



Arsenic removal through electrocoagulation: Kinetic and statistical modeling

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ABSTRACT

Arsenic removal from water/wastewater has been the subject of top priority among researchers/industries in recent years due its notorious characteristics. A novel technique of electrocoagulation has been attempted in the present investigation to remove arsenic from aqueous solution. Experiments were carried out in a batch electrochemical reactor using aluminum and mild steel sacrificial anodes. The mechanism of electrocoagulation has been modeled using adsorption isotherm kinetics. Experimental runs were designed by response surface methodology and the influence of combined effects of operating parameters on arsenic removal has been critically examined.

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1. Introduction

Arsenic, a toxic trace element present both in ground and surface water, pose potential threat to human beings and microorganisms. In recent years, arsenic contamination of water/wastewater has become a major social concern globally. Several parts of the world, in particular countries like India and Bangladesh having relatively high arsenic concentrations in their groundwater used for drinking. Except the presence of higher level of arsenic, the ground water is otherwise quite safe for drinking. Due to its high toxic effects on human health, recently the USEPA has lowered the maximum contaminant level for arsenic in drinking water from 50 to 10 mg/L [1]. Arsenic is a carcinogen and its ingestion may deleteriously affect the gastrointestinal tract, cardiac, vascular system and central nervous system [2]. Both organic and inorganic forms of arsenic are reported in natural water. However the inorganic form dominates due to its variable oxidation states. As(V) species are found in oxidizing environment, while As(III) presents in anoxic and reducing environments. In ground water, the major species, As(V) exists as monovalent (H_2AsO_4^-) and divalent (HAsO_4^{2-}) anions while, As(III) species exists as uncharged arsenious acid (H_3AsO_3) [3,4].

Numerous treatment techniques have been adopted to remove arsenic present in the water/wastewater. This includes precipitation, adsorption, ion exchange, ultra-filtration, reverse osmosis and oxidation/filtration [5–7]. Removal of arsenic by adsorption using

iron and aluminum salts is commonly used in industries compared to other treatment methods as this method can reduce the arsenic content less than 2 mg/L. However this method generates large amount of solid sludge. The other conventional methods are inefficient in removing arsenic to less than 10 mg/L. This necessitated the industries to look for an alternative treatment method for complete removal of arsenic present in water/wastewater without generation of secondary pollutant(s).

Electrocoagulation is an emerging treatment technology, which has been applied successfully to treat various industrial wastewaters [8–10]. Electrocoagulation (EC) is an efficient method where the coagulating agent is generated in situ by electro-oxidation of sacrificial anode and the process is carried out without addition of any chemicals. The generation of coagulants (flocks) can be controlled by the applied charge, thus the amount of solid sludge generation is very much minimized resulting in a lucrative technology for water/wastewater treatment. In our previous paper [11], we attempted to remove arsenic through electrocoagulation using Fe sacrificial anode. The objective of the present study is to extend the electrocoagulation with aluminum electrode and to develop statistical/kinetic modeling for electrocoagulation process. The combined effects of operating parameters on percentage arsenic removal were critically examined and the adsorption of pollutant on electro-coagulants has been modeled using adsorption isotherm models.

2. Response surface method

Design of experiments is a powerful tool for modeling and analyzing the influence of process variables on specific variable. The

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Nomenclature

f	the unknown function of response
k	the number of independent variables
p	tested factors
x_1, \dots, x_k	the independent variables
X	coded variable
y	the response of the system
ε	the statistical error
$\beta_0, \beta_i, \beta_{ii}, \beta_{ij}$	regression coefficients

most important aspect of design of experiment lies in the selection of the control factors. The possible operating parameters can be included to identify non-significant variables at the earliest opportunity. The RSM attempts to analyze the influence of the independent variables on a specific dependent variable (response). The independent variables denoted by x_1, x_2, \dots, x_k are presumed to be continuous and can be controlled with negligible error. The individual variables (x_1, x_2, \dots, x_k) and the response (y) can be related as follows [12]:

$$y = f(x_1, x_2, x_3 \dots, x_k) + \varepsilon \quad (1)$$

RSM postulates the functional relationship between the response (y) and the independent variables. The first and second-order RSM can be give as

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \varepsilon \quad (2)$$

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^k \beta_{ij} x_i x_j + \varepsilon \quad (3)$$

The response Y can be mathematically related to the variables as

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{44} x_4^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 \quad (4)$$

In the present work, the Box–Behnken method was used to find the relationship between the response functions and variables (Table 1). A class of three-level complete-factorial designs for the estimation of parameters in a second-order model has been developed (Table 2) using MINITAB 14 (PA, USA). The analysis was focused on verifying the influence of individual parameters on percentage arsenic removal.

