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Performance evaluation of montmorillonite and modified montmorillonite by polyethyleneimine in removing arsenic from water resources

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ABSTRACT

Arsenic, known as a pollutant, mostly found in ground water resources, has become a major problem in the most of underdeveloped and developing countries around the world. More than 140 million of people around the world, especially those who reside in South America and south-east of Asia, are gravely at risk of being exposed to arsenic pollution. In this study, the performance of montmorillonite (MMT) and modified montmorillonite by polyethyleneimine (MMT@PEI) in removing arsenic from water resources was evaluated and compared with each other. Response surface methodology used as experimental procedures and interaction of four variables, namely, detention time, pH, initial arsenic concentration, and the adsorbent concentration on the arsenic ion removal were conducted. It is noteworthy that all chemical experiments conducted in this study are in accordance with "Standard Methods for Water and Wastewater Experiments." Also, XRD analysis was performed to determine the characteristics of the adsorbent. Based on the results, maximum percentage of fluoride removal efficiencies by applying montmorillonite ($R^2 = 0.95$) and modified montmorillonite ($R^2 = 0.97$) were 45 and 96%, respectively.

Keywords: Potable water; Arsenic concentration; Montmorillonite; Response surface methodology

1. Introduction

Arsenic, which is a pollutant mostly found in groundwater resources, has become a major problem on a worldwide scale – under developed and developing countries, in particular [1]. Naturally, this element, by 74.9 Mw and with +5, +3, 0, and −3 values, can be found in various places, including rocks, soils, water, air, and biota (i.e. particularly among marine species) [1–3]. On one hand, arsenic is being used to produce many products; arsenic-based pesticides, medicine,

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wood preservation, electrolytic process, mining, etc. are some of the products and areas in which arsenic can be used. On the other hand, the released wastewater, which is produced by factories and also agricultural activities, into the environment can be the main sources of hazardous concentration of arsenic found in the water resources [4]. Arsenic exists in nature in two forms of organic and mineral, within which the latter one is found to be more toxic. The oxidation of this element, once it enters the water resources, depends on the pH of water and also redox potential of arsenite (+3) as well as arsenate (+5). The former one has higher toxicity than the latter form of arsenic; in fact, it is the presence of arsenite (+3) which raises concerns [1,5]. It should be mentioned that arsenite (Aso_3^{3-}) forms in anoxic condition, ground water resources in particular, while the presence of Arsenate (Aso_4^{3-}) can only be expected in surface water [6]. Arsenic, in terms of carcinogenicity, is categorized within the first group of carcinogenic compounds (i.e. the ones which mainly cause cancer in humans). Drinking water polluted with arsenic for long-term periods could cause arsenicosis, keratosis, and also lung, liver, kidney, and bladder cancers [1,3,6,7]. Based on the reports from previously conducted studies over the world, more than 140 million people around the world, especially those who reside in South America and south-east of Asia, are gravely at risk of being exposed to arsenic pollution [1,8,9]. Considering the above-mentioned effects of arsenic on people and the number of individuals whose health is at risk, developing appropriate technologies in order to efficiently remove arsenic from water resources is of high importance [6]. In this regard, various methods have been suggested and applied to remove arsenic from water resources and wastewaters, including adsorption, ion exchange, membranes, coagulation, electrocoagulation, wheat straw, and precipitation [10–17]. Among these methods, adsorption can be considered an appropriate way to be applied; since an adsorbent has been proven to have high removal efficiency of arsenic, the ability to be reused, no sludge resulted from the operation, and also easy usage by the operators [15]. It is noteworthy that high capital and operational costs, resulted sludge and toxic effluent, and also problematic issues of operation are the main disadvantages of other available methods [2,8]. However, it should be taken into account that most of the current adsorbents are of mineral-based types and very expensive; in fact, this problem lowers the applicability of adsorption method. Therefore, we tried to evaluate the performance of another potential adsorbent, known as montmorillonite (i.e. a member of smectic group rooted from clay minerals), in this study. The lower cost of this adsorbent, compared with the traditional ones, its chemical stability, high surface area, and rich interaction chemistry [18] are the reasons why we tried to evaluate its potentials regarding arsenic removal from water resources.

