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Arsenic Oxidation Using UV-Activated Persulfate in Aqueous Solutions: Optimization Using Response Surface Methodology Based on Box-Bencken Design

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ABSTRACT

Introduction: The use of arsenic contaminated water can cause a variety of adverse health effects in humans. Therefore, it is essential to seek out a method to remove arsenic more efficiently. This study examined the amount of arsenic oxidation by response surface methodology (RSM) based on Box-Bencken design**.**

***Materials* and Methods**: In this study, oxidizing arsenite to arsenate was performed by activation of persulfate with UV and the optimal conditions determined using the RSM based on Box-Bencken design to evaluate the effects of independent variables on the response (arsenite oxidation efficiency) performance and to predict the best response rate. In this study, the effects of different parameters such as pH (3-11), concentration of persulfate (4-14 mM), and initial concentration of arsenic (0.1-0.9 mg/l) on process efficiency were investigated. The number of tests in this study was 45, and the oxidation rate was measured using the UV visible spectrophotometer (DR 6000) and the molybdate colorimetric method.

***Results*:** Increasing the concentration of arsenic increased oxidation. However, with increasing pH, the oxidation rate decreased and the highest oxidation rate at all concentrations was observed at pH 3. The value higher than R^2 (0.934) indicated that the oxidation of arsenic (v) could be determined by this model.

Conclusion: Arsenite is a highly toxic metal that is difficult to remove by conventional treatment methods, but a pre-treatment phase can convert arsenite into arsenate and facilitate the removal process. In this study, the use of UV-activated persulfate increased the efficiency of arsenic oxidation to 96%.

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Introduction

Access to safe drinking water is one of the basic physical, chemical, and biological needs of humans. High concentration of arsenic in groundwater is one of the main problems affecting the health of millions of people [1, 2]. Water-soluble arsenic in groundwater is used for drinking and irrigation purposes [3].

Exposure to high levels of arsenic can lead to chronic toxicity and its symptoms include skin lesions (keratosis, melanosis, pigmentation, and blackleg), internal cancers, such as lung, liver,

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breast, and kidneys, and heart disease as well as growth anomalies in children [4, 5].

Due to the carcinogenicity of arsenic, World Health Organization lowered allowed arsenic levels in drinking water from 50 µg/l to 10 µg/l, but drinking water standards aim to reduce arsenic levels in water to less than parts-per million. Arsenite and arsenate are the forms of mineral arsenic, and the concern about drinking water supplies is due to these two forms of arsenic [6].

Various methods have been proposed for the removal of arsenic, such as coagulation and filtration, membrane processes such as reverse osmosis and electrodialysis, as well as absorption processes, but one step of oxidation maximizes the efficiency before implementation of any of the processes [7].

United States Environmental Protection Agency (USEPA) suggests to convert trivalent arsenic (arsenite) to pentavalent arsenic (arsenate) and then proceed with the removal of arsenate to achieve a highly efficient arsenic removal from drinking water during a pre-treatment step before the main removal process (using an oxidizing chemical or aeration step) [8].

In recent years, the use of Advanced Oxidation Processes (AOPs) as an acceptable and efficient technology has always attracted the attention of researchers and operators of water and wastewater treatment plants. Meanwhile, hydrogen peroxide and ozone have been successfully used in laboratory and real scales, but the short life of oxidizing agent, low solubility, storage and displacement, and high cost are the disadvantages of this method.

In recent years, a compound called persulfate with oxidation potential of 2.01 V has been identified. Persulfate is capable of oxidizing toxic and resistant organic compounds [9].

Some of the advantages of persulfate are low cost, high stability of the radical produced from it in different conditions, high solubility, solid form and, thus, ease of handling and storage. Regardless of these advantages, extensive studies on the use of persulfates have shown that the degradation of organic materials by persulfate at room temperature is low and slow [10, 11].

Therefore, activation of persulfate is necessary to accelerate the oxidation process. The activation of persulfate, as an AOP, is performed with heat, UV light, and intermediate metals. The final product of the activation is sulfate radical with an oxidation potential of 6.2 V. The reaction below shows the thermal activation of persulfate [9, 12, 13].

$S_2O_8^{2-}$ + heat or UV → $SO_4^{\bullet-}$

The UV/persulfate process is one of the newest and most efficient treatment methods. In addition, $SO_4^{\bullet-}$ is almost neutral and is not considered as a contaminant as the USEPA ranked it as the second agent in the water standards with a maximum allowable concentration of 250 mg/l in drinking water [14].

