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# Bipolar membrane electrolyzer for sodium hypochlorite generation

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## ABSTRACT

The objectives of this research work were the construction and evaluation of a laboratory scale electrolytic cell using a bipolar membrane for the production of sodium hypochlorite. The dependence of the cell operation factors on the sodium hypochlorite concentration and specific energy consumption was evaluated, using sodium chloride solutions previously prepared in the laboratory; the experiments were carried out following a factorial design with three levels (3, 4 and 5 V) for the electric potential and sodium chloride concentration (10 and 30 g/L), maintaining a constant electrolysis time of 120 min and recirculation flow of the solutions in both compartments at 350 mL/min. The results showed that the achieved sodium hypochlorite concentration is in the range of (540–1040 mg/L) and power consumption is in the range of (2.1–5.35 kW/kg of NaClO). The sodium hypochlorite concentration and energy consumption were strongly affected by the electric potential applied to the cell, as the voltage increased from 3 to 5 V. In conclusion, this research revealed that this is a promising technology, especially if low concentrations of sodium hypochlorite are used.

**Keywords:** sodium hypochlorite, electrolysis, bipolar membrane, factorial design.

## INTRODUCTION

Sodium hypochlorite (NaClO) is a major chlorine-based chemical disinfectant that has been widely used for the disinfection of industrial facilities, water management systems and domestic environments (Kim et al., 2021a; Wu et al., 2023). At present, the industrial synthesis of hypochlorite is achieved by the absorption of chlorine gas in a sodium hydroxide solution, which is obtained by electrolysis of a saturated sodium chloride brine (Amikam et al., 2018). Consequently, the development of a new technology to produce high quality NaClO has attracted considerable attention (Sánchez-Aldana et al., 2018, Wu et al., 2023). In situ hypochlorite synthesis has significant advantages, as it only requires sodium chloride in the feed stream for the generation of hypochlorite ions (Carneiro et al., 2024a). In addition, the methodology is characterized by its simplicity and safety, so its potential for application in industrial environments is substantial (Afify et al., 2023). Electrochlorination can be performed with a split or unsplit electrochemical cell (Tzedakis and Assouan, 2014) . In an unsplit cell, the cathode and anode are in a single chamber, whereas the final product is a solution containing NaClO and other chlorine species (Carneiro et al., 2024b) . A split cell has the cathode and anode separated by an ion exchange membrane, so that electrolysis of sodium chloride occurs separately (Kim et al., 2021a). A variety of cation exchange membranes (CEMs) are now readily available on the commercial market and can be used effortlessly for hypochlorite synthesis (Mei et al., 2018). Bipolar membranes (BPM) are a special variety of ion exchange membranes composed of a cation exchange layer and an anion exchange layer, which allow the generation of protons and hydroxide ions through the dissociation of water (Pärnamäe et al., 2021). As a method to improve the efficiency of electrolysis reactions, bipolar membranes are often installed between the anode and the cathode of electrochemical cells (Wu et al., 2023). In the literature, as far as it was possible to investigate, there are two studies on the production of sodium hypochlorite using bipolar membranes. These studies include, Wu et al. (2023) where a new electrolyzer for the electrolysis of NaCl was designed to produce high quality NaClO. The cell was separated into two chambers by a bipolar membrane. Jeon and Rhim (2016) prepared two types of bipolar membranes to perform the electrodialysis process with sodium hypochlorite formation. However, further research is needed to produce hypochlorites in a more efficient and controllable way through the electrolysis process (Kim et al., 2021a). Figure 1 shows a general scheme of the electro-synthesis process with sodium hypochlorite with bipolar membrane. The cell has two compartments, separated by a BPM. Sodium chloride solution enters the anodic chamber from the bottom, and sodium chloride solution is also introduced into the cathodic chamber. The working mechanism of the double chamber electrolyzer is described below.

In the BPM, the water in the intermediate layer dissociates into ions ($H^+$ *y* $OH^-$) under a direct electric field, as shown in Equation 1 (Pärnamäe et al., 2021)

$$H_2O \rightarrow H^+ + OH^- \quad (1)$$

At the cathode, the electric current decomposes water into hydroxyl ions and hydrogen gas, according to reaction 2.

$$H_2O \rightarrow H_{2(g)} + OH^- \quad (2)$$

At the anode, chloride ions are oxidized to produce chlorine gas (Equation 3) (Parra et al., 2024)

$$2Cl(aq)^- \rightarrow Cl_{2(g)} \quad (3)$$

The chlorine ($Cl_2$) that is generated reacts with the $OH^-$ produced from the dissociation of water in the bipolar membrane and leads to the formation of sodium hypochlorite (Eq. 4).

$$Cl_{2(g)} + OH^- \rightarrow Cl^- + ClO^- + H_2O \quad (4)$$

Previous studies have shown that the anolyte compartment is acidified and its pH is reduced to 5, as hydroxide ions are continuously consumed by the formation of hypochlorite (Shen et al., 2023).