3. Experimental

The electrolytic cell consists of a glass beaker of 250 ml capacity with a lid. Aluminum and mild steel with a submerged area of 4.2 cm² and stainless steel of same size were used as sacrifice anodes and cathode respectively. The electrode distance between anode and cathode was maintained constant of 1.5 cm during electrolysis. A direct current was supplied by a DC-regulated

Table 1

The level and range of variables chosen for electrocoagulation.

Factor	Variables	Range of actual and coded variables		
		–1	0	+1
a	Current density (Adm ⁻²)	0.5	1	1.5
b	Concentration (ppm)	50	75	150
c	Time (min)	10	30	50
d	pH	4	7	10

Table 2

The actual design of experiments and response of arsenic removal by electrocoagulation.

CD	As conc.	Time	pH	% As-r	
				Fe	Al
0.5	50	30	7	56	48
1.5	50	30	7	82	70
0.5	150	30	7	46	40
1.5	150	30	7	65	55
1	100	10	4	42	35
1	100	50	4	59	50
1	100	10	10	33	28
1	100	50	10	72	62
1.5	100	10	7	24	20
1.5	100	10	7	50	43
0.5	100	50	7	86	73
1.5	100	50	7	85	73
1	50	30	4	40	34
1	150	30	4	37	31
1	50	30	10	75	62
1	150	30	10	45	39
0.5	100	30	4	38	33
1.5	100	30	4	63	54
0.5	100	30	10	40	34
1.5	100	30	10	62	53
1	50	10	7	18	15
1	150	10	7	18	15
1	50	50	7	84	71
1	150	50	7	50	42
1	100	30	7	61	50
1	100	30	7	61	52
1	100	30	7	62	53

power source (HIL model 3161, 0–5 A and 0–30 V). Proper agitation was provided to maintain uniform concentration inside the cell. A stock solution As(V) was prepared by dissolving appropriate quantity of sodium arsenate Na₂HAsO₄·7H₂O in de-ionized water for experimentation. All the experiments were carried out under potentiostatic conditions at room temperature (28 ± 2 °C). The pH of solution was adjusted by adding either dilute HCl or NaOH. The samples were collected at every 5 min interval and analyzed for arsenic content using atomic absorption spectroscopy (AAS, model Varian A220).

4. Result and discussions**4.1. Electrocoagulation mechanism**

Electrocoagulation is a process involving chemical and physical phenomena, which use sacrificial electrodes for generation of coagulants. The generation of metallic cations takes place at the anode, while H₂ production occurs at the cathode. The generated metal hydroxides provide active surfaces for the adsorption of the polluting species present in the effluent. In general, aluminum or mild steel is used as sacrificial anode. The electrocoagulation mechanism can be summarized as follows. When a potential is applied through an external power source, the sacrificial electrode undergoes oxidation as given below [13]:

For aluminum anode:

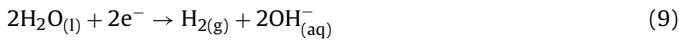


For mild steel anode:



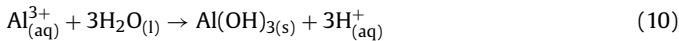
The cathodic reactions can be written as



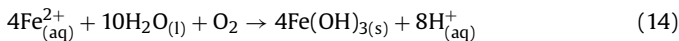
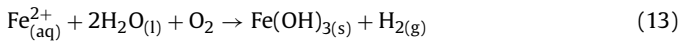


The bulk reactions for aluminum and mild steel anodes can be written as

for aluminum anode

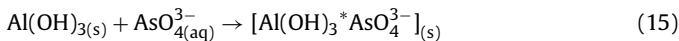


for mild steel anode

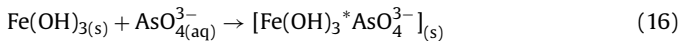


The formed aluminum/iron flocks incarcerate the arsenic present in the solution by precipitation and/or adsorption mechanism:

for aluminum anode



for mild steel anode



The electrolyte pH plays an important role in the separation of suspended particles; at low pH the separation is dominated by precipitation while adsorption dominates at high electrolyte pH [14].

Experiments were carried out according to the experimental conditions designed by RSM and the results are presented in both surface and contour plots. Fig. 1 shows the combined effect of applied current density and influent concentration on percentage arsenic removal in surface (Fig. 1a) and contour (Fig. 1b) plots. It has been observed from the literature that, in conventional chemical coagulation, the percentage of removal efficiency improved with an increase in the metal ion dosages [15]. Thus, it can be expected in electrocoagulation too that the arsenic removal depends on the amount of coagulant generated or the applied charge as the applied charge is directly proportional to the amount of coagulant generated [16,17]. It can be ascertained from Fig. 1 that the percentage of arsenic removal increased with applied charge. The increased amount of coagulants with the applied charge increased the percentage arsenic removal, as other parameters were kept constant. It can also be observed that the arsenic removal rate decreased with the increase in the influent concentration. The ratio of hydroxo cationic complexes to the initial effluent concentration decreased with an increase in the influent concentration, which eventually reduces the percentage arsenic removal. Similar observations were recorded for mild steel anode (Fig. 2).

