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Removal of arsenic from ground water by modified Sabzevar zeolite

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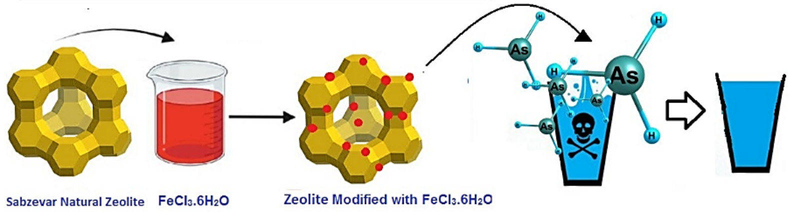
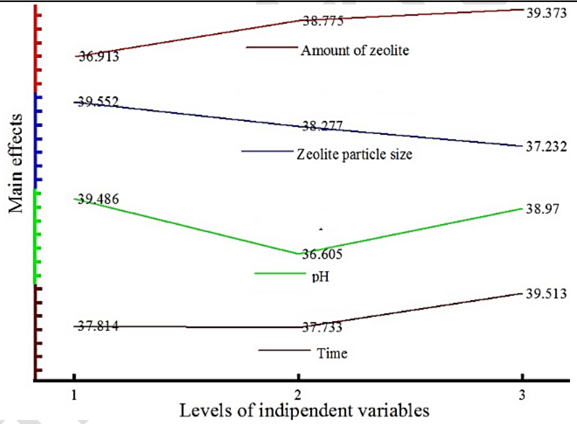
GRAPHICAL ABSTRACT

Zeolite modification method	Percentage of adsorption of arsenic
Unmodified	0.8
Modified with HDTMA	53.1
Modified with HCl	71.97
Modified with $FeCl_3.6H_2O$	97.18
Modified with NaCl + $FeCl_3.6H_2O$	96.86





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ABSTRACT

The existence of arsenic in water is one of the main problems of the world, especially in developing countries. One of the methods suggested for removal of arsenic is the use of adsorbents. In this study, natural Sabzevar zeolite was modified with HDTMA-Br, HCl, NaCl and $FeCl_3.6H_2O$ for the adsorption of arsenic. It was found that zeolite modified with $FeCl_3.6H_2O$ was best for the removal of arsenic. Contact time (h), pH, zeolite particle size (mesh) and amount of zeolite (g) as the important parameters were optimized by Taguchi method. The optimum values for these parameters were contact time of five h, pH=3, and 100-200 mesh particles. The maximum adsorption capacity of 99.5% was obtained at optimum condition. Reactivation of the modified zeolite using HCl showed that the reactivated zeolite adsorbed 98% of the arsenic, a slight decrease compared to the original material. The presented adsorption process is a green and economical process for removing arsenic from water and contaminated groundwater.

1. Introduction

Due to the increasing population of the world and the growing need for water, the exploitation of groundwater resources and saline water have been a necessity in recent decades (Pereira, 2017, ShenavaeiZare, Khoshsima and ZareNezhad, 2021a, Shenavaei Zare, Khoshsima and ZareNezhad, 2020). The quality of drinking water has a great impact on public health, so the chemical composition of surface and underground water is one of the criteria that determines the choice of water sources in different communities. The rapid growth of cities and the advancement of industrial, agricultural, academic and tourist activities is now affecting groundwater resources. Waste discharged into water resources has risen to the point that pollution of water resources has endangered the health of consumers (Massoudinejad and Ghaderpoori, 2016, Ahmadi Kamarposhti *et al.*, 2020). The existence The high amounts of this substance in water can cause skin diseases, liver and various types of cancers and can even lead to death in larger quantities (Ren *et al.*, 2014, Sanaei *et al.*, 2021). Because of the serious toxic effects of arsenic on human health and many international regulatory bodies have proposed 10 micrograms per liter (10 ppb) as the maximum allowable arsenic in drinking water (WHO, 2011). The release of arsenic into groundwater occurs through the erosion of mineral minerals and human activities, such as metal industry discharges and the use of pesticides containing arsenic. In Iran, research has shown that in some urban and rural areas of the country there has been a problem of water pollution with arsenic from ancient times. In some cities, drinking water contamination of 1,000 µg/L of arsenic has been reported and has caused serious problems for residents of the area (Hosseinpour *et al.*, 2011).