The RSM is a method of optimization that uses a set of mathematical and statistical techniques to model issues.

This method evaluates the interactions of several factors in a different spectrum, and illustrates the region, where the results are in that spectrum, as a three-dimensional surface.

In recent years, certain tools have been used to optimize analytical methods with significant advantages such as reducing the number of experiments, as well as reducing the use of reagents and laboratory works [6].

Given that the high concentrations of arsenic in groundwater in many countries, and no study has yet been conducted with the RSM, and also because trivalent arsenic is very toxic and hazardous, the aim of this study was to investigate oxidizing trivalent arsenic to pentavalent arsenic, which is the low-risk form of arsenic and is eliminated more easily by conventional treatment processes.

Materials and Methods

All chemicals used to prepare standard stock solutions were purchased from Merck Co., Germany [15, 16]. To adjust the pH, 0.1 M sulfuric acid and 0.1 M Sodium hydroxide were used [17]. To prepare arsenite stock solution 100 ppm, potassium

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arsenite salt of 99% purity was dissolved in distilled water.

To prepare this solution, we weighed 0.1734 g of sodium arsenite using a digital laboratory scale with precision 0.0001 g and then dissolved it in 1000 ml of distilled water. Under laboratory conditions, the concentration was synthetically kept at a range of 0.1-0.9 mg/l using deionized water (the water distilled two times) and adding sodium arsenite to it.

Then, a plexiglas reactor with a useful volume of 1, equipped with a UVc lamp (15 W), was prepared to conduct the experiments (Figure 1). The system was used discontinuously, and during the reaction time, a mechanical stirrer was used to keep the solution homogeneous. Using the appropriate materials and equipment and changing the pH by using a pH meter (HACH), and the concentration of potassium persulfate, necessary samples were collected and analyzed.

Finally, the amount of remained arsenic was measured as the response variable, which is shown by C_2 in Table 1.

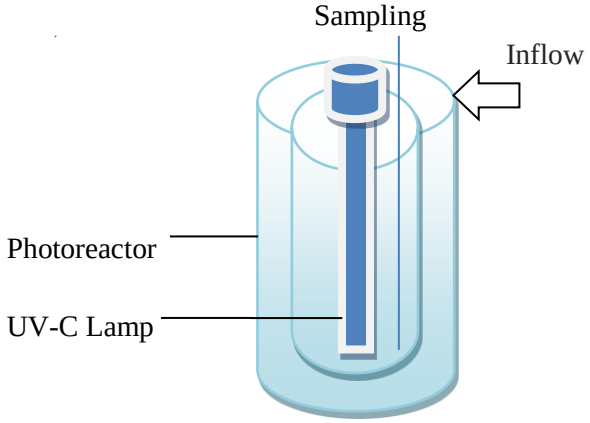


Figure 1: A schematic of the reactor used in this study

Measuring arsenate

Using UV visible spectrophotometer (DR 6000, HACH), and and the molybdate colorimetric method, the following steps were performed:

***Preparation of the reagent* A**

To prepare reagent 1, 13g of (NH_4) $6Mo_7O_{24}$ $4H_2O$ (99%, Aldrich) were mixed with 9 M H_2SO_4 (98%, Prolabo) in a 500 ml volumetric flask. This reagent (called ‘‘reagent A’’) is stable for 2 months if stored in an opaque flask.

Preparation of ascorbic acid

Another solution of 10 /100 ml ascorbic acid (99%, Aldrich) was prepared daily before use.

Measurement of As (V)

After preparing the reagents and according to the instructions, 1 ml ascorbic acid solution and 2 ml reagent A are successively added to a 40 ml sample aliquot in a 50 ml volumetric flask, and the volume completed by de-ionised water. A blank is prepared according to the same procedure using the appropriate volume of de-ionised water. The analysis is carried out in 1 cm quartz cells of the UV visible spectrophotometer [18], and the results for each of the 45 samples recorded. The RSM based on the Box-Bencken design was used to evaluate the effect of independent variables on the performance of response (arsenite oxidation efficiency) and to predict the best response rate [19].

In this study, the effect of independent variable X_1 was considered to indicate initial concentration of arsenic (mg/l), X_2 to indicate pH, and X_3 to indicate the concentration of potassium persulfate (mM) that were used at three minimum, moderate, and maximum levels of -1, 0, and +1, respectively, with three factors and five central points to determine the percentage of the error sum of squares (Table 2).

