Optimization of free chlorine, electric and current efficiency in an electrochemical reactor for water disinfection purposes by RSM

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Abstract

This study surveys the possibility to optimally produce active chlorine from synthetic saline solutions using electrolysis by Response Surface Methodology (RSM). Various operating parameters, such as sodium chloride concentration, electrical potential and electrolysis time were evaluated. Central composite design (CCD) was applied to determine the optimal experimental factors for chlorine production. The experimental design, statistical analysis of the data and optimization were performed using R 3.5.3 software. The results showed that the optimum value of electrical efficiency (42 mg Cl₂/kj) was obtained at the electrical voltage of 15.73 V during 15.63 min in the presence of 63.42 g/l of sodium chloride. The optimum point for current efficiency was 38.40%, which was obtained at the electrical voltage of 10.76 V during 6.70 min in the presence of 34.65 g/l of sodium chloride. Moreover, generated active chlorine was optimized based on energy consumption, which was 77 mg/l for the energy consumption of 0.2 kWh/l at a current density of 2000 mA/cm². The electrochemical production of the chlorine gas from saline or brine water can be extensively used for water disinfection.

Keywords Electrical efficiency · Current efficiency · Response Surface Methodology (RSM)

Introduction

Disinfection is one of the main processes carried out in water treatment facilities. Various methods have been proposed for this purpose and many of these methods were used in

Highlights

- Without lowering pH to below 2 and using low-cost and available graphite electrodes, active chlorine was formed in significant amounts
- The electrochemical production of free chlorine follows a first order model
- The results of this study led to the construction of two portable devices producing chlorine gas for water disinfection

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numerous treatment plants around the world [1]. Chlorination has been the most commonly adopted disinfection process for the treatment of drinking water. Active chlorine can be produced during the electrolysis of saline or sea-water [2]. The effect of this method on the disinfection of drinking water, swimming pool water and water cooling systems has been considerably proved [3]. Due to the lack of suitable methods for the removal of saline water produced during membrane processes, the use of the methods were limited [2, 4]. The alternative approach that can be developed for reuse of such brine water is the production of chlorine through its electrolysis [2]. In this method, the electric current is applied into the electrolyte through electrodes. In the process, the type of cathode and anode electrode should be of inert material such as graphite or platinum [3]. Chlorine evolution occurred on the surface of anode [3]. This method is low cost and environmentally friendly [5]. In addition, this method, which is a point of use method, reduces the risks and problems associated with the storage and transport of chlorine gas cylinders. This method has been successfully implemented for water disinfection in developing countries in emergencies [6]. In the electrochemical development of chlorine generation, attention to electrolysis parameters such as energy consumption, current density, current efficiency and electric efficiency is essential. Current efficiency is calculated from the percentage

ratio of the total chlorine produced to the theoretical chlorine production based on Faraday's laws of electrolysis. Electric efficiency, which represents energy efficiency of the system, is calculated by dividing the total chlorine produced by the electric energy consumed. Several studies have been conducted in this regard in different countries. Morita et al. surveyed the potential of disinfection of electrolyzed sodium chloride solutions at low concentrations [7]. Zaviska et al. [2] carried out a study to optimize the production of active chlorine from synthetic seawater by electrolysis. Spasojević et al. [8] carried out a study on current efficiency modeling in an electrochemical hypochlorite reactor. Hsu et al. [9] surveyed the effects of electrolysis time and electric potential on the chlorine generation of electrolyzed deep ocean water. Their results showed that current efficiency is significantly affected by electrolysis time and electric potential, and also electric efficiency or energy efficiency of the system was significantly affected by electrolysis time and electric potential. The aim of the present study is to optimally evaluate the current and electric efficiency of an electrolytic cell for active chlorine production from a synthetic saline effluent.

Materials and methods

Preparation of electrolyte solutions

The electrolyte solutions at the concentrations of 5, 15, 25, 35, 45 g/L were prepared by sodium chloride salt with a purity of 99.5% (Merck Company). The selection of these concentrations was based on sodium chloride amount of seawater.