There are several ways to remove arsenic from drinking water, including coagulation, oxidation, electrochemical methods,

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bioremediation, ion exchange, membrane processes and adsorption (Nicomel *et al.*, 2016, Ungureanu *et al.*, 2015, Yousefi *et al.*, 2022). At present, the adsorption method has been considered the most appropriate method for the removal of arsenic (Ungureanu *et al.*, 2015, Vieira *et al.*, 2017, Agrafioti, Kalderis and Diamadopoulos, 2014). One of the methods suggested for removal of arsenic is the use of zeolites as adsorbents. Zeolites, in particular, have been the focus of many studies throughout the world because of their low cost. Mineral zeolite with its special spatial structure, high cation exchange capacity, maintenance of the structure at high temperature, low cost, high efficiency and abundance is considered to be a good way to remove these contaminants from the environment (Agrafioti, Kalderis and Diamadopoulos, 2014, Mokhtari-Hosseini *et al.*, 2016, ShenavaeiZare, Khoshsima and ZareNezhad, 2021b, de Gennaro *et al.*, 2020, Shenavaei-Zare and Mokhtari-Hosseini, 2024).

This study was aimed at removing arsenic from underground water, using natural Sabzevar zeolite. Another aim of this research was to optimize adsorption process of arsenic from groundwater using natural zeolite and modified zeolite whose preparation and used were guided by the Taguchi method.

2. Materials and methods

In this research, the natural zeolite clinoptilolite from the Chah-e-Talkh region of Sabzevar was used. The chemicals, $FeCl_3$, NaCl, hexa decyl tri methyl ammonium bromide (HDTMA-Br), $AgNO_3$, $FeCl_3.6H_2O$, NaOH, KOH, HCl and HNO_3 were obtained from Merck Company. $Na_2HAsO_4.7H_2O$, was purchased from Sigma Aldrich Company.

2.1. Preparation and characterization of Sabzevar zeolite

After the natural zeolite was washed with distilled water and dried, it was sieved with American Standard Sieves and stored in the desiccator. The composition of Sabzevar zeolite obtained by XRF in my previous study is shown in Table 1 (Mokhtari-Hosseini, Bikhabar and Shenavaei Zare, 2023). According to this table, the mass ratio of SiO_2/Al_2O_3 is equal to 6.55 and 15.62% of loss on ignition (L.O.I.), which is still the same result that can be seen in the research of Guan et al (Guan *et al.*, 2010).

Table 1. The elemental composition of Sabzevar zeolite based on XRF measurements.

Elements	Perception, %
SiO_2	62.68
Al_2O_3	9.57
Na_2O	2.43
MgO	0.77
CaO	5.51
K_2O	1.76
TiO_2	0.17
MnO	0.09
P_2O_5	0.03
Fe_2O_3	0.041
SO_3	0.00
LOI	15.62

Transmission electron microscopy (CM120, Philips) and X-ray diffraction (XRD) (X' Pert PW 3040/60, Philips) were used to identify the structure, and the percentage of minerals of the Sabzevar natural zeolite. The functional groups in the natural zeolite Sabzevar were recorded using an FTIR spectrometer (Shimadzu, Japan).

2.3. Modification of zeolite

The surface of zeolite samples was modified as described in sections 2.3.1 – 2.3.4:

2.3.1. Zeolite modification by acid

In this method, natural zeolite (10 g) was added to 250 ml of 1M HCl and stirred at 150 rpm for 6 h at 60 °C. The modified zeolite was separated and washed with warm and cold distilled to obtain neutral pH. The zeolite was then dried at 80 °C for 24 h (Mokhtari-Hosseini *et al.*, 2016).

2.3.2. Zeolite modification by $FeCl_3.6H_2O$

A 120 g of natural zeolite was stirred in 360 ml of 20 mM $FeCl_3.6H_2O$ for 24 h at 150 rpm and 30 °C. The pH of the solution was adjusted to 9 with NaOH solution (2 M). After settling, the modified zeolite was rinsed with deionized water until all Cl ion was removed. Finally, the modified zeolite was dried at room temperature (Prasetyo and Soegijono, 2019).