The combined effect of electrolyte pH and the influent concentration on percentage removal for mild steel anode is given in Fig. 3. It has been well established that the electrolyte pH is an important parameter and has significant influence on the electrocoagulation efficiency. Both arsenite and arsenate show improved removal rate with influent pH [18]. Vik et al. [19] observed the effect of pH during the electrocoagulation process and reported that electrocoagulation can act as pH neutralization step. The pH increase in electrocoagulation was attributed to the formation of hydrogen gas at the cathode. Accordingly, the influent pH was considered as one of the key factor while designing the experimental runs. Experiments were carried out under controlled pH conditions by adding HCl or NaOH. It can be noticed from Fig. 3 that the percentage arsenic removal increased from acid pH to neutral with no significant improvement beyond the electrolyte pH of 7. This can be

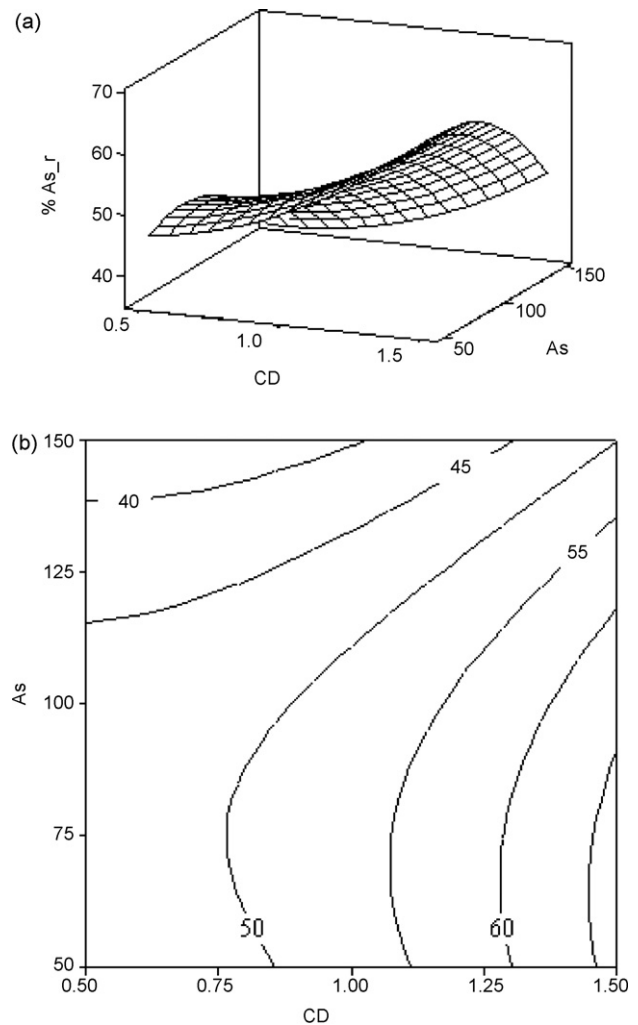


Fig. 1. Combined effects of current density and influent concentration on percentage arsenic removal, (a) response surface, (b) contour plot; electrode: aluminum.

explained that the solubility of $\text{Fe}(\text{OH})_3$ increases beyond the electrolyte pH value of 7 resulting formation of soluble $\text{Fe}(\text{OH})_4^-$ which does not contribute to the arsenic removal [20].

Fig. 4 shows the combined effect of electrolysis time and applied charge for aluminum anode. It can be noticed from the figure that the percentage arsenic removal increased with electrolysis time and applied charge. As stated earlier, the amount of flocks generated increased with an increase in the current density or electrolysis time and in turn the percentage arsenic removal. The mathematical relationship of percentage arsenic removal with process parameters such as applied current density (a); influence concentration (b); electrolysis time (c) and electrolyte pH (d) in coded factors for both aluminum and mild steel anodes can be given as

for aluminum anode

$$\begin{aligned} \% \text{As-r} = & 51.6667 + 8.33a - 6.5b + 17.9167c + 3.4167d + 3.667a^2 \\ & - 5.5833b^2 - 4.9583c^2 - 6.4583d^2 - 1.75ab - 5.75ac \\ & - 0.5ad - 7.25bc - 5bd + 4.75cd \end{aligned} \quad (17)$$

for mild steel anode

$$\begin{aligned} \% \text{As-r} = & 60.6 + 9.775a - 7.7667b + 21.05c + 4.0417d + 4.0458a^2 \\ & - 6.2667b^2 - 5.6167c^2 - 7.3792d^2 - 1.95ab - 6.725ac \\ & - 0.8ad - 78.45bc - 6.55bd + 5.325cd \end{aligned} \quad (18)$$