The present study was aimed to evaluate the performance of montmorillonite (MMT) and modified montmorillonite by polyethyleneimine (MMT@PEI) in removing arsenic from water resources. In addition, we tried to enhance the performance of these compounds by assessing the key factors of the process and model its trend by applying response surface methodology (RSM). RSM is able to indicate the effects of independent factors on the process and also each other. In addition, mathematical models to be used to predict different probable scenarios can be achieved by using RSM; it is due to these abilities of this tool that many previously conducted studies on adsorption processes have used it. Four independent variables were selected in this study, including initial concentration of arsenic, detention time, adsorbent' dosage, and pH of the solution, to be assessed at five levels (i.e. $+\alpha$, +1, 0, −1, and $-\alpha$). Design Expert Software V.7.0.0, was applied to find the proper variables.

2. Material and methods

2.1. Preparation of montmorillonite

Polyethyleneimine branched (mw ~ 25,000) and montmorillonite (K_{10}), purchased from Sigma–Aldrich (India), were used to synthesize the adsorbent. In order to do so, 0.5 g of polyethyleneimine branched was added to 100 ml of deionized water; once the compound was completely dissolved in the water, 50 g of sodium-based montmorillonite was added to the prepared solution. Then, in order to have a homogeneous solution, the container was put in a shaker for an hour while its temperature was kept still at 50˚C within this period. After that, 2 ml of HCl (36 wt%) was added to the container and the solution was being mixed for 2 h. At the end of this stage, the prepared solution was centrifuged in order to extract its residual solid matters. The extracted solids were being washed with deionized water for several times till the formed supernatant over the matters did not show any adsorption rate in 200 nm wavelength of spectrophotometer. The end product was a white powder which was put in vacuo at 40˚C to be completely dried.

In order to assess the structure of MMT@PEI, in terms of its crystal, physical, and morphological forms, SEM (36 mA) and XRD (40 KV, $5 < 2\theta < 10$) tests were applied.

2.2. Preparation of required solutions

By adding sodium arsenate ($Na_2HAsO_4{\cdot}7H_2O$) into the double-distilled water, the stock solutions of arsenic were prepared. In order for water molecules to bond with sodium arsenate compound, the compound was heated for 20 min at 105℃, and then put in desiccators to reduce its temperature.

2.3. Experiments

In order to assess the adsorption of arsenic by MMT and MMT@PEI adsorbents, a batch reactor was applied. The concentration of arsenic was measured using ICP-MS. The removal efficiency of the adsorption was calculated based on Eq. (1):

$$\mathrm{RE}\ (\%) = \frac{(C_0 - C_e) \times 100}{C_0} \tag{1}$$

RE = As removal efficiency, C_0 = As concentration in feed solution, C_e = As concentration in treated solutions.

The experiments were designed using central composite design, by which four variables at five levels were defined to be experimented on. The independent variables and their levels are shown in Table 1.

3. Results

The results of CCD test at different levels and also the removal efficiency recorded for every test is shown in Table 2. Y_1 and Y_2 represent the independent variables for the tests conducted on MMT and MMT@PEI, respectively.

Statistical test of ANOVA using Design Expert Software was conducted on the results (i.e. arsenic removal efficiency by MMT and MMT@PEI). The significance degree was set at <0.05 as the p-value of the tests. In order to reach the best possible second-order equation, the insignificant factors were ignored and then the remaining factors were entered to R software program. Eq. (2) is the result calculated by this software.

$$\begin{aligned} Y_2 = {} & \mathrm{FO}\,(X_1,\ X_2,\ X_3,\ X_4) + \mathrm{TWI}\ (X_1,\ X_2) \\ & + \mathrm{TWI}\ (X_1,\ X_4) + \mathrm{TWI}\ (X_2,\ X_4) + \mathrm{TWI}\ (\mathrm{X}_3,\ \mathrm{X}_4) \\ & + \mathrm{PQ}\ (\mathrm{X}_1,\ X_2,\ X_4) \end{aligned} \tag{2}$$

The credibility of a predictive model can be determined through statistical tests, including p-value, determination coefficients, and lack of fit test, and also sampling. Based on the result of ANOVA test, the model was statistically significant (p-value < 0.05).

Table 3 shows the correlation coefficient, (R^2 and adjusted R^2), in ANOVA analysis for application of MMT and MMT@PEI in Arsenic removal from water sources. The R^2 for adsorption of Arsenic on MMT and MMT@PEI were 0.95 and 0.96, which represents the high correlation between the independent variables and answers for 2 adsorbents (15).