2.3.3. Zeolite modification by HDTMA

Zeolite (5 g) and 2.5 g of HDTMA were stirred in 180 mL of deionized water for 24 h at 150 rpm and 30 °C. Then, the zeolite was separated, washed and dried at 100 °C for 24 h (Guo and Wang, 2016, Tayebee *et al.*, 2015).

2.3.4. Zeolite modification NaCl and $FeCl_3.6H_2O$

30 g Zeolite as added to NaCl solution (200 mL of 0.1N) and was boiled at reflux conditions for four h. The suspension was filtered and the previous step was repeated again. The zeolite was washed with distilled water five or six times until chloride ion was not detected in wash water using the $AgNO_3$ test. Then the zeolites were dried at 80 °C for 24 h to prepare the Na zeolite. In the next step, 100 mL of 0.1N $FeCl_3.6H_2O$ solution was added to 10 g of Na zeolite, and this process involves heating under reflux for about four h. The suspension was filtrated, and the previous step was repeated. The solid phase was washed with distilled water and finally dried overnight at 85 °C (Margeta *et al.*, 2013). The modified zeolites were stored in a desiccator until use.

2.4. Selection of the “best” modified zeolite for arsenic adsorption

Arsenic solutions were prepared using $Na_2HAsO_4.7H_2O$ salt with concentration of 1 ppm. For the selection of the “best” modified zeolite, 3 g of each modified zeolite was added to 30 ml of prepared solution of arsenic and was placed in the incubator for 24 at 150 rpm and 30 °C. After 24 h, the zeolite was separated from the mixture, and the concentration of arsenic in the solution was measured by atomic adsorption (AA-7000, Shimadzu, Japan) with heating in a graphite furnace (Jang *et al.*, 2011).

2.5. Optimization of arsenic adsorption process by Taguchi method

Removal efficiency and adsorption capacity of the arsenic adsorption process depend on several variables. So several experiments should be performed (Mokhtari-Hosseini, Bikhabar and Shenavaei Zare, 2023). According to the reports of other researchers and preliminary screening, four experimental variables (zeolite size, pH, contact time and the amount of zeolite) were selected for further investigation (Massoudinejad and Ghaderpoori, 2016). Using an experimental design, the number of experiments is reduced. In order to evaluate the four independent variables at three levels (Table 2), Taguchi method with L_9 array was used. The analysis of variance and predicted optimal conditions were conducted using Qualitec-4 software.

Table 2. Independent variables and their levels in L_9 array of Taguch.

Variable	Level 1	Level 2	Level 3
Time, h	1	7	15
Zeolite particle size, mesh	100-200	20-30	12-16
pH	3	6.5-7.5	10
Amount of zeolite, g	0.5	2.5	5

Arsenic solutions were prepared using $Na_2HAsO_4.7H_2O$ salt with concentration of 1 mg/L. To evaluate the effect of zeolite size, pH, contact time and the amount of zeolite on the efficiency of arsenic removal, zeolite modified by FeCl3.6H2O was used in 50 mL arsenic solutions, and experiments were performed according to Table 3. At the end of the experiments, aqueous solution was separated from zeolite, and the concentration of arsenic in the solution was measured by atomic adsorption (AA-7000, Shimadzu, Japan) with heating in a graphite furnace (Agrafioti, Kalderis and Diamadopoulos, 2014).

Table 3. L_9 array of Taguchi for optimization of arsenic adsorption process by $FeCl_3.6H_2O$ modified zeolite.

Factor test	Time, h	pH	particle size, mesh	Amount of zeolite, g
1	1	3	100-200	0.5
2	1	6.5-7.5	20-30	2.5
3	1	10	12-16	5
4	7	3	20-30	5
5	7	6.5-7.5	12-16	0.5
6	7	10	100-200	2.5
7	15	3	12-16	2.5
8	15	6.5-7.5	100-200	5
9	15	10	20-30	0.5

2.6. Assessment of the reduction of arsenic in one of the wells in Golestan province

To reduce the amount of arsenic in the water under optimum conditions, 5 g of zeolite 100-200 mesh, was added to 50 ml of well water adjusted to pH 2-3. After 15 h at 30 °C the zeolite was removed and the arsenic content of the water was measured by atomic adsorption (AA-7000, Shimadzu, Japan).

