

*Full Paper*

## **Effect of Electrolytic Cell Operating Parameters on Sodium Hypochlorite Formation and Energy Consumption**

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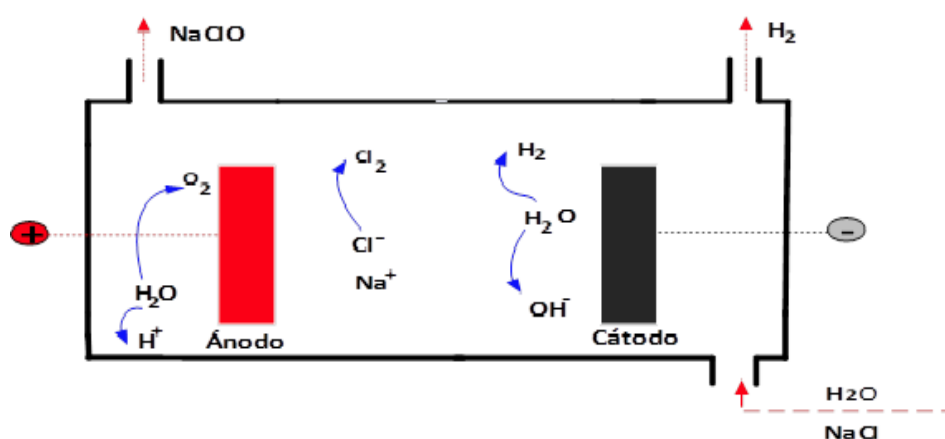
**Abstract-** Sodium hypochlorite (NaClO) is used on a large scale for water disinfection, fabric bleaching, and odor removal. It is preferred due to the environmental risk of liquid chlorine associated with storage and transport. The objective of the research was the electrochemical generation of sodium hypochlorite from an aqueous solution of sodium chloride. Different operating conditions affecting the sodium hypochlorite generation process were studied, including electrical potential (6 and 10 V), sodium chloride concentration (80 and 120 g/L), and electrolysis time (30 and 60 minutes). To analyze the effect of the factors with respect to the response variable such as sodium hypochlorite concentration and energy consumption per kilogram of hypochlorite produced, a two-level factorial design was used for each factor. The best operating conditions produced when the applied electric potential was 10 V, sodium chloride concentration 120 g/L, and treatment time of 60 minutes reaching a concentration of 2276 g/L NaClO and a free chlorine percentage of 0.23 %. As well, the energy consumption per kilogram of hypochlorite is in the range of 3.31 and 11.78 kWh/kg.

**Keywords-** Sodium hypochlorite; On-site generation; Full factorial design; Sodium chloride; Energy consumption; Electrolytic cell

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## 1. INTRODUCTION

Sodium hypochlorite is a strong oxidizing agent, a good disinfectant, bleach, extremely corrosive to metals, very alkaline, and widely consumed in daily life due to its low price and easy accessibility [1]. The most stable aqueous solutions of sodium hypochlorite are those with the following characteristics: low hypochlorite concentration, low concentration of sodium hypochlorite, and low concentration of sodium hypochlorite; pH 11.5-13; absence of metal ions; temperature under controlled storage below 30°C, storage time and packaging in light impermeable containers [2]. Sodium hypochlorite is produced electrochemically by electrolysis, using two inputs brine (water, common salt) and electricity from a concentration of sodium chloride of approximately 3% by weight [3]. Consumers are now increasingly producing their own hypochlorite solutions using electrolytic cells by electrolysis of sodium chloride solutions. Interest in the electrogeneration (on-site) of hypochlorite has increased in recent years, due to the environmental risks associated with the transport, storage, and handling of large volumes of liquid chlorine [4]. Currently, the most common cells used are non-split cells that use diluted brine or seawater electrolytes for the production of sodium hypochlorite and split cells that incorporate an ion exchange membrane [5]. Undivided cells are commonly used because of their simpler and more economical operation [6]. In the previous work, on-site sodium hypochlorite generation (OSEC) indicated that the electrolysis cells are designed for very low brine feed flow rates, narrow electrode spacings, achieved sodium hypochlorite concentrations close to one percent, and designed to provide 1 to 3 days of stored sodium hypochlorite. The electrochlorination in situ by brine electrolysis is shown in Figure 1.