Experimental unit

The experimental unit includes five main parts. One component was DC power supply with an adjustable electrical voltage of 0 to 30 V, and a current intensity of 0 to 5 A (Dazheng Co., China). The second component was an electrolytic cell with a volume of 200 ml that was contained with the sodium chloride solutions. The electrolytic cell contained a cathode electrode and an anode electrode. The type of the electrodes was graphite with a height of 4 cm and, diameter of 0.5 cm. The distance between the electrodes was also 1.5 cm. The third part was a vacuum pump (KnF Company) with a flow rate of 0.85 L/min. Besides, at the inlet of the pump, a cellulosic filter was used to capture carbon particles separated from the anode. The fourth component was an injection cell with a volume of 50 ml that was filled with deionized water. Finally, the fifth component was a cylindrical granular activated carbon column with a mean size of 4 mm. The column was placed for the adsorption of the excess chlorine gas in the outlet. The experimental unit is depicted in Fig. 1.

Experimental design

To determine the number of experiments and statistical analysis of data, experimental design (ED) and Response Surface Methodology (RSM) were used. In the design section, the factorial design (FD) was not employed and the type and range of variables were determined by similar studies. Experimental design of the electro-chlorination process was carried out using central composite design (CCD) methodology. The CCD is widely used for the response surface methodology (RSM). RSM is a collection of mathematical and statistical methods for modeling and analysis of a process in which a response of interest can be influenced by several variables. Indeed, it is used to determine the optimum operating conditions or to determine a region for the factors in which certain speciation are met. The CCD was used to describe the process in the experimental domain and also for the optimization of this process to have the best gas chlorine generation at the lowest energy consumption. This design is formed by uniformly distributed points within the space of the coded variable (X_i). One of the advantages of CCD is the possibility to explore the whole of the experimental region and the usefulness of interpolating the response. For the axial runs matrix, α has been chosen in order to have iso-variance property by using rotation. The experimental response associated to a CCD matrix is represented by a quadratic polynomial model:

$$Y = b_0 + \sum_{i=1}^k b_i \cdot X_i + \sum_{i=1}^K b_{ii} \cdot X_i^2 + \sum_j \sum_{i=2}^k b_{ij} \cdot X_i X_j \quad (1)$$

Where

- Y Experimental response
- b_0 Average of the experimental response
- b_i Estimation of the principal effect of the factor j for the response Y
- b_{ii} Estimation of the second effect of the factor i for the response Y
- b_{ij} Estimation of the interaction effect between factors i and j for the response Y

The coefficients of this model are calculated in the experimental region using the least square method [10]:

$$B = (X^T X)^{-1} X^T Y \quad (2)$$

Where, B , vector of estimates of the coefficients; X , the model matrix; Y , the vector of the experiment results. A three-factorial and a two-level central composite design, with six replicate at the center point led to a total number of twenty experiments employed for response surface modeling. The independent process variables used in this study were: the NaCl (electrolyte) concentration (X_1), the applied electrical

Fig. 1 Schematic diagram of the experimental unit for chlorine gas production: (1) power supply; (2) electrolytic cell; (3) graphite electrodes; (4) filter; (5) vacuum pump; (6) chlorination cell; (7) activated carbon column

potential (X_2), and electrolysis time (X_3). The experimental values of U_i can be calculated from the coded variables X_i using the following equation:

$$X_i = \frac{U_i U_{i,0}}{\Delta U_i} \quad (3)$$

Where $U_{i,0} = (U_{i,\max} + U_{i,\min})/2$, represents the value of U_i at the center of the experimental field; $\Delta U_i = (U_{i,\max} - U_{i,\min})/2$, represents the step of the variation, with $U_{i,\max}$ and $U_{i,\min}$ which are the maximum and minimum values of the effective variable U_i , respectively. Gas chlorine production (Y_1) and energy cost (Y_2) was considered as dependents factors (response). The values of process variables and their variation limits were selected based on the preliminary experiments. Experimental data were analyzed using R 3.6.2 program software including ANOVA in order to obtain the interactions between the process variables.

Experiments

In each experiment, the electrolytic cell was filled with the 100 ml intended solution, and then the electrolysis process was performed in the cell. Sodium chloride solutions were with different initial concentrations of 5, 15, 25, 35, 45 g/L. The electrical voltages (15, 12, 9, 6, 3 V) were applied by the DC power supply. During the process, the chloride ions present in the electrolyte solutions in the vicinity of the anode was reduced to chlorine gas. The collected chlorine gas in the headspace of the electrolytic cell was removed by a vacuum pump with a flow rate of 0.85 L/min and then injected into the chlorine injection cell contained with 30 ml of deionized water. Sodium hypochlorite was also formed in the electrolytic cell. Furthermore, the experiments were carried out at neutral pH. The supplementary tests were shown that chlorine gas can

be produced at neutral pH. To prevent the corrosion of the anode and as a result the entrance of the carbon particles into the chlorination cell, a cellulosic filter was placed on the output of the chlorine gas. After the end of the electrolysis process, 10 ml of the chlorinated water (with and without dilution) was considered to measure active chlorine. Active chlorine was measured by the colorimetric method and by DR5000 spectrophotometer (Hach Company). The free chlorine content of the samples was read as mg/l at the wavelength of 530 nm. All experiments were performed under standard conditions and ambient temperature (25 °C).