2.7. Reactivation of zeolite

To be economical, it must be possible to use the modified zeolite used for a long period of time. Hydrochloric acid (0.1N) was used to desorb arsenic from the zeolite. The spent absorbent was poured into HCl solution, and shacked (150 rpm, 24 h and ambient temperature). The desorbed zeolite was filtered, washed and dried. Then, in order to evaluate the efficacy of reactivated zeolite, its function in removing arsenic was studied at optimum condition (Behera, Mishra and Radhika, 2022, Carneiro *et al.*, 2024, Yadav *et al.*, 2022).

3. Results and discussion

3.1. Characterization of Sabzevar zeolite

According to the XRD results in Fig. 1, the Sabzevar zeolite has 76% clinoptilolite and 24% calcite. The XRD spectrum showed that the peaks at 2θ angles of 9.8314◦, 11.1262◦, 12.9522◦,16.876727◦, 17.2877◦, 19.0590◦, 22.3770◦, 25.0103◦, 25.9766◦, 26.5886◦, 27.9838◦, 30.2087◦, 31.9329◦, 32.7889◦, 35.2494◦, 44.8871◦, 46.2845◦, 50.0600◦, and 51.5918◦ correspond to the crystal planes (hkl), (020), (200), (-201), (-311), (111), (-131), (020), (200), (-201), (-311), (111), (-131), (131), (-312), (-222), (-402), (-422), (151), (530), (-261), (222), (-643), (-4153), (-314), (-772), and (-10003) of clinoptilolite, according to the ICDD PDF standard card of clinoptilolite (card #01-080-0464). Additionally, the diffraction peaks at 2θ angles of 29.3645◦, 37.0046◦, 39.4022◦, 43.1427◦ , 47.5091◦, and 48.5056◦ are attributed to the crystal planes (104), (110), (113), (202), (018), (116), and (214) of calcite (card #01-072-1937).

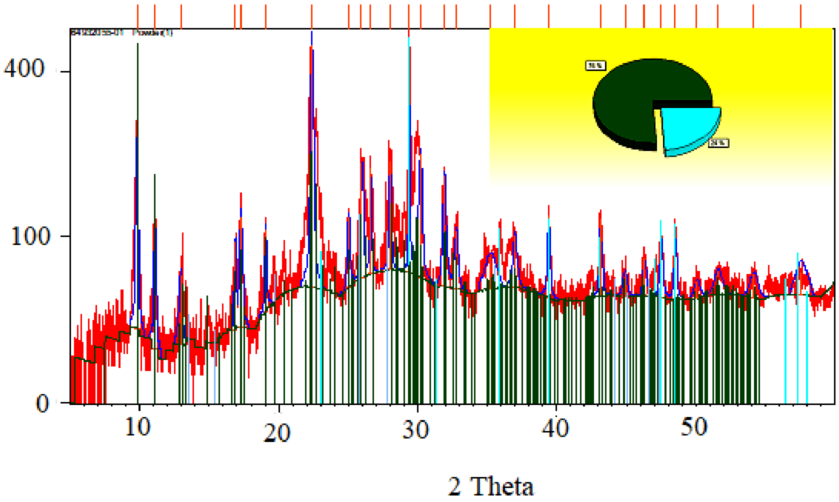
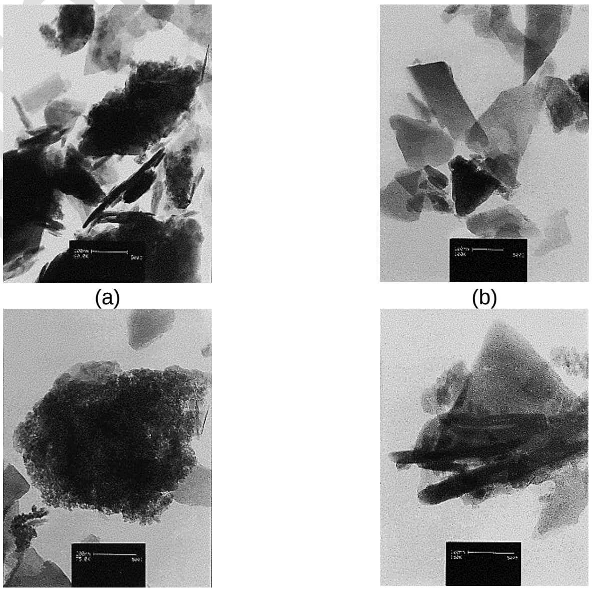
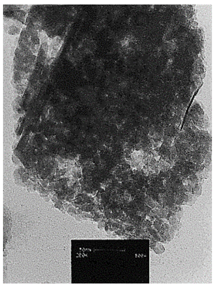


Fig. 1. XRD results of natural Sabzevar zeolite powder.

