



## Arsenic removal from drinking water with conventional and modified adsorbents: the factorial design of experiments

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### ABSTRACT

Many areas of Pakistan are facing the problem of arsenic concentrations in underground water exceeding WHO limits. The kinetics, isotherm, and column efficiency of arsenic removal were tested for the following four adsorbents: alumina, iron-doped alumina (FA), and iron- and aluminum-doped brick particles (BF and BA). Alumina removed 85–99% arsenic in batch and continuous processes, respectively, with 1.22 mg/g adsorption capacity. FA had a higher adsorption capacity of up to 3.03 mg/g with a longer time to attain equilibrium. BF and BA had adsorption capacities of 0.16 and 0.02 mg/g, respectively, and took longer time to attain equilibrium. Statistical factorial design  $2^4$  showed that the variables were significant and optimized the process.

*Keywords:* Arsenic; Alumina; Iron doping; Aluminum doping; Drinking water

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### 1. Introduction

Arsenic contamination in drinking water is a major issue for water treatment scientists and engineers. Once underground water is contaminated with arsenic, the only solution is to find an alternative water source or treat the water for arsenic removal. Arsenite, arsenate, and organic arsenic are the different forms of arsenic. Arsenite is more toxic with higher cellular uptake and is more difficult to remove by conventional technologies [1–6].

The arsenic removal technologies were already tested including adsorption, oxidation, co-precipitation, coagulation, lime treatment, ion exchange, membrane technologies, and ion exchange membrane

[1,7–19], some of which have already been applied on a commercial scale. The AWWA reference book has discussed many arsenic removal technologies [20]. Both commercial and domestic scale technologies are being tested for arsenic removal, although each has its own pros and cons. Potential treatments at both the scales are considered in this research to optimize different technologies.

On a large scale, adsorption is one of the most commonly used processes [16,21]. Activated alumina (AA) has been extensively studied as an adsorbent for arsenic removal [22,23]. AA can remove a large quantity of arsenic due to its high internal surface area ranging from 200 to 300 m<sup>2</sup>/g. The adsorption of arsenic on AA is technically ligand exchange or chemisorption [24]. The arsenic removal by AA

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depends on various factors, such as pH, temperature, and initial arsenic concentration.

In this study, conventional and modified adsorbents are tested for arsenic removal efficiencies. The kinetics, adsorption capacity, and efficiency of adsorbent column for arsenic removal are tested with AA and modified AA. Brick particles, a cheap material, are doped with iron and aluminum salts to increase the arsenic removal efficiency, which is optimized by using statistical analysis and factorial design to study the effects of independent variables and their interactions. Different variables affecting the overall process efficiency were tested to optimize the arsenic removal by activated and iron-doped alumina (FA). The factorial design  $2^4$  was used to find their interactions and thereby minimize the overall process efficiency [9–11, 25].

## 2. Materials and methods

### 2.1. Preparation of materials

#### 2.1.1. Activated alumina

Alumina from Merck (for chromatographic adsorption analysis) was sieved and to afford a +62  $\mu\text{m}$  fraction that was then washed with DI water to remove the fine suspension remaining in the supernatant and easily settled alumina was obtained and activated for further use.

#### 2.1.2. Iron-doped alumina

Alumina (+62  $\mu\text{m}$  fraction) was washed, dried, and soaked in freshly prepared 10% ferrous sulfate solution. The alumina changed from white to red color. The supernatant was drained and collected for iron estimation. FA was dried at 80°C for 8 h, calcined at 600°C for 6 h and left to cool in a furnace. This red material was used in this study without any further treatment. The FA contained 37 mg Fe/g alumina.

#### 2.1.3. Alumina-doped red brick particles

Red brick was ground and sieved to ensure a consistent particle size of 1–2 mm. This fraction was then washed to remove the fine powder and air dried at ambient temperature for 3 days. The resulting brick particles were soaked in 10%  $\text{Al}_2(\text{SO}_4)_3$ , and dried at 80°C for 6 h after the supernatant had been drained. The obtained material was dipped in dilute NaOH solution for 1 h and then the supernatant was drained. This material was dipped in distilled water for 2 h to stabilize, the supernatant was drained and the

particles left to air dry. The dried material was calcined at 600°C for 6 h and left to cool in a furnace.