Results

During the preliminary experiments, the experimental domain has been determined. The types of variables and their levels based on the CCD design have been shown in Table 1. Considering the minimum and maximum values of the initial concentrations of sodium chloride (15 and 35 g/L), the electrical potential (6 and 12 v) and the electrolysis times (5 and 10 min), along with the central and axial points, a total of 20 tests were designed in two blocks (Table 2). As shown in Table 2, the maximum free chlorine formation in the chlorination cell was 96 mg/L, which was

Table 1 Experimental range and levels of independent factors

Factor	Unit	Rang and Levels				
		$-\alpha$	-1	0	1	$+\alpha$
NaCl	g/L	5	15	25	35	45
Voltage	V	3	6	9	12	15
Time	min	2.5	5	7.5	10	12.5

obtained at a concentration of sodium chloride 45 g/L, electrical potential of 9 V (current intensity of 1.5 A) electrolysis time of 12.5 min. The experiment region investigated for gas chlorine production, and the coded values are shown in Table 1. The order and number of experiments, the observed and predicted free chlorine concentration, as well as the error value (difference between observed and predicted values) have been also depicted in Table 2. Furthermore, regression coefficients are also given in Table 3. The ANOVA results of the response surface quadratic model of the free chlorine production are also shown in Table 4. In Fig. 2, the response surface for the amount of free chlorine produced as a function of electrical potential and the initial concentration of sodium chloride is given. In Fig. 3, the response surface of the electrical efficiency is given as a function of the electrolysis time and the initial concentration of sodium chloride. And finally, in Fig. 4, the response surface of current efficiency is shown versus electrolysis time and the initial concentration of sodium chloride. The optimization of active chlorine as a function of energy consumption is also shown in Fig. 5 that was 77 mg/l for the energy consumption of 0.2 kWh/l. In addition the figures related to residuals have been demonstrated in Figs. 6, 7, 8 and 9.

Discussion

In the electrolysis of electrolyte containing chloride ions, chlorine is generated at the anode (Eq. (4)) and hydrogen gas is produced at the cathode [11]. Due to the dissociation of the produced chlorine in water, hypochlorite and chloride ions are produced as illustrated by Eq. (5) [12]. The chlorine gas (Cl_2) is transferred from the electrolytic cell into a storage tank by using a vacuum pump as described in Fig. 1.

These reactions are as follows [13]:

The formation of chlorine gas in the electrolyte cell is due to the oxidation of chloride ions around the anode. Injection of this gas into the deionized water cell resulted in the formation of residual free chlorine. Also in the electrolytic cell, free chlorine reacted in the form of hypochlorite ion with sodium ions is produced by the electrolysis of sodium chloride and, as a result sodium hypochlorite is formed.

The results of this study demonstrate that the amount of free chlorine formation increased with an increase in each of

Table 2 Experimental design and the responses variables of free chlorine and energy consumption