The degrees of significance, calculated by ANOVA test, for arsenic removal by MMT and MMT@PEI are shown respectively in Tables 4 and 5.

3.1. XRD analysis

Morphological characteristic of montmorillonite was assessed using XRD. The former one shows the morphology of montmorillonite as well as the distances between its layers, and the latter one provides specific information about the surface of adsorbent and its characteristics. Fig. 1, which is taken by X-ray, shows the preparation steps of MMT and MMT@PEI. As it can be seen, the graph shows the peaks of 7.3 and 6.5 at 2θ for MMT and MMT@PEI, respectively. From these results, it is clear that an increase in the distance between the layers had occurred, which in fact indicates the bonding effects of replacement agents (i.e. polyethyleneimine) on the distances between the layers (9).

Table 1
Independent variables and levels of the CCD design

Variables	Units	Symbol	$-\alpha$	−1	0	+1	$+\alpha$
pH	–	X_1	2	4	6	8	10
Time	min	X_2	0	20	40	60	80
Dosage of adsorbent	(g/l)	X_3	2	4	6	8	10
Concentration of arsenic	mg/l	X_4	0.5	1	1.5	2	2.5

Table 2
CCD design with coded factor levels for arsenic removal

Run	pH	Time	Dosage of adsorbent	Arsenic	Removal Y_1	Y_2
1	−1	−1	−1	+1	5.35	20.35
2	0	$+\alpha$	0	0	47.12	93.12
3	+1	−1	−1	+1	5.17	30.17
4	0	0	0	0	43.72	83.72
5	0	0	0	0	44.13	84.13
6	0	$-\alpha$	0	0	12.11	58.37
7	+1	−1	+1	+1	15.97	58.37
8	+1	+1	−1	−1	37.65	92.65
9	−1	+1	+1	+1	37.38	85.88
10	−1	+1	−1	+1	23.28	56.28
11	−1	−1	−1	−1	15.42	46.42
12	0	0	$+\alpha$	0	47.92	97.92
13	−1	+1	+1	−1	42.76	83.76
14	+1	−1	−1	−1	25.35	76.35
15	+1	−1	+1	−1	40.74	86.74
16	−1	+1	−1	−1	31.64	75.64
17	0	0	0	0	44.36	84.36
18	0	0	0	$-\alpha$	50.31	94.31
19	+1	+1	+1	−1	49.67	94.67
20	0	0	0	0	44.08	84.08
21	+1	+1	−1	+1	23.28	64.28
22	$+\alpha$	0	0	0	6.26	50.26
23	+1	+1	+1	+1	38.21	86.71
24	0	0	0	0	43.89	83.89
25	0	0	$-\alpha$	0	22.54	57.54
26	−1	−1	+1	−1	28.18	65.18
27	0	0	0	$+\alpha$	24.76	49.76
28	−1	−1	+1	+1	14.35	41.85
29	$-\alpha$	0	0	0	7.37	30.37
30	0	0	0	0	43.53	83.53

Table 3
Regression analysis for arsenic removal by MMT and MMT@PEI, quadratic response surface model

Source	Estimate	Std. error	T-value	Pr (>|t|)	p-value Prob. > F
Model	43.95	1.52	28.77	<2.2e−16	0
X_1	1.95	0.76	2.55	0.0195	0.01
X_2	7.99	0.76	10.43	2.653	0
X_3	5.73	0.78	7.32	6.082	0
X_4	−7.12	0.76	−9.29	1.686	0
X_1X_4	−2.78	0.94	−2.96	0.0079	0.001
X_2X_4	2.54	0.94	2.70	0.0139	0.01
X_1^2	−9.18	0.71	−12.86	7.974	0
X_2^2	−3.49	0.71	−4.88	0.0001	0
X_3^2	−2.08	0.71	−2.91	0.0088	0.001
X_4^2	−1.50	0.71	−2.11	0.0481	0.01

Table 4
ANOVA results for arsenic removal by MMT as the response (Y_1)