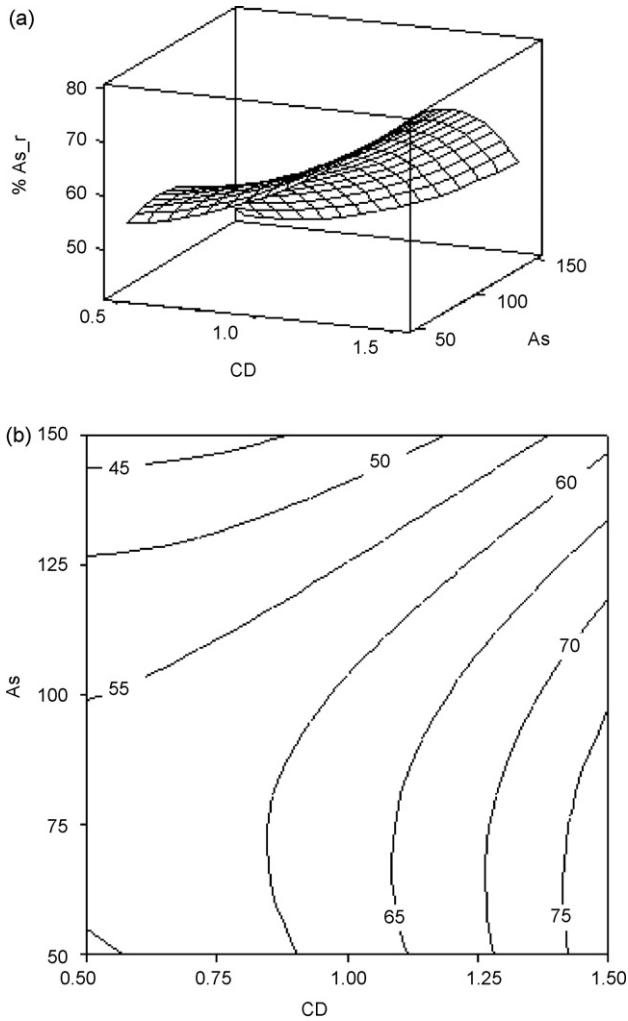


Fig. 2. Combined effects of current density and influent on percentage arsenic removal, (a) response surface, (b) contour plot; electrode: mild steel.

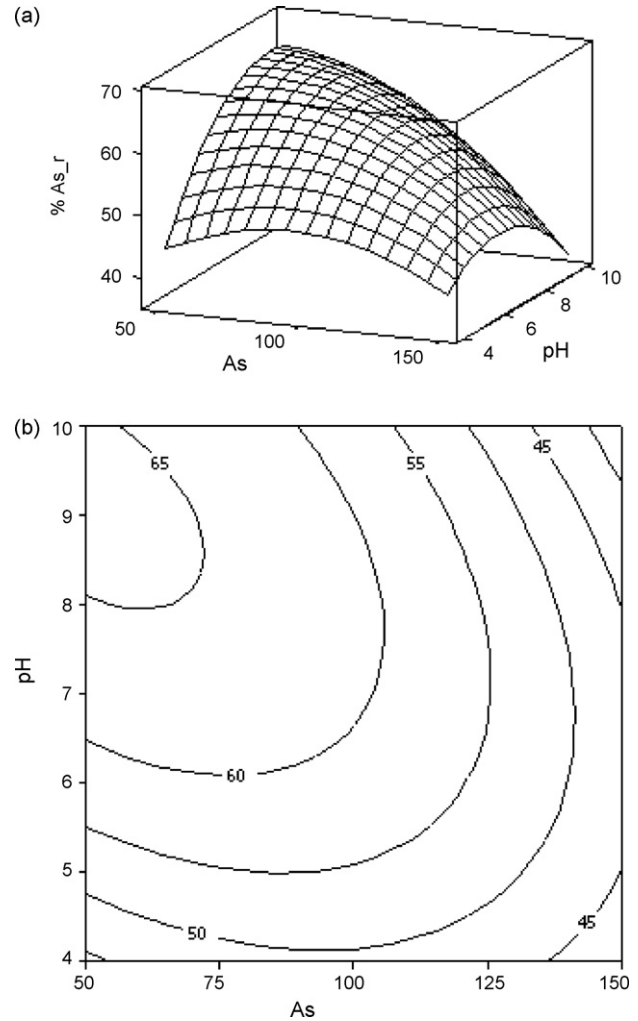


Fig. 3. Combined effects of influence concentration and electrolyte pH on percentage arsenic removal, (a) response surface, (b) contour plot; electrode: mild steel.