These results are consistent with other studies (Guan *et al.*, 2010). According to the TEM results (Fig. 2), zeolite nanoparticles have non-spherical morphology and uncertain geometrical shape. Also, the surface of zeolite particle has spongy or porous state.


(a)
(b)
(c)
(d)



(e)

Fig. 2. TEM results of Sabzevar zeolite powde with different magnification (a) 50 K, (b) 75 K, (c) 100 K, (d) 150 K, and (e) 200 K.

The peaks of the broad 3400-3500, 1600-1700 and 1400-1450 cm^{-1} in the FTIR spectrum of zeolite (Fig. 3) correspond to the vibration of H_2O molecules associated with K and Na in the channels and cages in the zeolite structure. The 1062, 671, 561 and 466 cm^{-1} bands correspond to Si-O stretching, Al-O-Si, Al-O and Si-O-Si bending vibrations in zeolite, respectively. The peaks of the broad 2354 cm^{-1} correspond to the CO_2 molecules in the atmosphere.

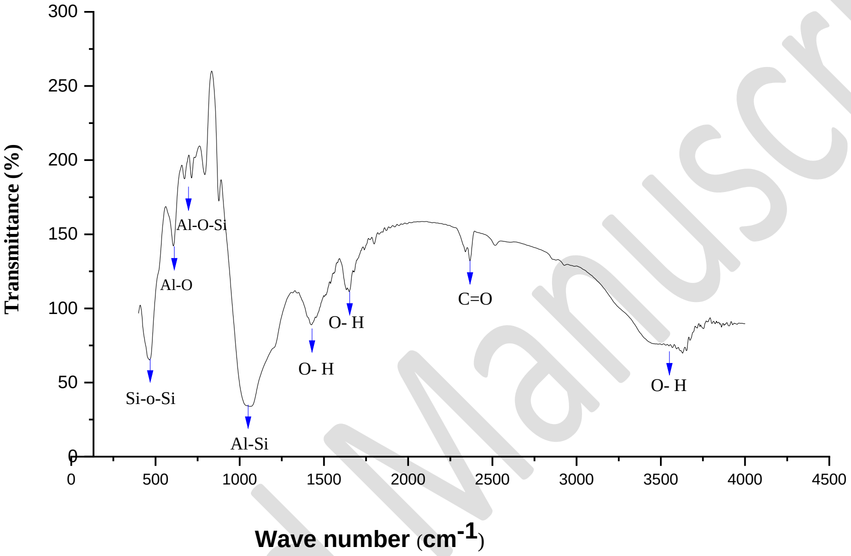


Fig. 3. FTIR spectra of natural Sabzevar zeolite.

3.2. Selection of the "best" modified zeolite for arsenic adsorption

Table 4 shows the results of arsenic-adsorption efficiency with different methods of zeolite modification and compares them with the unmodified zeolite. According to the results (Table 4), the activation of zeolite increased the adsorption of arsenic, with the best modification being with $FeCl_3.6H_2O$.

3.3. Taguchi method

The effect of contact time, pH, zeolite particle size and amount of zeolite were studied and analyzed by the Taguchi statistical method. For this purpose, each factor was studied at three levels using an orthogonal L_9 array, the levels of each factor, for the nine tests based on the Taguchi statistical method are given in Table 5. The results of the experiments were analyzed according to L_9 array using Qualitec-4 software. Table 6 shows the main effects of the variables. The last column of table 6 shows the effect of surface change on the rate of change in adsorption percentage. AS shown in this table, the greatest change in the percentage of adsorption is observed in the change in pH.

Table 4. Adsorption of arsenic on four types of modified Sabzevar zeolite.

