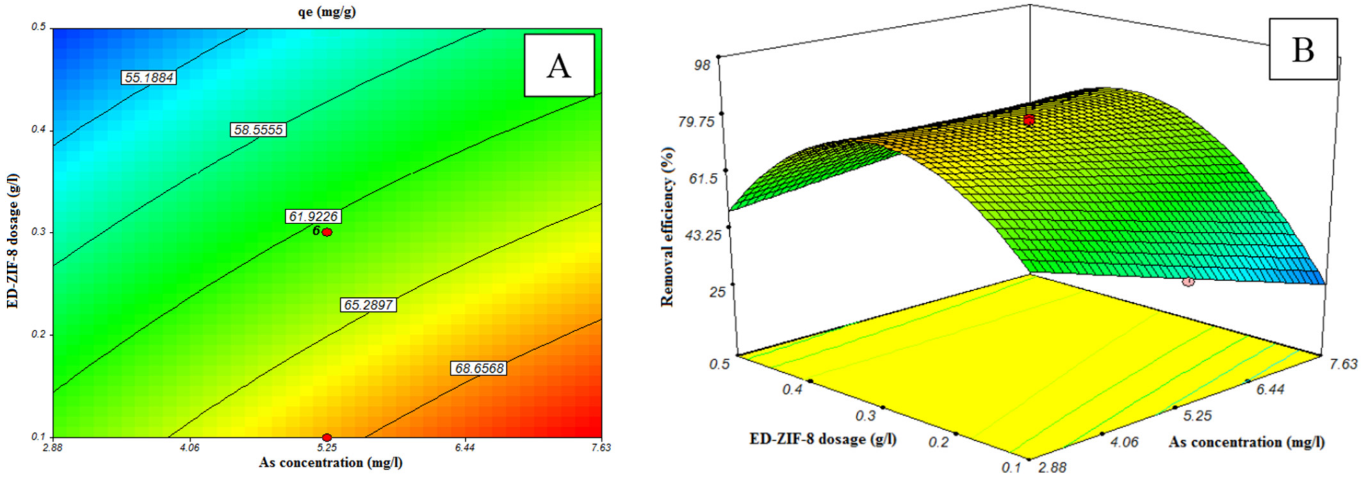

Fig. 5. Interaction effects of ED-ZIF-8 dosage and initial concentration changes on arsenic adsorption.

less adsorption at higher temperatures. The low entropy values also indicated that the entropy changes with the adsorption process were minor.

3.6. Interfere of co-existing anions in adsorption of arsenic

In natural conditions (e.g. groundwater), there are no target pollutants alone. In other words, they are found along with other elements. In this study, the presence of bicarbonate, sulfate, chloride, nitrate, fluoride, and phosphate along with arsenic was simulated with different charge capacities. The results showed that bicarbonate, sulfate and chloride had the least interference (5% decrease in adsorption) in arsenic removal. Fluoride, nitrate (35% decrease in adsorption) and phosphate (82% decrease in adsorption) have a greater effect on the arsenic removal using ED-ZIF-8. When all the anions with arsenic were in contact, totally 35% decrease in adsorption efficiency occurred. The findings of Jian et al. showed that sulfate and nitrate have no effect on the adsorbent efficiency for arsenic (IV and V) removal. Furthermore, phosphate and carbonate can interfere with arsenic adsorption [33]. The reason for this phenomenon can be described that elements which have an adsorption behavior similar to arsenic can interfere with the arsenic adsorption [36]. Previous studies have also shown that elements such as carbonate could be adsorbed on active sites such as zinc and reduce the arsenic adsorption [37,38]. Liu et al. study showed that sulfate and chloride have no effect on the arsenic adsorption, but carbonate and phosphate have a great influence. Phosphate has the greatest effect on reducing arsenic adsorption [20]. To evaluate the efficiency of ED-ZIF-8 in real samples, two villages were sampled (a spring and a well). Table 2 shows the measurement of various parameters before and after the adsorption process. To evaluate these samples, optimum conditions were used. Initially, the arsenic concentration in both samples was higher than the standard values. After exposure of the adsorbent to optimal conditions, the amount of arsenic remained below the standard value. After adsorption, the solution pH did not change at all and there was no need for the final correction of pH.

4. Conclusions

In this work, adsorbents cubic ZIF-8 and ED-ZIF-8 were synthesized. To functionalization of ZIF-8, a solution of 30% ethylenediamine was used. Central Composite Design method was used to design of experiments. After functionalization, the arsenic adsorption capacity increased from 72 to 83.5 mg per gram of adsorbent. According to the selected model, the best adsorption conditions were arsenic concentration 2.88 mg/l, pH 5, adsorbent dosage 0.2 g/l, contact time 31.67 min. The findings showed that the experimental data obtained fitted with kinetic model pseudo-order-reaction and isotherm model of Langmuir. The equilibrium time of ZIF-8 and ED-ZIF-8 for arsenic adsorption was approximately 5 and 6 h, respectively. The results showed that bicarbonate, sulfate, chloride had the least interference in the removal of arsenic. Fluoride, nitrate and phosphate have a greater effect on the removal of arsenic using ED-ZIF-8

Table 2
Chemical parameters of groundwater before and after treatment with ED-ZIF-8.

	Unit	Gharaberakh (well)		Saloja (spring)	
		Before	After	Before	After
pH	–	7.75	6.5	7.68	6.3
Chloride	mg/l	21	16	34.15	30.1
Solphate	mg/l	107	85	65	60
Arsenic	mg/l	0.034	<0.01	0.029	<0.01
Fluoride	mg/l	0.2	0.1	0.3	0.12
Nitrate	mg/l	35	26	21.81	14
Phosphate	mg/l	0.06	0.03	0.03	0.01

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.molliq.2018.01.163.

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