Source	Estimate	Std. error	T-value	Pr (>|t|)	p-value Prob. > F
Model	82.19	1.44	56.76	<2.2e−16	0
X_1	6.43	0.85	7.50	5.983e−07	0
X_2	11.83	0.85	13.81	5.096e−11	0
X_3	9.24	0.85	10.78	2.750e−09	0
X_4	−11.10	0.85	−12.96	1.434e−10	0
X_1X_2	−2.56	1.04	−2.44	0.0248938	0.01
X_1X_4	−2.76	1.04	−2.63	0.0168024	0.01
X_2X_4	4.39	1.04	4.19	0.0005474	0
X_3X_4	3.90	1.04	3.71	0.0015692	0.001
X_1^2	−10.67	0.79	−13.45	7.860e−11	0
X_2^2	−1.81	0.79	−2.28	0.0347254	0.01
X_4^2	−2.73	0.79	−3.45	0.0028345	0.001

Table 5
ANOVA results for arsenic removal by MMT@PEI as the response (Y_2)

Type of coefficient of regression	MMT	MMT@PEI
R^2	0.9591	0.9769
Adj. R^2	0.9376	0.9627
p-value	4.7e−11	2.09e−12
F-statistic	44.59	69.11

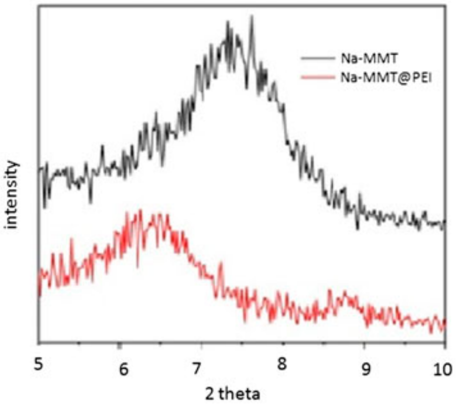

Fig. 1. X-ray diffraction patterns of MMT and MMT@PEI.

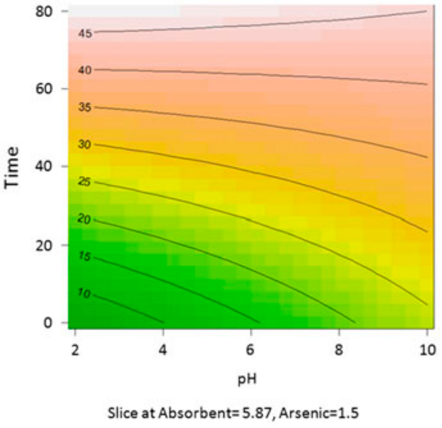

Fig. 2. Contour plots for the effect of pH and time on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

3.2. Effect of different parameter

Figs. 2–7 show the interactive effects of independent variables on the responses. In these Figures, red and green colors represent high and low removal efficiency of arsenic, respectively. The effects of pH and detention time on the removal efficiency of MMT are shown in Fig. 2. As it can be seen, the removal efficiency of arsenic by MMT meets its peak (45%) when the detention time increases from 10 to 80 min at a pH range of 6–8.

In Fig. 3, the effects of pH with adsorbent dosage on removal efficiency of arsenic by MMT are shown. Based on the result, an increase from 2 to 10 g/lit in adsorbent dosage improves the efficiency of the adsorption process.

The same range of pH 6–8 was seen to be the suitable pH of the solution in this matter. The effects of pH with arsenic concentration on the removal efficiency by the same adsorbent are shown in Fig. 4. It was observed that removal efficiency increases at optimum pH when the arsenic concentration lowers from 2.5 to 0.5. At alkaline pH of the solution and 0.5 mg/l of arsenic concentration, the removal efficiency reached 55%.

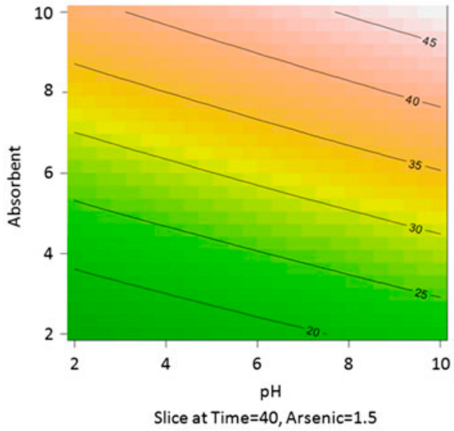

Fig. 3. Contour plots for the effect of pH and adsorbent dosage on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