#### 2.1.4. Iron-doped red brick particles

The brick particles (1–2 mm size) were soaked in freshly prepared 10% ferrous sulfate solution, the supernatant was drained, and the solid material was dried at 80°C for 6 h and calcined at 600°C in a furnace for 6 h. This material was used without any further treatment.

## 2.2. Methods

### 2.2.1. Kinetic study

One-liter samples of arsenite solution were prepared with 100  $\mu\text{g}/\text{L}$  arsenic in four different round bottom flasks and the pH was adjusted to 6.5. Known amounts of the four adsorbents, i.e. AA, FA, BA, and BF, were added and the mixture was shaken continually until the end of the test. From each flask, samples of supernatant were taken at predetermined time intervals to check the decrease in arsenic concentration. Samples were acidified with nitric acid and kept at 4°C until analysis.

### 2.2.2. Isotherm study

For each adsorbent, 200 ml of 100  $\mu\text{g}/\text{L}$  arsenite solution was prepared in six round bottom flasks and the pH was adjusted to 6.5. Increasing amounts of adsorbent were added and the mixture was shaken overnight. Samples of supernatants were taken from each flask, acidified with nitric acid and kept them at 4°C until analysis.

### 2.2.3. Continuous column run

A glass column (4.5 cm i.d.) of 1,000 ml volume was used as a vertical reactor. The column was filled with 900 g of AA to a total volume of 900 ml using the slurry method. A top layer of 100 ml of water was maintained above the AA bed to avoid bed disturbance. The empty bed contact time was maintained at 15 min throughout the experiment. Samples of 50 ml were collected out of 500 ml batch collections of effluent water. The samples were acidified with nitric acid and kept at 4°C until analysis.

### 2.2.4. Statistical analysis by the factorial design

Statistical models of the reactions were developed by factorial designs and a minimum number of well

chosen experiments was performed to determine the optimal values of the process parameters. Factorial design is an empirical technique used to evaluate the relationship between the experimental variables and the corresponding response. The factorial design  $2^4$  was chosen for this experiment with four independent variables: time ( $X_1$ ), temperature ( $X_2$ ), pH ( $X_3$ ) and arsenate concentration ( $X_4$ ) [25,26].

The behavior of the system is explained by the following quadratic equation.

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_1X_2 + b_6X_1X_3 + b_7X_1X_4 + b_8X_2X_3 + b_9X_2X_4 + b_{10}X_3X_4 + b_{11}X_1X_2X_3 + b_{12}X_1X_2X_4 + b_{13}X_1X_3X_4 + b_{14}X_2X_3X_4 + b_{15}X_1X_2X_3X_4 \quad (1)$$

where  $b_0$  represents the global mean and  $b_{1*}$  the regression coefficient corresponding to the main factors, effects and interactions. The experimental plan was developed by MINITAB 15 statistical software and the results obtained accordingly were interpreted by the same software to estimate the response of the dependent variable, i.e. the percentage removal of arsenic.

### 2.3. Arsenic analysis

Water samples from all processes were acidified with nitric acid and kept at 4°C until analysis. The water samples were analyzed by the ICP-OES Perkin Elmer, Optima 5000 using the hydride generation method.  $\text{NaBH}_4$  0.5% (w/v) was used as a reducing agent dissolved in 0.05% NaOH. The reducing agent and the acidified sample were continuously mixed in the hydride generation assembly with a three-channel peristaltic pump. The arsine gas generated by the mixing was striped with argon gas into the plasma and the arsenic was estimated at a wavelength of 189.042 nm.