Zeolite modification method	Percentage of adsorption of arsenic
Unmodified	0.8
Modified with HDTMA	53.1
Modified with HCl	71.97
Modified with $FeCl_3.6H_2O$	97.18
Modified with NaCl + $FeCl_3.6H_2O$	96.86

Table 5. Taguchi design matrix (L_9 array) and results for arsenic adsorption process by $FeCl_3.6H_2O$ – modified zeolite.

Factor test	Time, h	pH	Zeolite particle size, mesh	Amount of zeolite (g)	Removal efficiency, %	
					Repeat 1	Repeat 2
1	1	3	100-200	0.5	87.43	84.91
2	1	6.5-7.5	20-30	2.5	70.94	62.22
3	1	10	12-16	5	83.86	81.21
4	7	3	20-30	5	97.20	98.46
5	7	6.5-7.5	12-16	0.5	95	99.40
6	7	10	100-200	2.5	99.58	99.72
7	15	3	12-16	2.5	99.33	99.44
8	15	6.5-7.5	100-200	5	99.80	99.79
9	15	10	20-30	0.5	81.84	89.10

Table 6. Main effect of variables.

Variable	Surface			
	1	2	3	L_2-L_1
Time, h	37.814	37.733	39.513	-0.082
pH	39.486	36.605	38.97	-2.882
Zeolite particle size, mesh	39.552	38.277	37.232	-1.275
Amount of zeolite, g	36.913	38.775	39.373	1.862

3.3.1. Effect of particle size

As shown in Fig. 4a, with decreasing the size of zeolite particles, increases the adsorption and smaller particles size are more effective. The reason for this can be related to the increase in contact surface. This was also noted by other researchers. (Menhage-Bena *et al.*, 2004, Salem Attia, Hu and Yin, 2014).

3.3.2. Effect of time

The effect of contact time was studied at 1, 7 and 15 h. As shown in Fig. 4b, showed increasing percentage of adsorption with increases time (Fig. 4b). This experiment also showed that there was no significant difference at the shorter times of 1 h and 7 h. But with increasing the contact time to 15 h, the adsorption increases sharply. Ulmanu et al. (2003) showed that increasing time-to-contact increases the adsorption of heavy metals by natural zeolites (Ulmanu *et al.*, 2006, Ulmanu *et al.*, 2003). These results are consistent with Chutia et al studies (Chutia *et al.*, 2009). Better adsorption can be within 1 h compared to 7 h due to the abundance of active sites on the surface of the adsorbent for rapid binding of metal ions. However, the second step is the slow diffusion of metal ions within the adsorbent. Communication time is considered to be 15 h with an adsorption efficiency of 39.6 % in the equilibrium time. Sanai et al. (2021) also addressed this issue (Sanaei *et al.*, 2021).

3.3.3. Effect of zeolite amount

Fig. 4c shows that increasing the amount of zeolite increases the amount of adsorption because the higher amount of zeolite increases the availability of ion exchange sites and for this reason the adsorption rate increases. This was also noted by Sanaei *et al.* (Sanaei *et al.*, 2021).