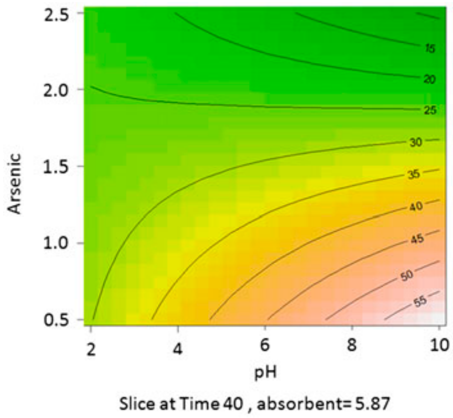

Fig. 4. Contour plots for the effect of pH and arsenic concentration on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

The effects of detention time with adsorbent dosage, and also detention time with arsenic concentration on the removal efficiency of MMT can be seen from Figs. 5 and 6, respectively. As it is shown in red color in Fig. 5, increasing the dosage and detention time improves the efficiency of arsenic removal (55%).

Fig. 6 shows that the maximum removal efficiency of arsenic, considering the interactive roles of the concentration and detention time on the response, is 50% with 80 min of detention time and 0.5 mg/l of arsenic concentration.

The interactive effects of arsenic concentration with adsorbent dosage are shown in Fig. 7. Based on the results, the maximum removal efficiency of arsenic (55%) is related to the increase in adsorbent dosage and decrease in arsenic concentration.

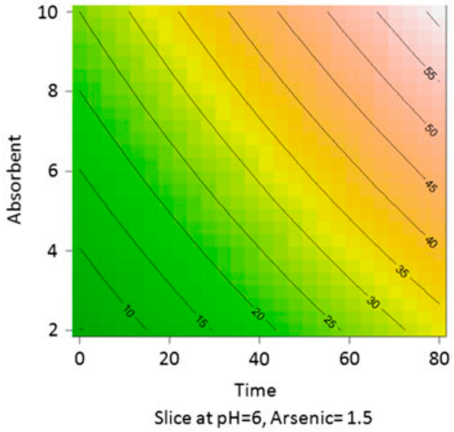

Fig. 5. Contour plots for the effect of time and adsorbent dosage on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

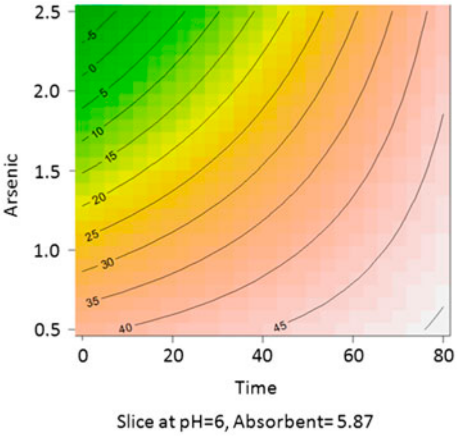

Fig. 6. Contour plots for the effect of time and arsenic concentration on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

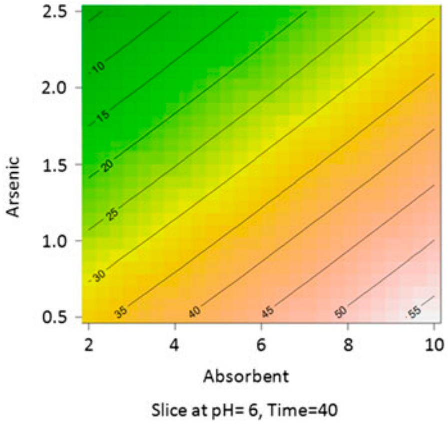

Fig. 7. Contour plots for the effect of adsorbent dosage and arsenic concentration on arsenic adsorption using MMT. Values on contour plots represent arsenic removal (%).

Figs. 8 and 9 show the interactive effects of independent variables on the efficiency of MMT@PEI. Similar to the above-mentioned figures, red and green colors represent high and low removal efficiency of arsenic, respectively.

The effects of pH along and also detention time with arsenic concentration on the removal efficiency of MMT@PEI are respectively shown in Figs. 8 and 9.

It was seen that lowering the concentration from 2.5 to 0.5 mg/lit at pH 6–8 increases the efficiency to 90%.