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Table 1: Test design for three levels and three factors

Run Order	Factor1: Arsenic;C_1	Factor2: pH	Factor3: S_2O_8	C_2	R
1	0.5	7	9	0.37	75
2	0.9	11	9	0.39	43
3	0.1	11	9	0.08	21
4	0.9	7	4	0.42	47
5	0.5	7	9	0.36	72
6	0.9	7	4	0.41	46
7	0.1	7	4	0.07	34
8	0.5	3	4	0.42	84
9	0.9	7	14	0.46	51
10	0.5	11	14	0.34	68
11	0.1	7	14	0.06	44
12	0.5	7	9	0.37	74
13	0.5	11	4	0.32	65
14	0.5	7	9	0.34	68
15	0.5	3	4	0.42	84
16	0.5	7	9	0.33	66
17	0.5	7	9	0.38	76
18	0.1	7	4	0.06	41
19	0.1	7	4	0.06	43
20	0.5	7	9	0.34	68
21	0.5	3	4	0.45	90
22	0.9	3	9	0.64	71
23	0.5	11	14	0.33	66
24	0.9	3	9	0.65	72
25	0.5	7	9	0.36	72
26	0.5	11	14	0.29	58
27	0.5	11	4	0.3	60
28	0.1	11	9	0.08	18
29	0.5	7	9	0.31	63
30	0.9	11	9	0.38	42
31	0.1	3	9	0.02	78
32	0.9	7	4	0.39	43
33	0.1	7	14	0.06	35
34	0.1	11	9	0.07	26
35	0.1	3	9	0.21	79
36	0.5	11	4	0.29	58
37	0.1	7	14	0.05	46
38	0.5	3	14	0.48	96
39	0.9	3	9	0.76	85
40	0.5	3	14	0.47	95
41	0.1	3	9	0.02	82
42	0.5	3	14	0.48	96
43	0.9	7	14	0.36	40.5
44	0.9	7	14	0.35	38.8
45	0.9	11	9	0.33	37.2

Table 2: The ranges and experimental values of independent variables

Variable	Symbol	+1	0	-1
Arsenic concentration	C_1	0.9	0.5	0.1
pH	pH	11	7	3
Persulfate concentration	S_2O_8	14	9	4

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The total number of experiments in the Box-Bencken design was 45 tests. The model used in the RSM is generally a quadratic relationship. The data from the Box-Bencken design were used to determine the proportion of quadratic polynomial regression equations.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{44} X_2^2$$

Ethical issues

Ethical approval was obtained from the Ethics Committee of Shahid Sadoughi University of Medical sciences, Yazd, Iran (ID: IR.SSU.SPH.REC.1395.135).

Results

According to the RSM based on the Box-Bencken design and the model presented in Table 1, after adjusting the desired pH level, and then adding the specified amounts of potassium persulfate, as the oxidizing agent, they were exposed to the UV lamp for a specified period. The values of remained arsenic (V) (C_2), were measured as the arsenic (V) and response variable.

Regarding on the recording of the initial concentration of arsenic and calculating the final concentration of arsenic (V), oxidation efficiency was recorded at each step of the experiment. The results of the study are presented in Table 1.

Based on the results of Table 1, it can be inferred that the lowest efficiency of arsenic oxidation by the UV/$k_2S_2O_8$ process is 18% and the highest efficiency 96%.

The following equation represents the experimental relationship between the experiment variables and the percentage of efficiency as encoded, and the result of the RSM is based on the Box-Bencken design:

$$\text{Oxidation efficiency} = 70.5 + (2.9 \times X_1) - (18.7 \times X_2) - (25 \times X_1^2) + (9 \times X_2^2) + (5.6 \times X_1 \times X_2)$$

As the above equation shows, a number of factors are positive and some others are negative. The negative values in the resulting equation indicate increased efficiency with decreasing the parameter and the positive values in the equation represent a direct correlation between the efficiency and the parameter.

It should be noted that X_1 represents the initial concentration of arsenic (mg/l), X_2 pH, and X_3 the concentration of potassium persulfate. To determine the quality of the proposed polynomial model, the coefficient R_2 and adjusted R^2 were used. Analysis of variance (ANOVA) was used as the statistical method to analyze the responses [20].

According to the results from ANOVA test (Table 3), the oxidation of arsenic by the UV/$k_2S_2O_8$ process was well described by the model.

It should also be noted that the effect of all studied parameters, except for persulfate, on the oxidation efficiency of arsenic is significant with regard to the statistically significant level of lower than 0.05.