## 3. Results and discussion

### 3.1. Kinetic studies

The rate of arsenic removal by AA and the three modified materials were tested to compare their kinetics (Fig. 1). During the initial reaction time, the adsorption on AA was faster. Once equilibrium was reached, the final arsenic removal with FA (92%) was higher than that with AA (85%), which suggests that this technique can be utilized in large-scale facilities to increase the total arsenic removal, although at longer empty bed contact time. BA and BF were developed from brick particles, which is a very cheap raw

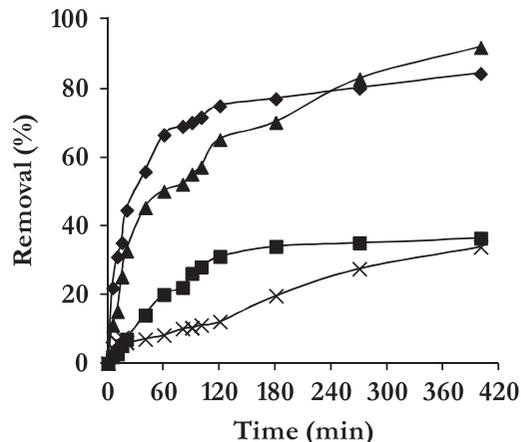


Fig. 1. Kinetics of arsenic removal by different adsorbents (◆ AA ▲ FA ■ BF × BA).

material. Unprocessed brick particles were not active in arsenic removal. BA and BF removed 34 and 36% of the arsenic, respectively. These materials can be used domestically by simple application. The removal rate was very slow but appreciable removal was attained after a contact time of 5–7 h (Table 1).

### 3.2. Isotherm study

FA afforded a higher adsorption capacity than unprocessed AA. Adsorption capacities were 1.22 and 3.03 mg/g for AA and FA, respectively. This improvement promises to be highly beneficial at large-scale water treatment facilities that can reduce the bed size and increase the treated bed volumes. According to their adsorption capacity, AA and FA can theoretically treat 12,000 and 30,000 bed volumes of water containing 100 µg/L of arsenic, respectively. Further study is needed to optimize the conditions for using FA as an alternative adsorbent to AA. The increase in adsorption capacity and decrease in adsorption rate may have been due to the differences in the affinity and the number of active sites on the adsorbents; further study will be necessary to confirm this.

Table 1  
Adsorption capacities of the four different adsorbents for arsenic calculated from isotherms

Adsorbent	Adsorption capacity (mg/g)	$r^2$
Activated alumina	1.22	0.99
Iron doped alumina	3.03	0.95
Iron doped brick	0.16	0.97
Aluminum doped brick	0.02	0.91

Although the adsorption capacities of the two low-cost materials developed from brick particles were very low at 0.16 and 0.02 mg/g for BF and BA, respectively, they can nevertheless be used to treat 250 and 1,600 bed volumes of water containing 100 µg/L of arsenic, respectively, in large-scale application. BA and BF can be utilized at the domestic scale in the form of modified kitchen earthenware, especially in small communities where installation of large-scale water treatment plants is not economically feasible.

### 3.3. Column study

Practical application of AA was tested in a glass column packed with AA. In another study, the maximum measured arsenic in the Lahore district was above 100 µg/L. The column was run with real tap water containing 20 µg/L arsenic and spiked with 100 µg/L of arsenic to give a final arsenic concentration of 120 µg/L. This AA-packed column was tested to treat 30 L of water: 99% removal of arsenic water was achieved with less than 1 µg/L of residual arsenic in the treated water. The AA column remained active and according to theoretical adsorption capacity would have been capable of treating 12,000 L of water.

### 3.4. Statistical analysis by the factorial design

The independent variables studied for their optimization were time ( $X_1$ ), temperature ( $X_2$ ), pH ( $X_3$ ),

Table 2

Four independent variables and their levels used in this study for both AA and FA

Independent variables		Low level – 1	High level + 1
Time (min)	$X_1$	5	20
Temperature (°C)	$X_2$	10	30
pH	$X_3$	5	7
As concentration (µg/L)	$X_4$	10	100

AA and FA are given in Table 3. Arsenic removal varied between 16 to 97% for AA and 8 to 84% for FA. The students “*t*” test and Fisher “*F*” test were used to determine the significance of the regression coefficients of the parameters. The *p* values were used as a tool to check each of the interactions among the variables. The variables with *p* value less than 0.1 were considered significant. More significant were the terms of coefficients when the value of “*t*” was large and the value of *p* was small. The effects of the variables studied were significant as the *p* value for all the variables was less than 0.1 for AA and only one variable was insignificant for FA with *p* above 0.1 (Table 4). The dependence of the arsenic removal on the main effects is shown in the Fig. 2.