4. Discussion

4.1. Assessing adsorption rate of arsenic by variance analysis

From the statistical point of view and also based on the results of variance analysis, shown in Tables 6 and 7, the developed model for the application of both MMT and MMT@PEI in removing arsenic from water resources is significant (p-value < 0.05) [18]. This, in fact, shows that the selected variables are highly credible in this issue. In addition, R software's output was a polynomial-quadratic first-order (FO-PQ) model for arsenic removal.

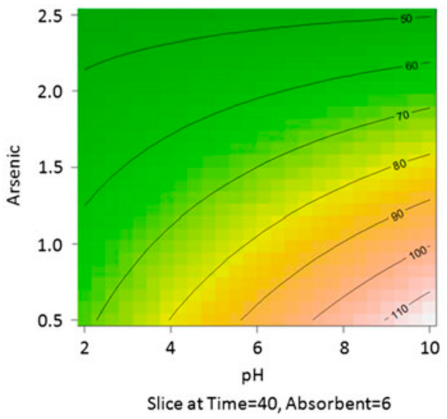

Fig. 8. Contour plots for the effect of pH and arsenic concentration on arsenic adsorption using MMT@PEI. Values on contour plots represent arsenic removal (%).

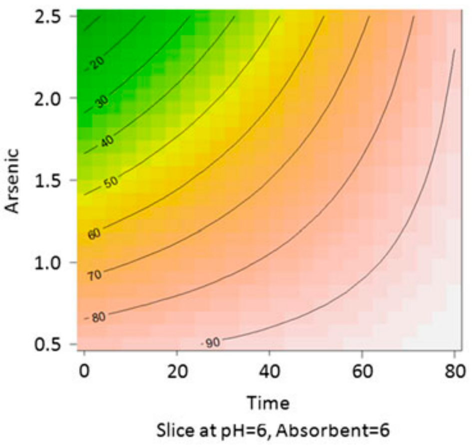

Fig. 9. Contour plots for the effect of time and arsenic concentration on arsenic adsorption using MMT@PEI. Values on contour plots represent arsenic removal (%).

4.2. Effect of pH on adsorption of arsenic

Based on the results, pH, as an independent variable, significantly affects the arsenic removal by MMT and MMT@PEI. Previously conducted studies also mentioned pH as an important factor affecting the process of adsorption [19]). Maximum adsorption of arsenic by montmorillonite (45%) was seen at pH 6; for MMT@PEI, the maximum of 90% removal efficiency was met at pH 6–8. Based on the results, the adsorption of arsenic depends highly on this variable. This could be due to the changes in arsenic form at different pH. In other words, arsenic changes to arsenate ($H_3As–O_4$) when the pH of water is between 6 and 8; it is noteworthy that this form can be efficiently adsorbed by proper media [20]. The following equations also indicate the dependence of arsenic's form on the level of pH [2]:

$$H_3AsO_4 \leftrightarrow H_2AsO_4^- + H^+, \quad pK_{a1} = 2.3 \tag{3}$$

$$H_2AsO_4^- \leftrightarrow HAsO_2^{4-} + H^+, \quad pK_{a2} = 6.8 \tag{4}$$

$$HAsO_2^{4-} \leftrightarrow AsO_3^{4-} + H^+, \quad pK_{a3} = 11.6 \tag{5}$$

The forms of arsenic expected to be found at pH 3–6 are mostly H_2AsO^{4-} and $HAsO_2^{4-}$, while the anion form of H_2AsO^{4-} exists at higher pH (pH 8–11). It should be mentioned that the presence of these two forms at the same time occurs when the pH of the solution is between 6 and 8. Hence, it can be seen how the adsorption rate of arsenic is dependent on pH. At pH 4, based on the above-mentioned Eqs. (1)–(4), arsenate is mostly present in its natural form (H_3AsO_4) and monovalent, but the former one is the polyvalent form found at this pH. That is why the adsorption rate at pH 4 is lower than at pH 6. Increasing the pH from 6 to 8 changes the monovalent arsenate to polyvalent form, which can be easily adsorbed [16]. In a study conducted by Chutia et al., it was found that pH 6 is the optimum level in removing arsenate by zeolite

Table 6
ANOVA results for arsenic removal by MMT as the response (Y_1)