Experimental Number	NaCl concentration (g/L)	Experiment plan Electrical potential (v)	Electrolysis time (min)	Observed free chlorine (mg/ L)	Predicted free chlorine (mg/ L)	Error (€)	Average current intensity (A)	Current density (mA/ cm ²)	Energy consumption (kwh/l)
1	35	6	10	22.4	50.99	-28.59	0.7	350	0.024
2	25	9	7.5	55	40.77	14.22	1.3	650	0.050
3	15	6	5	2.5	-9.14	11.64	0.4	200	0.007
4	35	12	10	63.5	90.69	-27.19	2.3	1150	0.157
5	15	6	10	4	18.52	-14.52	0.5	250	0.017
6	35	12	5	59	63.01	-4.01	2.3	1150	0.078
7	35	6	5	13	23.31	-10.31	0.9	450	0.015
8	15	12	10	60	58.22	1.77	1.7	850	0.116
9	25	9	7.5	41.5	40.77	0.72	1.3	650	0.050
10	25	9	7.5	41.5	40.77	0.72	1.3	650	0.050
11	15	12	5	23.5	30.55	-7.05	1.1	550	0.038
12	25	9	7.5	53.5	40.77	12.72	1.3	650	0.050
13	25	3	7.5	0.75	1.07	-0.32	0.1	50	0.001
14	45	9	7.5	96	73.23	-22.76	1.5	750	0.058
15	25	15	7.5	77.5	80.47	-2.97	2.4	1200	0.154
16	25	9	2.5	10.25	13.09	-2.84	1.3	650	0.016
17	25	9	12.5	95	68.44	26.55	1.5	750	0.096
18	25	9	7.5	51.5	40.77	10.72	1.4	700	0.054
19	25	9	7.5	45	40.77	4.22	1.4	700	0.054
20	5	9	7.5	0.09	8.30	-8.21	0.4	200	0.015

Table 3 Regression coefficients of proposed model

	Regression coefficient	Standard error	T-value	Pr > F
Intercept	45.09795	7.27508	6.1990	0.0001016
NaCl (X_1)	16.23250	4.55990	3.5598	0.0057827
Electrical potential (X_2)	19.85000	4.55990	4.3532	0.0014364
Time (X_3)	13.83750	4.55990	3.0346	0.0125779
NaCl: Electrical potential	1.26250	6.44867	0.1958	0.8487059
NaCl: Time	−3.01250	6.44867	−0.4672	0.6504050
Voltage: Time	3.76250	6.44867	0.5835	0.5725114
x_1^2	−1.43977	3.63754	−0.3958	0.7005588
x_2^2	−3.66977	3.63754	−1.0089	0.3368289
x_3^2	−0.29477	3.63754	−0.0810	0.9370121

Multiple R-squared: 0.7776, Adjusted R-squared: 0.7359

the independent variables (Table 3). The most effective variables were electrical voltage, initial concentration of sodium chloride and electrolysis time, respectively. By increasing the applied electrical voltage, the amount of energy given to each coulomb increases the charge, so that each electron exchanged between the electrodes will have more energy. Subsequently, chlorine molecules are formed at a higher rate. Moreover, when more chloride ions are present in the solution, the conductivity of the solution increased and, as a result, more electrons will flow. In addition, the presence of more chloride ions forms more chlorine molecules. Electrolysis time is another important parameter that can increase the oxidation of chloride ions and can form chlorine gas in the electrolyte solution headspace. Key et al. [6] reported that maximum free chlorine concentration in the electrolytic cell was 5600 mg/L, which occurred at a concentration of sodium chloride 25 g/L and electrolysis time of 10 min. They concluded that the maximum free chlorine concentration was formed in the range of 5 to 15 min [6]. Engracia et al. [14] reported free chlorine released from electrolysis in synthetic saline water (200 mg/L), which was achieved at the time of 45 min. Saha et al. [15] designed a new electro-chlorinator using graphite electrodes for water disinfection. In their study, the optimal amount of free chlorine was reported to be 2.5 mg/L, which was obtained at the electrolysis time of 30 min.

In this study, generated active chlorine was optimized based on energy consumption, which was 77 mg/l for the

energy consumption of 0.2 kWh/l (Fig. 5). Zaviska et al. optimized the electrochemical production of active chlorine from the synthetic saline effluent by CCD experimental design. The optimal amount of active chlorine in their study was 31 mg/L, which was reported at the sodium chloride concentration of 47 g/L, electrolysis time of 27 min and a current intensity of 1.06 A. They concluded that the variables of the current intensity and electrolysis time were two effective factors in the production of active chlorine [2]. Since electrical potential is the main driving force of the system, the higher the voltage, the higher the amount of electric current that passes through the system and the higher the current density will be. Current density is significantly ($p < 0.001$) affected by electric potential. Increasing electric potential and increased current density enhanced the chlorine generation rate. According to Fig. 2 the

Fig. 2 Response surface for the amount of free chlorine produced as a function of electrical voltage and initial concentration of sodium chloride

Table 4 ANOVA results for the response surface first order model for free chlorine production

Source	d.f. ^a	Sum of square	Mean square	F-value	Pr > F
Model	3	13,583.9	4528.0	18.6465	1.79×10^{-5}
Residual	16	3885.3	242.8		
Lack of fit	1	3700.3	336.4	9.0917	0.01228
Pure error	5	185.0	37.0