**Figure 1.** Diagram of the sodium hypochlorite generation process

When a potential difference is applied to the undivided electrolytic cell between the anode and cathode, reactions are generated at the electrodes. At the anode the chloride ion is oxidized to chlorine gas, as shown in equation (1).



Electrical current losses may also be due to the anodic oxidation of water to form oxygen and hydrogen ions, eq. (2)



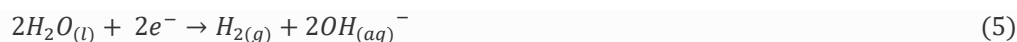
Previous work has shown that metal oxide coating is the most efficient material to change the selectivity of the electrode towards chlorine and hypochlorite evolution reactions versus competing reactions, e.g. oxygen evolution reaction [7]. The usual anode electrodes used in electrochemical cells for NaClO production are platinum-coated titanium. (Ti/Pt) (good), ruthenium oxide coated titanium (Ti/RuO<sub>2</sub>) (excellent) [4]. The electrochemical system continuously forms three chlorine species including chlorine gas (Cl<sub>2</sub>), hypochlorous acid (HClO), and hypochlorite ion (ClO<sup>-</sup>) depending on the pH of the solution. Hypochlorous acid (HClO) and the hypochlorite ion (ClO<sup>-</sup>) can be formed by hydrolysis of electrogenerated chlorine in situ (equations (3) and (4))[8]:



The dissociation of HClO to produce H<sup>+</sup> and ClO<sup>-</sup> according to the reaction of Eq. (4) [9].

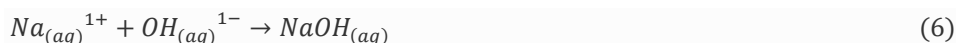


In general, the chlorine available as (HOCl y OCl<sup>-</sup>) is the main indirect oxidizing agent for wastewater treatment [10]. At the cathode, the water decomposes to form hydrogen gas and hydroxyl ions, as shown in eq. (5)[11]

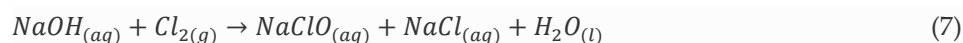


The usual cathodes used in cells for the production of NaOCl are stainless steel (risky); (Ti (good); iridium oxide-coated titanium (very good). The by-product of electrolysis is hydrogen, which is emitted into the atmosphere. Other side reactions occur, but the only important ones that produce an increase in overvoltage are those that result in the formation of layers on the cathode surface, e.g., magnesium and calcium hydroxides and various salts that precipitate from the alkali catholyte when their solubility products are exceeded.

Next, the hydroxyl ions, formed in the equation (5), combine with the sodium ions to form sodium hydroxide, as shown in the equation (6).



The electrolysis products formed at the anode and cathode (i.e., molecular chlorine and sodium hydroxide) respectively are free to mix and result in the on-site generation of sodium hypochlorite (NaClO), as shown in equation (7).



The objective of the present study was to examine the production of sodium hypochlorite using an electrolytic cell. The effect of sodium chloride concentration, electrical potential and electrolysis time was studied with respect to the sodium hypochlorite concentration achieved and specific energy consumption.

## 2. MATERIALS AND METHODS

### 2.1. Chemicals

The chemicals used were analytically pure sodium chloride (NaCl), sodium hydroxide (NaOH), hydrochloric acid (HCl), potassium iodide (KI), sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>), glacial acetic acid and starch, Chemical compounds (salts, acids, and sodium hydroxide) of high purity were provided by the CIMATEC S.A.C company. All solutions were prepared with deionized water, of 18 MΩ. cm supplied by electroionization equipment (EDI) brand of Chiwaterc Water.