Arsenic removal by AA and FA can be predicted using the coefficients given in Table 4. The variables with a *p* value above 0.1 were insignificant and were therefore removed from the model.

$$Y_{AA} = 59.73 + 9.79X_1 + 9.83X_2 + 5.92X_3 + 12.68X_4 - 3.84X_1X_2 - 1.95b_6X_1X_3 - 4.93X_1X_4 - 6.35X_2X_3 + 10.61X_2X_4 + 4.42X_3X_4 + 1.31X_1X_2X_3 + 2.00X_1X_2X_4 + 4.58X_1X_3X_4 - 7.00b_{14}X_2X_3X_4 - 2.74X_1X_2X_3X_4 \quad (2)$$

$$Y_{FA} = 50.91 + 4.52X_1 + 6.21X_2 - 4.79X_3 + 17.53X_4 + 0.87X_1X_2 - 2.35X_1X_3 + 0.68X_1X_4 + 2.82X_2X_3 - 3.70X_2X_4 - 1.50X_3X_4 + 2.36X_1X_2X_3 + 1.23X_1X_3X_4 - 2.68X_2X_3X_4 - 1.80X_1X_2X_3X_4 \quad (3)$$

and initial arsenic concentration ( $X_4$ ). The levels of the four independent variables studied for both AA and FA are given in the Table 2. The combined effects of these parameters on the response of the dependant variable, i.e. the percentage arsenic removal, were analyzed by the statistical software MINITAB 15. The experimental results obtained for 2<sup>4</sup> full factorial design and the corresponding fits and residuals for

### 3.5. Analysis of variance

The statistical significance of mean square due to regression and mean square residual error was tested using ANOVA test. ANOVA technique subdivides the total variation in a set of data into component parts associated with specific sources of variation for the purpose of testing hypotheses on the parameter of the

Table 3  
Experimental data, arsenic (As) removal, fits and residuals

Run	Time (min), $X_1$	Temp. ( $^{\circ}$ C), $X_2$	pH, $X_3$	Conc. ( $\mu$ g/L), $X_4$	As removal%		Fit		Residual	
					AA	FA	AA	FA	AA	FA
1	5	10	5.0	10.0	16.14	20.58	15.84	21.32	-0.29	0.74
2	20	10	5.0	10.0	78.69	41.00	78.10	43.22	-0.59	2.22
3	5	30	5.0	10.0	33.28	38.32	32.78	37.20	-0.50	-1.11
4	20	30	5.0	10.0	55.69	46.04	55.48	44.96	-0.21	-1.08
5	5	10	7.0	10.0	40.78	19.22	38.71	19.22	-2.07	0.00
6	20	10	7.0	10.0	57.76	8.72	58.65	10.17	0.89	1.45
7	5	30	7.0	10.0	42.53	40.45	42.02	40.45	-0.51	0.00
8	20	30	7.0	10.0	54.31	49.09	54.82	50.53	0.50	1.44
9	5	10	5.0	100.0	25.09	65.64	25.65	66.59	0.55	0.95
10	20	10	5.0	100.0	31.47	78.27	30.94	78.12	-0.53	-0.14
11	5	30	5.0	100.0	93.97	69.43	94.03	70.23	0.06	0.79
12	20	30	5.0	100.0	97.69	84.13	97.68	83.95	0.00	-0.18
13	5	10	7.0	100.0	64.80	57.47	64.91	57.11	0.11	-0.36
14	20	10	7.0	100.0	86.12	62.26	86.43	61.91	0.32	-0.35
15	5	30	7.0	100.0	85.14	60.15	85.63	59.05	0.49	-1.10
16	20	30	7.0	100.0	94.58	70.01	94.06	70.59	-0.52	0.58