The ‘p’ and ‘t’ tests were used to analyze the significance of regression coefficient (Tables 3 and 4). It can be noticed from Tables 3 and 4 and the Eqs. (17) and (18) that the coefficients for the linear effect of influent concentration, applied charge and electrolyte pH are significant compared to their coefficients in the quadratic term. Larger magnitude of *t*-value and smaller magnitude of *p*-value show the significance of corresponding coefficient terms. Tables 5 and 6

show the result of analysis of variance (ANOVA), carried out to determine the significant effects of process variables on percentage arsenic removal for aluminum and mild steel anodes respectively. It can be noticed from tables that the *F*-statistics values for the regressions are higher. The large *F*-values indicate that most of the variation in the response can be explained by the regression model equation. The associated *p*-value is used to estimate whether the

Table 3
Estimated regression coefficient and corresponding *t* and *p* values for arsenic removal, electrode: aluminum.

Term	Coef	SE Coef	<i>t</i>	<i>p</i>
Cons	51.6667	4.686	11.026	0.000
<i>a</i>	8.3333	2.343	3.557	0.004
<i>b</i>	-6.5	2.343	-2.774	0.017
<i>c</i>	17.9167	2.343	7.647	0.000
<i>d</i>	3.4167	2.343	1.458	0.170
<i>a</i> × <i>a</i>	3.6667	3.514	1.043	0.317
<i>b</i> × <i>b</i>	-5.5833	3.514	-1.589	0.138
<i>c</i> × <i>c</i>	-4.9583	3.514	-1.411	0.184
<i>d</i> × <i>d</i>	-6.4583	3.514	-1.838	0.091
<i>a</i> × <i>b</i>	-1.75	4.058	-0.431	0.674
<i>a</i> × <i>c</i>	-5.75	4.058	-1.417	0.182
<i>a</i> × <i>d</i>	-0.5	4.058	-0.123	0.904
<i>b</i> × <i>c</i>	-7.25	4.058	-1.787	0.099
<i>b</i> × <i>d</i>	-5	4.058	-1.232	0.241
<i>c</i> × <i>d</i>	4.75	4.058	1.171	0.265

Table 4
Estimated regression coefficient and corresponding *t* and *p* values for arsenic removal, electrode: mild steel.

Term	Coef	SE Coef	<i>t</i>	<i>p</i>
Constant	60.6	5.553	10.913	0
<i>a</i>	9.775	2.777	3.521	0.004
<i>b</i>	-7.7667	2.777	-2.797	0.016
<i>c</i>	21.05	2.777	7.581	0
<i>d</i>	4.0417	2.777	1.456	0.171
<i>a</i> × <i>a</i>	4.0458	4.165	0.971	0.351
<i>b</i> × <i>b</i>	-6.2667	4.165	-1.505	0.158
<i>c</i> × <i>c</i>	-5.6167	4.165	-1.349	0.202
<i>d</i> × <i>d</i>	-7.3792	4.165	-1.772	0.102
<i>a</i> × <i>b</i>	-1.95	4.809	-0.405	0.692
<i>a</i> × <i>c</i>	-6.725	4.809	-1.398	0.187
<i>a</i> × <i>d</i>	-0.8	4.809	-0.166	0.871
<i>b</i> × <i>c</i>	-8.45	4.809	-1.757	0.104
<i>b</i> × <i>d</i>	-6.55	4.809	-1.362	0.198
<i>c</i> × <i>d</i>	5.325	4.809	1.107	0.29