The high linear regression coefficient for arsenic oxidation by persulfate (R^2 = 0.934 and R^2 adjusted = 0.917) shows that the model worked appropriately and predicted well.

The closer to 1 the R^2 value is, the greater the power of the model in describing response variations as a function of independent variables.

pH effect

The pH parameter is an important variable in the oxidation process due to its effect on the amount and type of produced radicals. For this purpose, the effects of pH 3, 9, 11 on the efficiency of the UV/persulfate process were investigated at concentrations 0.1, 0.5, and 0.9 mg/l of arsenic.

0.1 N NaOH and H_2SO_4 solutions were used to adjust the pH to the desired value. The findings are illustrated in Figure 2. According to Figure 2, the UV/persulfate process at pH 3 had the highest efficiency in removing arsenic.

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Table 3: The analysis of variance (ANOVA) for the quadratic arsenate oxidation model

Source	Mean Square	F-value	P-value
Corrected Model	1506.30	73.59	.000
Intercept	142344.99	6955.00	.000
Arsenic	201.89	9.86	.004
pH	8430.01	411.89	.000
S_2O_8	63.99	3.13	.087
Arsenic pH	387.60	18.94	.000
Arsenic S_2O_8	13.67	0.67	.420
pH S_2O_8	33.33	1.63	.211
Arsenic pH S_2O_8	.	.	.
Error	20.47		
Total			
Corrected Total			

Effect of Persulfate Concentration

To determine the optimum concentration of persulfate for the removal of arsenic using the UV/persulfate process, the concentrations 4, 9, and 14 mM of persulfate were examined. The results are illustrated in Figures 3 and 4.

As it can be observed, the oxidation efficiency of arsenic in the UV/persulfate process does not undergo a specific trend with changing the persulfate concentration.

By increasing the concentration of persulfate, an excessive amount of sulfate ion is produced which acts as an interfering radical. According to the equations below, it is an agent for the conversion of sulfate radical to persulfate. On the other hand, persulfate radical reacts with persulfate; therefore, anion sulfate is produced, all of which lead to destruction of persulfate radical and reduce the removal efficiency.

$$SO_4^{\bullet -} + S_2O_4^{2-} \longrightarrow SO_4^{2-} + S_2O_8^{\bullet -}$$

$$SO_4^{\bullet -} + SO_4^{\bullet} \longrightarrow S_2O_8^{\bullet -}$$

In the Figure 2, the interaction effects and binary interactions of the studied factors were used to determine the amount of arsenic oxidation with pH at different concentrations of potassium persulfate and arsenite.

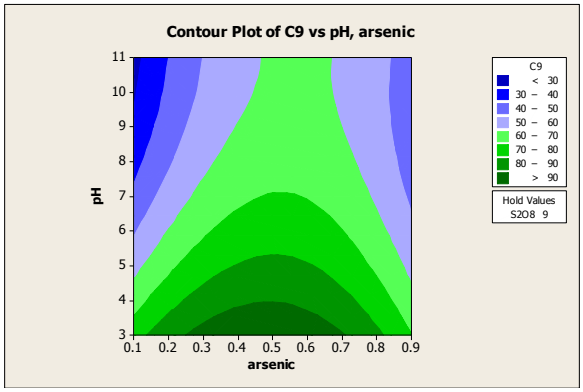


Figure 2: Interaction effects and binary interactions of the studied factors (arsenic and pH)

The effect of initial arsenic concentration

In order to investigate the effect of initial arsenic concentration on the UV/persulfate process efficiency at pH 3, concentrations 0.1, 0.5, and 0.9 mg/l of arsenic were investigated and, as shown in Figures 3 and 4, the highest oxidation (96%) carried out at 0.5 mg/l concentration with 96% efficiency.

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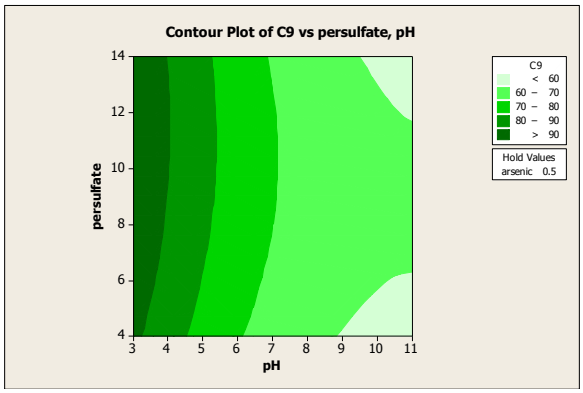