Fig. 3 Response surface for the electrical efficiency as a function of electrolysis time and electrolyte concentration

response surfaces were increasing, which shows the value of the response variable increases with an increase in each of the independent variables. Data analysis and interactions between dependent and non-dependent variables were performed through the multi-way analysis of variance (ANOVA). The p value in Table 3 shows that the effect of independent variables and the intercepts on chlorine production was statistically significant at 95% confidence interval ($p < 0.05$). Furthermore, according to this table, there was no significant interaction between the variables, and also the free chlorine

Fig. 4 Response surface for the current efficiency as a function of electrolysis time and electrolyte concentration

Fig. 5 The optimization of active chlorine as a function of energy consumption

production was the function of the first-order polynomial equation. The quality of the proposed model was evaluated by the coefficient R^2 and the lack of fit (LOF). R^2 equal to 0.77 provides a fairly good correlation between predicted and observed values (Table 3). The lack of fit that describes the fitting of the model to the data is presented in the analysis of variance table of the model. The value of this parameter was 0.01 that is statistically insignificant (Table 4). The lack of fit in a very good model should be greater than 0.05. The adequacy of the model was also evaluated by the residual values against the predicted values. The random dispersion of the residuals around the straight line shows that there are no increasing or decreasing trends. This suggests that the proposed model was relatively good (Figs. 6, 7, 8 and 9). Current efficiency was calculated from the percentage ratio of the total chlorine produced to the theoretical chlorine production based on Faraday's laws of electrolysis [9]. Electric efficiency,

Fig. 6 The residual values against fitted values

Fig. 7 The standardized residuals against theoretical quantiles

which represents the energy efficiency of the system, was calculated by dividing the total chlorine produced by the electric energy consumed. The optimum point of electrical efficiency (42 mgCl₂/kj) was obtained at 63.42 g/l electrolyte concentration, electrical potential of 15.73 V and electrolysis time of 15.63 min (Fig. 3). The optimum condition of current efficiency (38.5%) was obtained at an electrolyte concentration of 34.65 g/l, electrical potential of 10.76 V and electrolysis time of 6.70 min (Fig. 4). Spasojević et al. conducted modeling of current efficiency in an electrochemical hypochlorite reactor. In their study, theoretical dependences of the anodic current efficiency on the overall anodic current density and hypochlorite concentration were established [8].

Fig. 8 The The squared standardized residuals against fitted values

Fig. 9 The standardized residuals against leverage values

Current efficiency was directly correlated with electrolyte concentration and inversely correlated with electrical potential and electrolysis time variables. The highest influence was related to electrolysis time and the least effect was related to electrical potential. As shown in Fig. 4, current efficiency is significantly affected by electrolysis time and electrolyte concentration ($p < 0.001$). The longer the electrolysis time and the higher the electric potential, the lower the current efficiency will be. Also, the higher the electrolyte concentration, the higher the current efficiency will be. Hsu et al. surveyed the effects of electrolysis time and electric potential on the chlorine generation of electrolyzed deep ocean water. Their results showed that current efficiency is significantly affected by electrolysis time and electric potential, and also electric efficiency or energy efficiency of the system was significantly affected by electrolysis time and electric potential. In their study, current efficiency and electric efficiency were %82 and 46.5 mgCl₂/kj, respectively. Electric efficiency of the system was also significantly ($p < 0.001$) affected by electrolysis time and electrolyte concentration in a similar manner (Fig. 3). Moreover, the optimization of active chlorine as a function of energy consumption is shown in Fig. 5.

Conclusion In many regions of the world there is no access to disinfectant compounds for water treatment and consequently this study was designed to overcome this problem. In the study, without lowering pH to below 2 and using low-cost and available graphite electrodes, active chlorine was formed in significant amounts. Based on the results of the study, the active chlorine produced was optimized based on the energy consumption that was 77 mg/l for energy consumption of 0.2 kWh/l. Besides, the electrochemical production of free chlorine was based on first order model. Prolonged electrolysis reduced active chlorine concentration in the electrolyte and

reduced current efficiency as well as electric efficiency, especially in high electric potential situations. Therefore, the optimal choice of electrolysis time depends on chloride concentration in saline water and the electric potential used for electrolysis. According to the results, it is concluded that the electrolysis process can be used for chlorine gas generation from saline or seawater for the purpose of water disinfection.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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