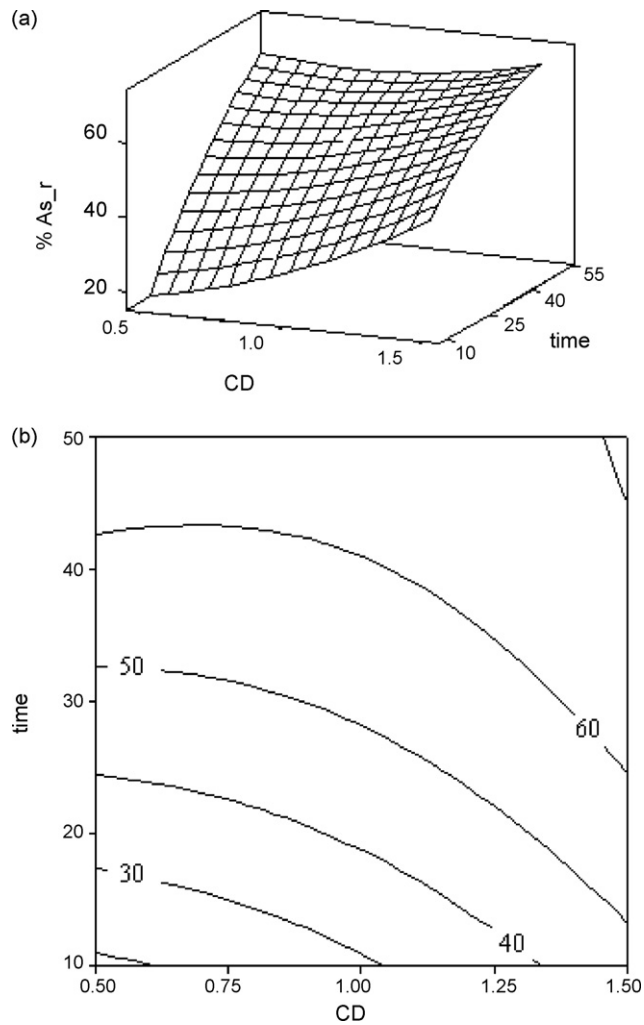


Fig. 4. Combined effects of current density and electrolysis time on percentage arsenic removal, (a) response surface, (b) contour plot; electrode: aluminum.

Table 5
ANOVA results for the percentage arsenic removal, electrode: aluminum.

Source	DF	Seq SS	Adj-SS	Adj-MS	F	p
Regression	14	6528.77	6528.77	466.34	7.08	0.001
Linear	4	5332.5	5332.5	1333.13	20.24	0
Square	4	650.27	650.27	162.57	2.47	0.101
Interaction	6	546	546	91	1.38	0.298
Residual error	12	790.42	790.42	65.87		
Lack-of-fit	10	785.75	785.75	78.58		
Pure error	2	4.67	4.67	2.33		
Total	26	7319.19				

$R^2 = 0.89$, $R^2_{adj} = 0.766$.

Table 6
ANOVA results for the percentage arsenic removal, electrode: mild steel.

Source	DF	Seq SS	Adj-SS	Adj-MS	F	p
Regression	14	8977.4	8977.41	641.24	6.93	0.001
Linear	4	7383.7	7383.71	1845.93	19.95	0
Square	4	824.4	824.38	206.1	2.23	0.127
Interaction	6	769.3	769.32	128.22	1.39	0.296
Residual error	12	1110.2	1110.15	92.51		
Lack-of-fit	10	1110.2	1110.15	111.02		
Pure error	2	0	0	0		
Total	26	10087.6				

$R^2 = 0.89$, $R^2_{adj} = 0.762$.

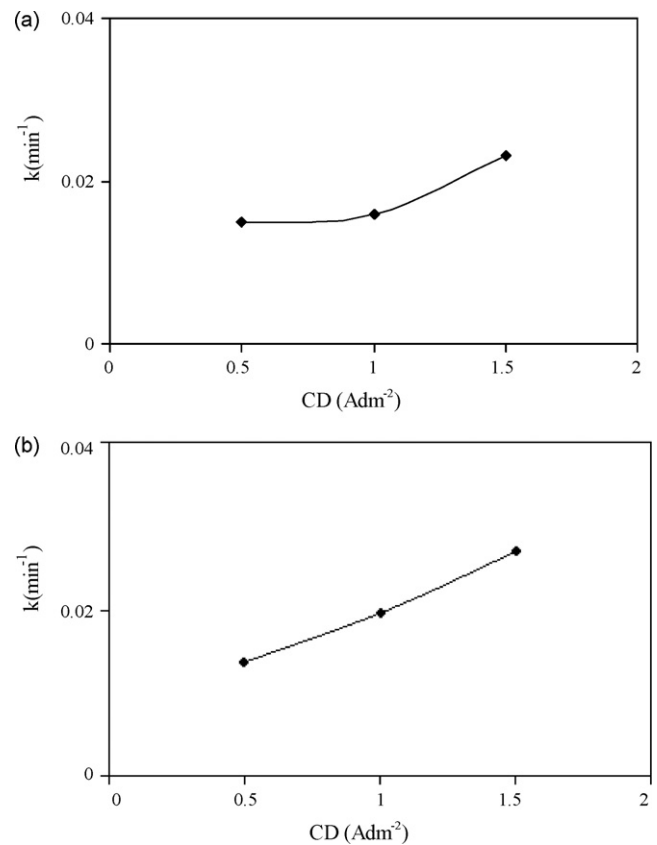


Fig. 5. Variation of reaction rate constant with applied current density. Influent concentration: 100 ppm; pH 7; supporting electrolyte concentration: 100 ppm electrode: (a) aluminum; (b) mild steel.