Figure 3: Interaction effects and binary interactions of studied factors (persulfate and pH)

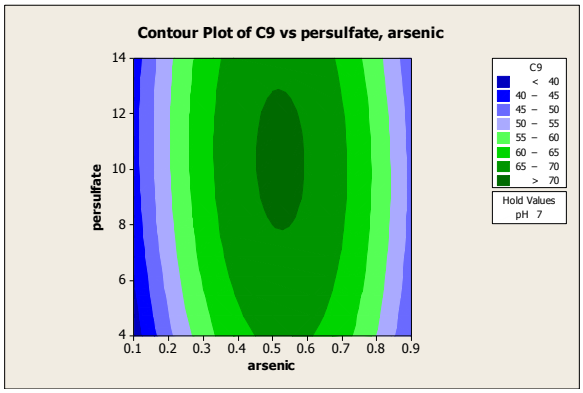


Figure 4: Interactions effects and binary interactions of the studied factors (persulfate and arsenic)

Discussion

In this study, the oxidation of arsenic 3 using the process of photooxidation with persulfate and UV light as well as using the RSM based on the Box-Bencken design was investigated, and the effects of the studied variables were investigated. It should be noted that UV alone cannot oxidize arsenite; therefore, it is used as an activator in this process [21]. In addition, persulfate is not capable of producing sulfate radical in the absence of UV light.

The effect of initial arsenic concentration

Investigating changes in the initial concentration of arsenic showed that oxidation efficiency increased with increasing the concentration of the contaminant, so that the oxidation efficiency increased from 80% to 96% at 0.1 mg/l of arsenic when compared with 0.5 mg/l of arsenic in similar conditions, but the oxidation efficiency did not increase when the arsenic concentration increased from 0.5 mg/l to 0.9 mg/l.

Increasing the concentrations of contaminants leads to the formation of intermediate products that absorb UV rays, and therefore less UV ray is absorbed by persulfate, and consequently, less $SO_4^{\bullet -}$ is produced [22].

Another reason for this is that in a steady radiation of UV, a certain amount of radicals are produced in the environment. This amount of radicals is capable of absorbing and oxidizing a certain amount of contaminant molecules [23, 24].

Therefore, if the initial concentrations of the pollutant increase, the amount of radicals in the environment is not sufficient to oxidize the additional molecules. Thus, it can be argued that with increasing the concentration, all the radicals in the pollutant oxidation are consumed and, as a result, the oxidation efficiency will be decreased [25] .

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pH effect

In the oxidation process of arsenite, the oxidation rate increased with decreasing pH, and the highest oxidation efficiency was obtained at pH 3 with 96% efficiency and the lowest efficiency at pH 11 with 20% efficiency.

These findings indicate that one of the factors influencing the speed of the chemical reactions is the pH of the environment, which affects treatment in different ways. Jamshidi et al. evaluated phenol removal using advanced photochemical oxidation processes and concluded that the best removal efficiency was obtained at acidic pH [26].

In a study carried out on the removal of the acid orange dyes 7 and reactive black 5 by using zero valent iron powder in the presence of UV light and hydrogen peroxide, the highest efficiency in the UV/H_2O_2 process was observed in the acidic pH [27].

The effect of persulfate concentration

As illustrated in Figure 3, increasing the initial concentration of persulfate to 4 mM leads to increased system performance. Consequently, increasing the concentration of persulfate has little effect on increasing the efficiency of the system, and may even partially reduce it.

In the study by Piri et al., the effect of different concentrations of persulfate on the removal of Chlorophenol-4 in the AOP based on persulfate (UV/$Na_2S_2O_8$) was investigated. In order to investigate the effect of different concentrations of sodium persulfate, this substance at concentrations 8.4-84 mM was added to the Chlorophenol-4 solution.

Based on the results, at a concentration of 1.5 M of 4-chlorophenol after 30 min, the removal efficiency in the photolysis of UV alone was 8.5%, while at a concentration of 8.4 mM of sodium persulfate at optimal concentration, after 30 minutes of radiation, the removal efficiency reached 30.3%. Adding sodium persulfate at concentrations over 8.4 mM reduced the efficiency of its removal [28].

Conclusion

The results of this study show that the arsenite oxidation process by using this method is effective and this method can be used with a high (96%) efficiency; and it is easy to use this method to convert toxic arsenite into its less hazardous form that can be removed more efficiently, i.e., arsenate, and by using conventional treatment processes.

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Conflicts of Interest

There is no conflict of interest for the authors.

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