### 2.2. Data analysis

The concentration of hypochlorite ions in solution was measured by titration with a 0.1 M solution of sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>), using potassium iodide(KI), acetic acid ( CH<sub>3</sub>COOH) and drops of aqueous starch solution as an indicator, According to reference [12]. A total chlorine test kit (Model 16900; Hach Co.; Iodometric Method 8209) was used. The electric current intensity was recorded from the DC power supply at the beginning of every 2 min and then every 5 min of the test.

### 2.3. Electric Energy Consumption (SEC)

Energy consumption is an important parameter that can be used to evaluate the performance of an electrochemical technology installation. The amount of energy consumed per unit mass (kg) of sodium hypochlorite produced was estimated by equation (8):

$$SEC \left( \frac{kWh}{kg NaClO} \right) = \frac{E_{cel} \int_0^t I dt}{C_{NaClO} V_s} \quad (8)$$

where SEC is the specific energy consumption (kWh/kg), E<sub>cell</sub> is the applied electric potential (V), I is the applied current (A), t is the electrolysis time (h), V<sub>s</sub> is the solution volume (L) equal to 1.2 L and C<sub>NaClO</sub> is the concentration of sodium hypochlorite in (kg/L).

### 2.4. Experimental design

We have investigated the influence of three main factors: electrical potential (X1), initial sodium chloride concentration (X2) and treatment time (X3), on the achieved sodium hypochlorite concentration and specific energy consumption (CEE) using the experimental design methodology by factorial matrix (2<sup>k</sup>). In this type of experimental design, the variables

(k) are set at two levels: low and high normalized as (-1) and (+1). This design was used to investigate the main and interaction effects of the factors on the formation of sodium hypochlorite and subsequently to optimize the treatment process. The selected factors and their levels are presented in Table 1, while the design of the experiment matrix and responses are shown in Table 3. In order to select these main factors and the study levels, some previous tests have been taken as reference, among which the works on the optimization of the parameters of the electrolytic process to obtain sodium hypochlorite for water disinfection [13], effects of the process conditions on chlorine generation and storage stability of electrolyzed deep ocean water [14] and Effects of electrolysis time and electric potential on chlorine generation of electrolyzed deep ocean water have been considered [15].

**Table 1.** Factors and levels

N°	Factors	Unit	Notation	Levels	
				Less	Major
1	Electrical potential	V	X1	6	10
2	NaCl concentration	g/L	X2	80	120
3	Electrolysis time	min	X3	30	60

## 2.5. Electrolytic cell

The electrolytic cell used in the experiment contains two titanium anodes with ruthenium and iridium oxide coating and two pure titanium cathodes, at the ends of the cell pure copper material for contact of electric current of the electrolytic cell with the electrical source. Table 2 shows the most important parameters of the electrodes.

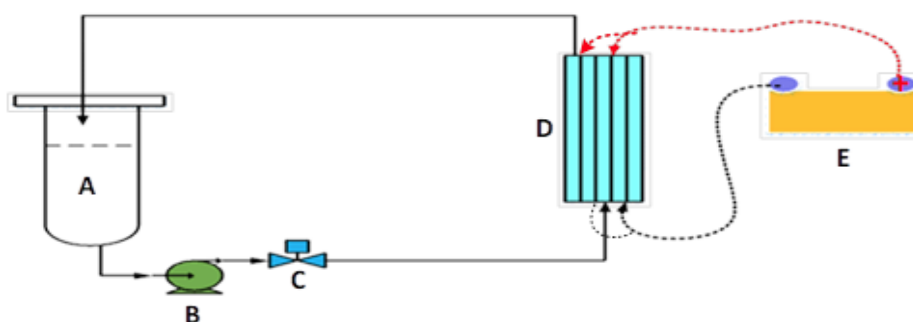
**Table 2.** Most important electrode parameters