Table 4  
Coefficients,  $t$ ,  $p$  and standard deviations for arsenic removal by AA and FA

Term	Effect		Coefficient		Standard deviation		$t$		$p$	
	AA	FA	AA	FA	AA	FA	AA	FA	AA	FA
Constant			59.73	50.91	0.17	0.25	344.47	206.91	0.000	0.000
Time, $X_1$	19.57	9.04	9.79	4.52	0.17	0.25	56.44	18.36	0.000	0.000
Temp, $X_2$	19.66	12.41	9.83	6.21	0.17	0.25	56.68	25.22	0.000	0.000
pH, $X_3$	11.84	-9.57	5.92	-4.79	0.17	0.25	34.14	-19.45	0.000	0.000
Conc., $X_4$	25.37	35.06	12.68	17.53	0.17	0.25	73.15	71.24	0.000	0.000
$X_1X_2$	-7.68	1.74	-3.84	0.87	0.17	0.25	-22.14	3.54	0.000	0.003
$X_1X_3$	-3.90	-4.69	-1.95	-2.35	0.17	0.25	-11.25	-9.54	0.000	0.000
$X_1X_4$	-9.85	1.36	-4.93	0.68	0.17	0.25	-28.40	2.77	0.000	0.014
$X_2X_3$	-12.70	5.64	-6.35	2.82	0.17	0.25	-36.63	11.46	0.000	0.000
$X_2X_4$	21.21	-7.39	10.61	-3.70	0.17	0.25	61.16	-15.02	0.000	0.000
$X_3X_4$	8.84	-2.99	4.42	-1.50	0.17	0.25	25.49	-6.08	0.000	0.000
$X_1X_2X_3$	2.62	4.73	1.31	2.36	0.17	0.25	7.56	9.60	0.000	0.000
$X_1X_2X_4$	4.00	0.49	2.00	0.25	0.17	0.25	11.52	1.00	0.000	0.331
$X_1X_3X_4$	9.15	2.47	4.58	1.23	0.17	0.25	26.39	5.01	0.000	0.000
$X_2X_3X_4$	-14.00	-5.35	-7.00	-2.68	0.17	0.25	-40.35	-10.87	0.000	0.000
$X_1X_2X_3X_4$	-5.48	-1.80	-2.74	-1.80	0.17	0.25	-15.80	-7.30	0.000	0.000

model [25]. The ANOVA data in Table 5 indicate that the  $p$  value for all the terms for both AA and FA was less than the chosen alpha level (0.05), which supports the fitness of the data obtained to the regression model developed. The coefficient of determination ( $R^2$ ) for arsenic removal by AA and FA was 99.93 and 99.79%, which indicated that the model could explain

99.93 and 99.79% of the sample variation by the selected independent variables for AA and FA, respectively. This model could not explain only 0.07 and 0.21% of the sample variation, respectively. The  $R^2$  (adjusted) for AA and FA were equal to the  $R^2$  (calculated), which demonstrated the accuracy in the selection of main effects variables.

Table 5  
Analysis of variance for AA and FA for percentage arsenic removal

Source	DF		Seq SS		Adj SS		Adj MS		F		p	
	AA	FA	AA	FA	AA	FA	AA	FA	AA	FA	AA	FA
Main effects	4	4	12,427	12,451	12,427	12,451	3,107	3,113	3,229	1,607	0.000	0.000
2-Way interactions	6	6	6,885	978	6,885	978	1,147	163	1,192	84	0.000	0.000
3-Way interactions	4	4	2,420	458	2,420	458	605	115	629	59	0.000	0.000
4-Way interactions	1	1	240	103	240	103	240	103	250	53	0.000	0.000
Residual error	15	15	14	29	14	29	1	2				
Total	31	31	21,986	14,022								