F-statistics are large enough to indicate statistical significance. The lower *p*-value indicates that the model is considered to be statistically significant. The model adequacies were checked by R^2 and $adj-R^2$. A higher value of R^2 (0.89) shows that the model can explain the response successfully. The model adequacy has also been verified with the $adj-R^2$ value. The ANOVA indicates that the second-order polynomial model is significant and adequate to represent the actual relationship between the response of arsenic removal efficiency and the variables.

In the electrocoagulation process, the rate of arsenic removal is proportional to the influence concentration and the amount of corresponding hydroxides formed, i.e.,

$$\frac{-dC}{dt} = kCC_a \quad (19)$$

where, C_a refers adsorbent concentration in the system. Since the generation of ferric and aluminum hydroxide can be assumed constant for a given current density, the above equation can be simplified to pseudo first-order kinetics as

$$\frac{-dC}{dt} = kC \quad (20)$$

The integration of Eq. (20) yields

$$\log \frac{C}{C_i} = -kt \quad (21)$$

The reaction rate constant '*k*', can be estimated from the plot $\log[C/C_i]$ versus electrolysis time. Fig. 5 shows the influence of applied current density on reaction rate constant. It can be noticed that the reaction rate constant increases with an increase in the applied current density. It is obvious that the rate of coagulant generation increases with applied current density and in turn the

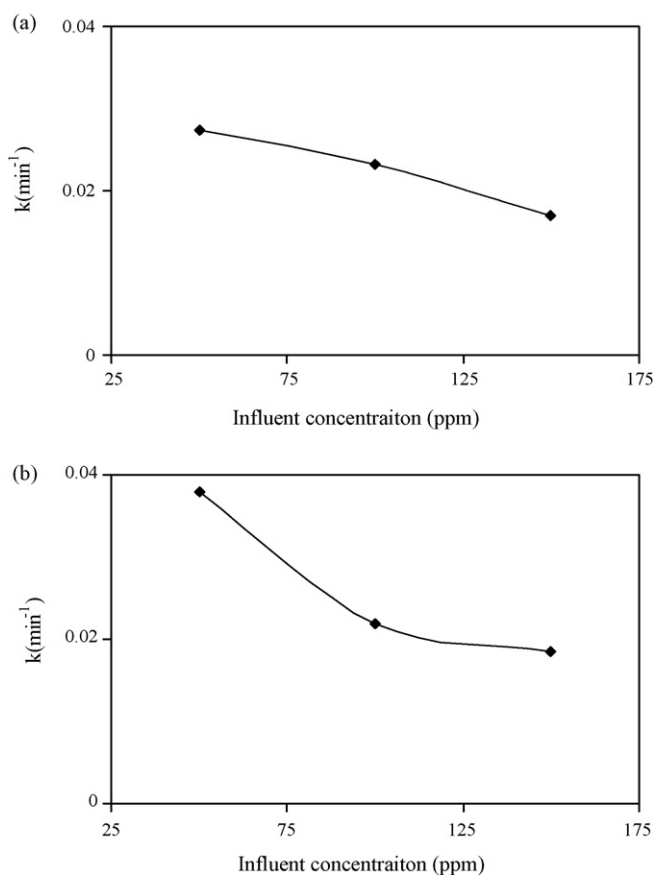


Fig. 6. Influence of arsenic initial concentration on reaction rate constant. Current density: 1 Adm^{-2} ; supporting electrolyte concentration: 100 ppm, pH 7; electrode: (a) aluminum; (b) mild steel.

reaction rate constant. Fig. 6 shows the influence of initial effluent concentration on reaction rate constant. It can be ascertained that the rate constant decreases with an increase in the initial arsenic concentration. This could be due to the decrease in ratio of hydroxo cationic complexes to the initial effluent concentration, which eventually decreases the rate of arsenic removal and in turn the reaction rate constant.

4.2. Adsorption isotherm

As stated earlier, in electrocoagulation, two distinct process takes place: generation of flocks (electro-dissolution) and adsorption of pollutant on generated flocks (physical adsorption). The removal of pollutant (adsorption on flocks) is very similar to conventional adsorption except the generation of flocks [21]. The electrode consumption and amount of flocks generated can be estimated according to Faraday's Law [22]. Since the amount of coagulant can be estimated for a given time, the pollutant removal can be modeled by adsorption phenomenon. It is attempted to test the various adsorption isotherm models for COD removal.