N°	Name	Electrolyzer
1	Basic materials (cathode & anode)	Pure titanium
2	Anode coating material	20 um ruthenium & iridium oxides
3	Electrode plate thickness	1.5 mm
4	Distance between the electrode plate	1.8 mm

## 2.6. Experimental procedure

The scheme used to carry out the experiments of this study is shown in Figure 2. The electrolysis experiments of the sodium chloride solution were carried out in an electrolytic cell in discontinuous mode, at room temperature (21°C). For each experiment, container (A) was filled with 1.2 L of sodium chloride solution of 80 g/L and 120 g/L concentrations. The electric

potential applied to the cell was adjusted to 6 and 10 V, kept constant during each experiment, while the electric current was monitored every 5 minutes of treatment. The electrolysis time was 30 and 60 minutes according to the proposed experimental design. Electricity was provided by a DC regulated power supply (0-5 A, 0-30 V). The solution is circulated between the electrolyte storage vessel and the cell by means of a centrifugal pump; the flow rate is adjusted by means of a plastic valve installed in the inlet pipe, as shown in Figure 2. The recirculation flow rate was kept constant throughout the experiment at 3 L/min. The piping system and valve is made of PVC material for hot water and have a diameter of 0.5 inches. A sample of 5 mL of solution was taken at the beginning and at the end of the experiment for sodium hypochlorite analysis. All experiments were repeated twice to check the veracity. An experimental setup of the electrolytic cell process is provided in Figure 2. The experiments were planned according to the factorial design shown in Table 1.



**Figure 2.** Experimental equipment: (A) vessel; (B) pump; (C) flow control valve; (D) electrolytic cell; (E) DC power

### 3. RESULTS AND DISCUSSION

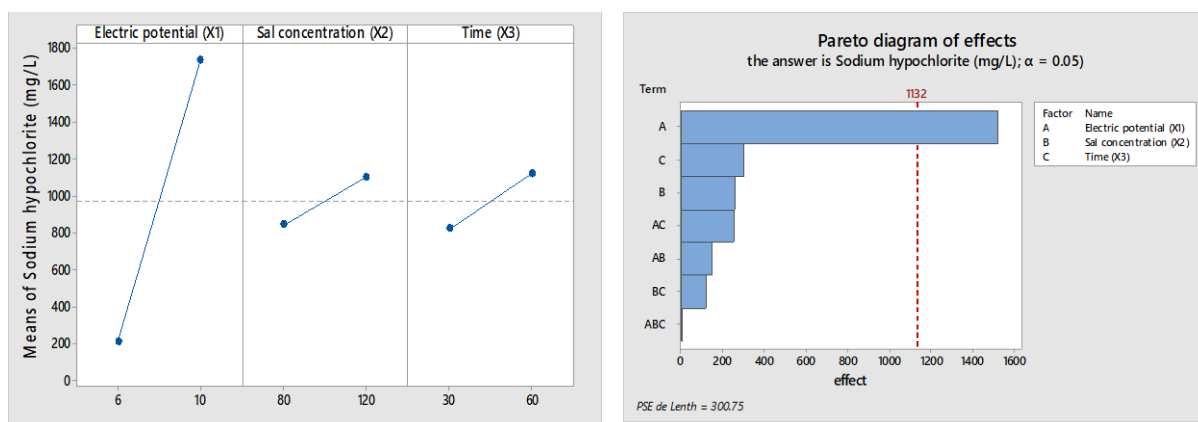
Table 3 shows the results of sodium hypochlorite concentration, percentage of chlorine and specific energy consumption according to the proposed experimental design.