Source	Sum of squares	df	Mean square	*F*-value	Pr (>*F*)
FO (X_1, X_2, X_3, X_4)	3,556.9	4	889.21	63.5275	9.906e−11
TWI (X_1, X_4)	123.7	1	123.70	8.8376	0.007818
TWI (X_2, X_4)	105.5	1	105.53	7.5394	0.012852
PQ (X_1, X_2, X_3, X_4)	2,454.7	4	613.68	43.8427	2.430e−09
Residual	265.9	19	14.00	–	–
Lack of fit	147.1	13	11.31	0.5710	0.813345
Pure error	118.9	6	19.81	–	–

Table 7
ANOVA results for arsenic removal using MMT as the response (Y_1)

Source	Sum of squares	df	Mean square	*F*-value	Pr (>*F*)
FO (X_1, X_2, X_3, X_4)	9,363.4	4	2,340.85	132.8998	4.208e−13
TWI (X_1, X_2)	105.5	1	105.47	5.9881	0.0249
TWI (X_1, X_4)	122.3	1	122.32	6.9448	0.017
TWI (X_2, X_4)	309.6	1	309.58	17.5764	0.0005
TWI (X_3, X_4)	243.7	1	243.67	13.8343	0.0015
PQ (X_1, X_2, X_3, X_4)	3,246.4	3	1,082.14	61.4373	1.181e−09
Residual	317	18	17.61	–	–
Lack of fit	316.6	13	24.35	270.4240	0.318
Pure error	0.5	5	0.09	–	–

media modified with surfactants [4]. In addition, Chen et al., reported pH 6–8 to be the optimum level of pH in arsenate adsorption by volcanic ash modified by ferric [8]. Ansar et al., studied the adsorption of arsenic by MMT and reported pH 6 to be the optimum level of this variable in their work [12]. Also, in a study conducted by Yousef et al., on arsenic removal by zeolite modified with HDTMA reported that the optimum pH ranges from 6 to 8 [21]. In another study conducted by Oriano et al., aimed to evaluate the performance of modified montmorillonite with poly[N-(4-vinylbenzyl)-N-methylD-glucamine], 90% removal efficiency of arsenic was found at pH 6 [22].

4.3. Effect of detention time on arsenic adsorption

Based on the results, the rate of arsenic adsorption was positively correlated with the detention time of the process; in other words, the efficiency gradually increased when the detention time of the process increased from 20 to 80 min. The maximum removal efficiency for MMT was found to be at the detention time of 80 min, although the effect of this variable on the performance of MMT@PEI was not significant. With 10 min detention time, the removal efficiency of 40% was observed for MMT. In fact, its efficiency in removing arsenic was seen to be positively correlated with the detention time. Nemade et al., applied red soil for removing arsenic. They reported that the efficiency of removing arsenic increases with the increase in detention time. Also, they found 50 min to be the optimum detention time for their work [23]. In addition, the optimum detention time of 1 h for removing arsenic by modified montmorillonite with poly[N-(4-vinylbenzyl)-N-methylD-glucamine] was reported by Oriano et al. [22].

4.4. Effect of arsenic concentration on its removal

In this study, the concentration of 1–2 mg/l of arsenic was used in the experiments. The results showed that the efficiency of arsenic removal increases when the concentration decreases.

4.5. Comparing the performance of MMT with MMT@PEI in removing arsenic

Based on the results, arsenic can be more efficiently adsorbed by MMT@PEI than by MMT. Applying polyethyleneimine to modify the adsorbent can be the main reason for this difference in the removal efficiency. In fact, polyethyleneimine, which is an agent with positive electron, increases the positive sites over the adsorbent's surface. We speculate that this could be the main reason for this result. In this study, we found that modifying montmorillonite using polyethyleneimine significantly increases the adsorption rate of the media in removing arsenic from water resources. This can be seen from the difference in the adsorption rate of arsenic by 97 and 45% over MMT@PEI and MMT Medias, respectively.

5. Conclusion

The performance of two adsorbents, MMT and MMT@PEI as mentioned above, in removing arsenic from water resources was evaluated and compared with each other in this study. The maximum adsorption rate occurred at high dosage of adsorbents and low concentration of arsenic. Applying RSM and also variance analysis, FO-PQ model was achieved. Based on the results, modified montmorillonite using polyethyleneimine efficiently removes arsenic from water and can be considered as a suitable choice in handling arsenic problem in the polluted water resources.

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