4.2.1. Langmuir isotherm

The Langmuir isotherm assumes monolayer deposition of adsorbate on homogenous adsorbent surface (coagulant). The mathematical expression of Langmuir isotherm can be given as

$$q_e = \frac{K_L C_e}{1 + a_L C_e} \quad (22)$$

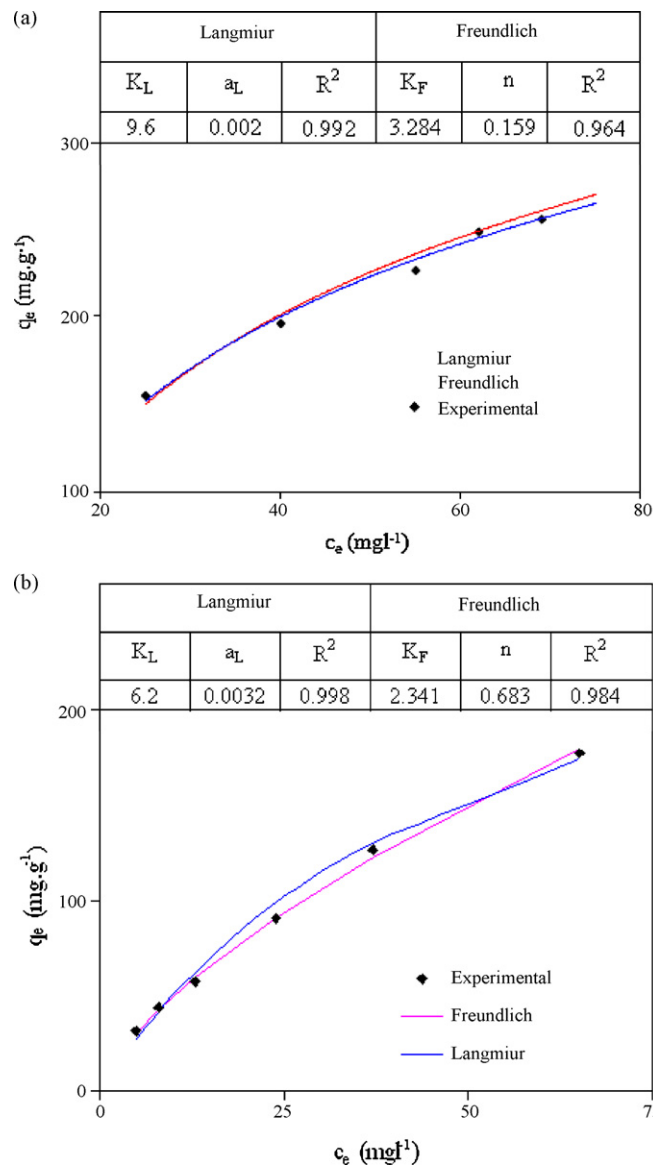


Fig. 7. Adsorption of isotherm of arsenic by charged hydroxo cationic complexes, electrode (a) aluminum; (b) mild steel.

The linearization of the above equation results

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{a_L}{K_L} C_e \quad (23)$$

The binding constant (K_L) and the sorbent capacity (a_L) can be estimated by plotting C_e/q_e against C_e . The model simulations along with experimental observations for both aluminum and mild steel along with the experimental values are shown in Fig. 7.

4.2.2. Freundlich isotherm

The Freundlich isotherm is an empirical model relates the adsorption intensity of the sorbent towards adsorbent. The isotherm is adopted to describe reversible adsorption and not restricted to monolayer formation. The mathematical expression of the Freundlich model can be written as

$$q_e = K_F C_e^{b_F} \quad (23)$$

where K_F and b_F are the constants which give adsorption capacity and adsorption intensity, respectively. A linear form of the Fre-

undlich model can be written as follows:

$$\ln q_e = \ln K_F + b_F \ln C_e \quad (24)$$

Plot of $\ln q_e$ versus $\ln C_e$ gives a straight line with slope K_F and intercept b_F . The model simulation and the constants K_F and b_F values for both aluminum and mild steel anodes along with experimental values are given in Fig. 7. It can be observed from the correlation coefficient (R^2) that the Langmuir isotherm model fits well than the Freundlich isotherm for both aluminum and mild steel anodes. From the above analysis, it can be concluded that the adsorption of arsenic on electrochemically generated flocks follows monolayer adsorption.

5. Conclusion

Experiments were carried to remove arsenic by electrocoagulation covering wide range in operating conditions. The percentage of arsenic removal shows significant influence with applied charge and solution pH. The electrocoagulation has been modeled using adsorption isotherm models and observed Langmuir isotherm model match satisfactorily with the experimental observations. The experimental data were analyzed using response surface methodology. Three-level four factorial Box–Behnken experimental design was applied in the present investigation. Regression equations have been developed for percentage arsenic removal and solved using statistical software tool MINITAB14.

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