**Table 3.** Factorial design response

N°	Factors			Response variables		
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Sodium hypochlorite (mg/L)	Percentage of chlorine (%)	Energy consumption (kWh/kg NaClO)
1	6	80	30	189	0.02268	3.31
2	10	80	30	1315	0.14171	6.97
3	6	120	30	188	0.02287	4.43
4	10	120	30	1596	0.15232	5.32
5	6	80	60	125	0.00984	5.42
6	10	80	60	1747	0.17820	10.60
7	6	120	60	348	0.05254	4.38
8	10	120	60	2276	0.23203	11.78

### 3.1. Sodium hypochlorite concentration

The main effects of each factor on the response variable (ER) are presented in Figure 3. As expected, the results reveal that changing the level of the electrical potential factor from the low level to the high level had a positive effect on the increase in the concentration of sodium hypochlorite reached. Increasing the potential from 6 to 10 V increased the concentration of hypochlorite from 200 to 1780 mg/L. In previous studies by [16] the authors point out that increasing the electric current density also increases the concentration of sodium hypochlorite. Likewise, it is observed that the concentration of sodium hypochlorite increases with increasing time, reaching at the high level (60 minutes) a concentration of 1200 mg/L of hypochlorite. These results are supported by previous studies developed by [17]. Studies reveal the same trend [18]. The statistical significance of the main factors and their interactions were also confirmed by the Pareto diagram in Figure 3b. For a 95% confidence level ( $\alpha$  value of 0.05), the t-value was 1132 (vertical reference line). Values exceeding the reference line are considered significant. According to Figure 3, the applied electric potential was the only factor statistically significant at the 0.05 level, which corroborated the ANOVA results. An increase of sodium hypochlorite was observed with the concentration of NaCl solution was evident for 120 g/L. Generally, the use of high concentrations of NaCl aqueous solution is always recommended for the industrial production of hypochlorite.



**Figure 3.** a) The main effects plot for sodium hypochlorite concentration; b) Pareto chart of the standardized effects

### 3.2. Analysis of Variance (ANOVA)

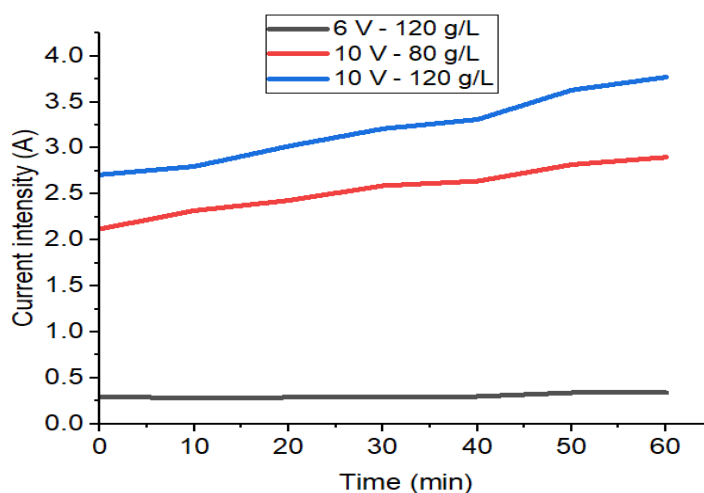
Analysis of variance (ANOVA) was performed on sodium hypochlorite concentration to test for significance. The details of the ANOVA are shown in Table 4. The F value of the model of 8.35 indicates that the model is significant. P values  $<0.05$  indicate that the model parameters are significant. In this case, the applied electric potential (X1) is the most significant parameter ( $p=0.003$ ).