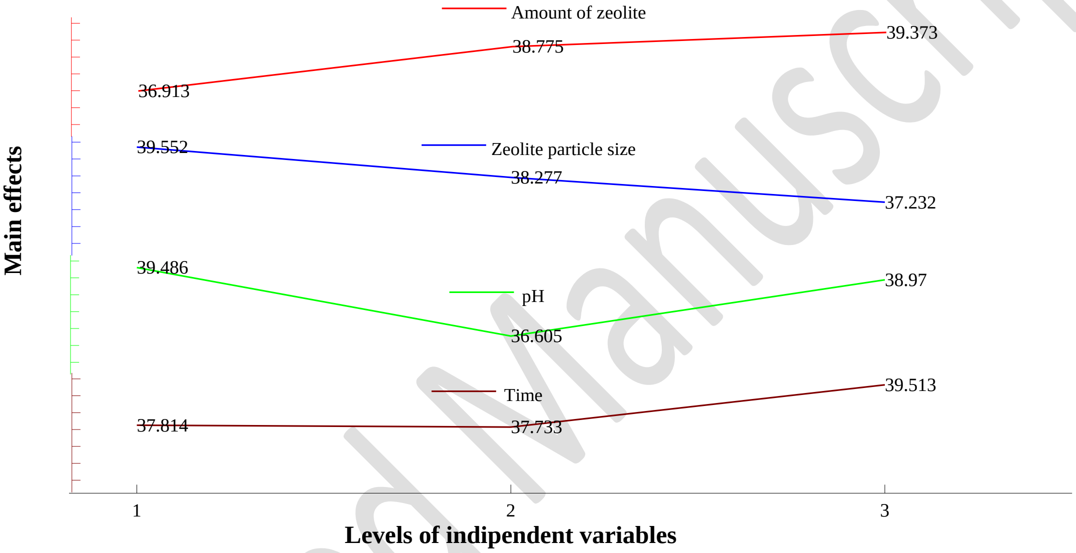


Fig. 4. The main effects of particle size; time; amount of zeolite and pH on arsenic removal by $FeCl_3.6H_2O$ modified zeolite from aqueous solution.

3.3.4. Effect of pH

As seen in Fig. 4d in acidic and alkaline environments, better adsorption is observed depending on the neutral pH. Menhage-Bena et al. (2004) showed for arsenic solution with pH less than 7, better adsorption accrued in pH= 3 whereas solution with pH higher than 7 did not show any significant changes in adsorption (Menhage-Bena *et al.*, 2004). In the pH 3.0 the predominant arsenic species formed was $H_2AsO_4^-$ thus resulted in the dramatic increase in arsenic removal percentage owing to the improved electrostatic attraction. At pH of 6.5 – 7.5 and 10.0, arsenic existed predominantly as $HAsO_4^{-2}$ and AsO_4^{-3} and its removal were decreased (Zhou *et al.*, 2021).

3.3.5. Statistical analysis

Table 7 shows the values obtained from the analysis of the results by Qualitek-4 software. As shown in Table 8, the most effective variable in the range of variables is pH, which has a 37% share in the responses and the least effect is time.

Table 7. Analysis of the orthogonal array L_9.

Variable	DOF	Sum of Squares	Variance	Pure Sum	Percentage
Time, h	2	6.06	3.03	6.06	15.867
pH	2	14.16	7.08	14.16	37.077
particle size	2	8.094	4.047	8.094	21.194
Amount of zeolite	2	9.876	4.938	9.876	25.86

One of the advantages of the Taguchi method is the optimal process conditions. Optimum conditions for the process at the studied levels can see in Table 8. The predicted response from the software in this situation is 100% adsorption. The confirmation test was obtained in these conditions; adsorption percentage was 99.52 which is consistent with the predicted value of the software.

3.4. Assessment of the reduction of arsenic in one of the wells in Golestan province

The amount of arsenic contamination in the Chai well in Golestan Province was determined by atomic adsorption to be 20 ppb. By using the batch method, in the optimum conditions (Table 8) obtained in the previous step and with both modified (with $FeCl_3$) and unmodified zeolite, the amount of arsenic adsorption was investigated. According to the results, the modified zeolite, adsorbed nearly 80 percent of arsenic in the well-water sample.

3.5. Reactivation of modified zeolite

It was found that the zeolite used to remove arsenic from an aqueous solution, could be reactivated using 0.1N HCl and reactivated zeolite showed 98% of the original adsorption, which was 99.5%.

Table 8. Optimal and predicted conditions by the software Qualitek-4.

Variable	Surface description	Surface
Time	15	3
pH	3	1
Zeolite particle size	100-200	1
Amount of zeolite	5	3

4. Conclusions

This study showed that efficiency in removing arsenic from water with modified zeolite can be increased by crushing the particles and adjusting pH to outside of the neutral range. Under optimal conditions, about 99.5% of arsenic was removed. Reactivation of the modified zeolite using HCl showed that the reactivated zeolite adsorbed 98% of the arsenic, a slight decrease compared to the original material. Considering the abundance of zeolites in natural mines, their low cost and ease of modification and the possibility of reactivation, modified zeolite from the Sabzevar may be suitable for removal of arsenic from aqueous solution as a low cost method for solving biological problems.

Author Contributions

Fatemeh Hoveidamanesh: Investigation, methodology.
Zahra Beagom Mokhtari-Hosseini: Conceptualization, supervision, project administration, writing - review & editing.
Toktam Shenavaei Zare: Conceptualization, writing - review & editing.
Hashem Akhlaghi: Writing - review & editing.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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Data Availability Statement

The data presented in this study is available on request from the corresponding author.

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