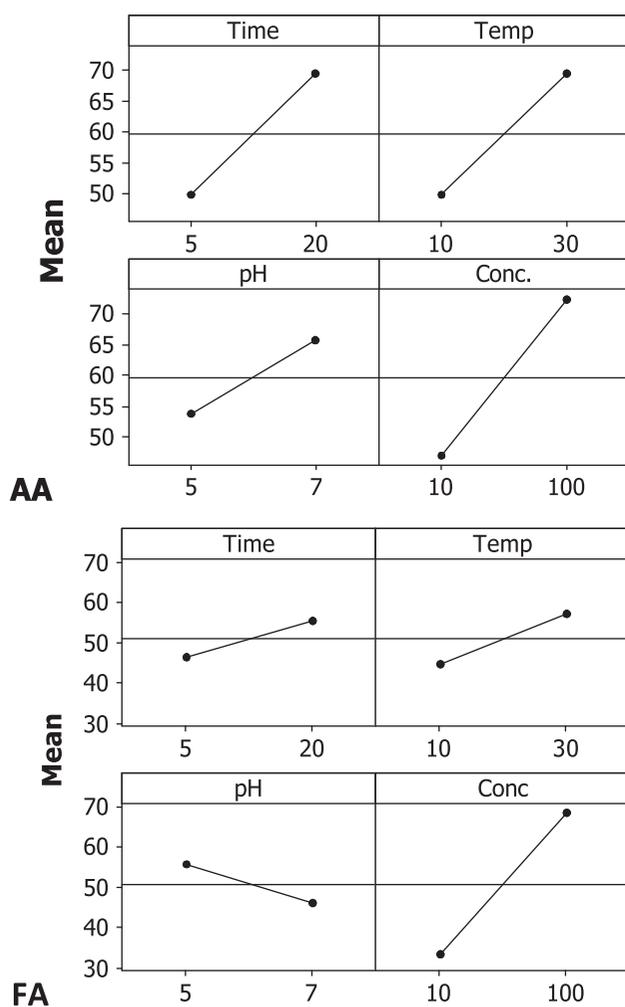


Fig. 2. Main effects plots for AA and FA in the main effects vs. data means of As removal.

The optimization of these processes for arsenic removal suggested that AA can remove up to 97.68% of arsenic in a batch process at 20 min, 30°C, pH 5, and an initial arsenic concentration of 100 µg/L. FA can remove up to 83.95% of arsenic in a batch process

after 20 min at 30°C and pH 5 with an initial arsenic concentration of 100 µg/L. The predictions were compatible with the actual data obtained in this study.

Statistical analysis showed that an increase in each of the variables, especially time ( $X_1$ ), increased the efficiency of AA. In the case of FA, all factors except pH, and especially the arsenic concentration ( $X_4$ ), increased the arsenic removal. Increased pH decreased the arsenic removal by FA. However, during optimization it was found that the ideal conditions were the same for both AA and FA, possibly because the negative effect of pH alone ( $X_3$ ) was nullified by the combined positive effects, especially that of pH and temperature ( $X_2X_3$ ).

In the batch test the mechanism of adsorption was same on both AA and FA. The only difference was that the rate of adsorption resulted in 20 min being insufficient to attain equilibrium; therefore, FA could not reach its equilibrium in this time and showed lower arsenic removal.

#### 4. Conclusion

Four different conventional and modified adsorbents were tested for arsenic removal from water. The adsorption capacities of AA, FA, BF, and BA were 1.22, 3.03, 0.16, and 0.02 mg/g, respectively. The adsorption capacity and the final arsenic removal were higher with FA but the initial arsenic removal rate was higher with AA. This was attributed to the difference in affinity and the number of active sites on the adsorbents. These materials are recommended for large-scale water treatment facilities. Statistical factorial design  $2^4$  was used to optimize the conditions for batch testing of arsenic removal by AA and FA. Equations were developed to predict the optimized operational conditions.

The processed brick particles had lower adsorption capacities and slower kinetics. Nevertheless, as these materials are very cheap they can be used at a domestic scale for single use as modified earthenware.

Further work is needed to study the applicability of these processes.

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