**Table 4.** Analyses of variance (ANOVA) for sodium hypochlorite concentrate

Source	Sum of squares	Contribution (%)	df	Mean square	F-value	p-value
Model	0.050	90.7%	5	0.0101	8.35	0.0305
A-Electric potential	0.0445	80.76	1	0.0445	36.94	0.0037
B-NaCl concentration	0.0014	2.54%	1	0.0014	1.2	0.335
C-Electrolysis time	0.0022	3.99	1	0.0022	1.84	0.2466
AC	0.0012	2.17	1	0.0012	1.03	0.3684
BC	0.0009	1.6	1	0.0009	0.7635	0.4316
Residual	0.0048	8.71	4	0.0012		
Lack of Fit	0.0042	7.62	3	0.0014	2.26	0.4469
Pure error	0.0006	1.08	1	0.0006		
Cor Total	0.0551	100	9	0.0445		

### 3.3. Electric Current Analysis

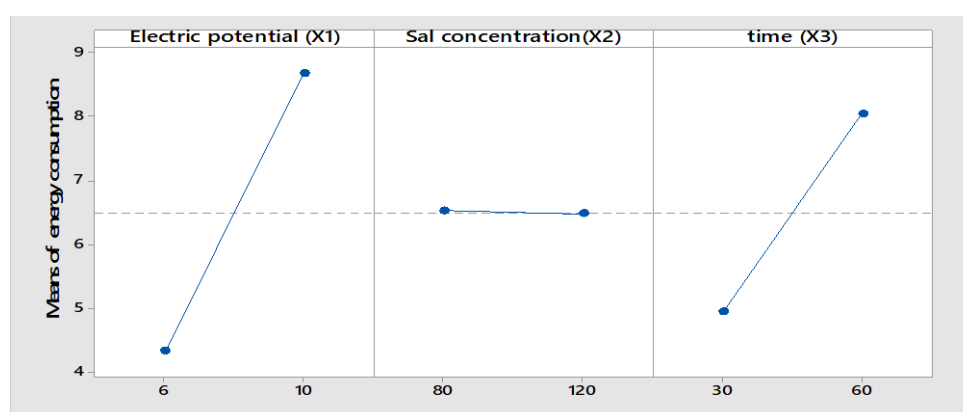
Figure 4 shows the variation of electric current versus electrolysis time for sodium chloride concentrations (80 and 120 g/L) and applied electric potential of (6 and 10 V). The results indicate that the current intensity increases as the electrical potential applied to the cell increases. Likewise, it is observed that for 6 V the electric current remains constant during the 60 minutes of electrolysis. The trend of increasing electric current was observed for 10 V in the experiments for both concentrations of sodium chloride, however, a higher current intensity is reached for a concentration of 120 g/L of sodium chloride solution.

**Figure 4.** Evolution of the electric current as a function of time



### 3.4. Energy Consumption Analysis

Figure 5 presents main effect plots for averages of specific energy consumption caused by electric potential (X1), salt concentration (X2), and electrolysis time (X4). It was observed that increasing the applied electric potential (X1) had a positive effect on energy consumption. The results reveal that the energy consumed to produce one kilogram of sodium hypochlorite increases with the applied voltage. At 6 volts it achieves a power consumption of 4.2 kWh/kg vs. 8.8 kWh/kg NaClO at 10 V. Likewise, it is observed that the energy consumption does not present significant changes with the change of sodium chloride concentration (X2), however, a higher energy consumption is observed with a longer electrolysis time (X3).



**Figure 5.** Mean of specific energy consumption

## 4. CONCLUSION

The in-situ production of sodium hypochlorite is a promising alternative to reduce the lack of drinking water supply in remote and isolated areas of the city. The main objective of this research work was to study the effects of the operating factors of an electrolytic cell, such as applied electric potential, sodium chloride concentration, and electrolysis time, on the efficiency of the in situ electrochemical production of sodium hypochlorite using a batch electrolysis cell and to determine the specific energy consumption. The best operating conditions of the cell occurred when the applied electrical potential was 10 volts, sodium chloride concentration 120 g/L, and treatment time of 60 minutes reaching a concentration of 2276 mg/L NaClO and a free chlorine percentage of 0.23%. Likewise, the energy consumption per kilogram of hypochlorite is in the range of 3.31 and 11.78 kWh/kg NaClO.

### Declarations of interest

This document has not been published and is not under consideration for publication elsewhere. Submission has been authorized by all authors. The authors declare that they have no conflicting interests.

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