In this work, a laboratory-scale electrolytic cell with bipolar membranes was built and used for the in situ production of hypochlorite and sodium hydroxide. The dependence of the cell operation factors on the production of sodium hypochlorite was evaluated, using brines previously prepared in the laboratory; the experiments were carried out following an experimental design with three levels of variation of voltage, salt concentration and time.

## MATERIALS AND METHODS

### Chemicals

The chemicals used were analytically pure sodium chloride (NaCl), sodium hydroxide (NaOH), hydrochloric acid (HCl), potassium iodide (KI), and sodium thiosulfate ($Na_2S_2O_3$), provided by the CIMATEC S.A.C company. All solutions were prepared with deionized water, of 18

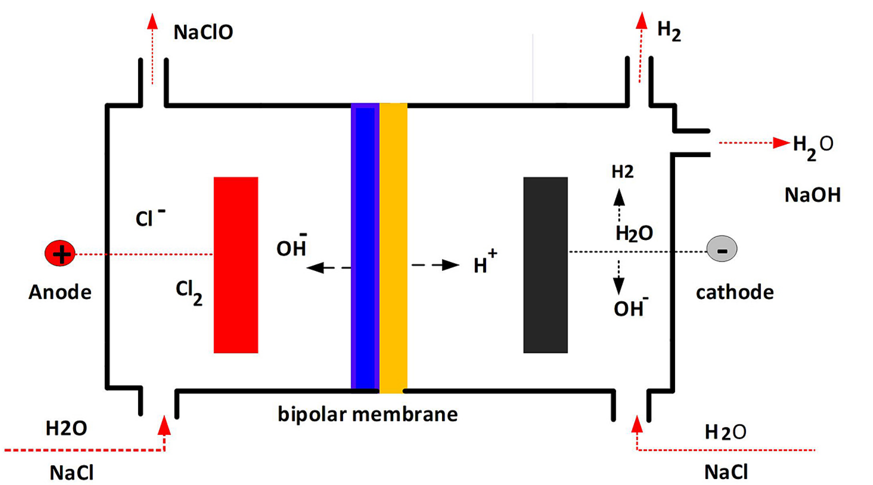


**Figure 1.** Schematic diagram of electrosynthesis with bipolar membranas

$M\Omega.\ cm$. The electrodes used were Ti (cathode) and titanium coated with oxides (Ti-$RuO_2$–$IrO_2$) was used as anode, both electrodes of dimensions 100 × 50 × 1 mm, the electrodes were purchased from the company Fujiang Wiztech Intelligence Technology co., LTD.  Fujian Province, China. The synthetic solution was prepared by dissolving sodium chloride in deionized water to obtain concentration of 10 g/L NaCl (brackish water), 30 g/L NaCl (seawater). Sodium chloride concentrations were established on the basis of previous work (Han et al., 2022).

## Sodium hypochlorite analysis

Conventionally, the iodometric titration method is used for the quantification of sodium hypochlorite (Shen et al., 2023) . In this method, sodium hypochlorite first reacts with potassium iodide under acidic conditions. The iodine formed is then titrated with a standard solution of sodium thiosulfate and the concentration of sodium hypochlorite can be determined (Kim et al., 2021b).

## Bipolar membrane

The company (Fumatech Bwt GmbH, Bietigheim Bissingen, Germany) supplied the bipolar membrane (FBM) for this study, the characteristics of which are shown in Table 1 (Fumasep FBM, n.d.). The electrosynthesis equipment consisted of a bipolar membrane with an effective surface area of 90 $cm^2$. Fumasep bipolar membranes are frequently used in water electrolysis research, but have practical limitations due to their high ohmic resistance.(Ge et al., 2022)

**Table 1.** Bipolar membrane characteristics

| No. | Characteristics | Values |
|---|---|---|
| 1 | Dimensions | 200 × 300 mm |
| 2 | Thickness | 0.13 a 0.16 mm |
| 3 | Maximum operating temperature | 40 °C |
| 4 | Ohmic resistance | ~1.26 Ω |

## Factorial design

In this study, a factorial design was applied to investigate the effect of independent variables such as sodium chloride concentration and electric potential on the achieved concentration of sodium hypochlorite. For this purpose, a matrix table with two factors at two levels for sodium chloride concentration (X1) and three levels for electric potential (X2) was constructed, as shown in Table 2. The electric potential ranges were selected from a literature review (Shen et al., 2023), as well as preliminary research conducted by the author of this study. During the trials, the electrolysis time was kept at 120 minutes, the solution recirculation flow rates at 350 mL/min and solution volume at 0.8 L in both compartments. The ambient temperature for all experiments ranged between 18 and 21 °C.

## Calculation of energy consumption (SEC)

The amount of energy consumed per unit mass (kg) of sodium hypochlorite produced was estimated by Equation 5.

$$SEC\left(\frac{kWh}{kg\ of\ NaClO}\right) = \frac{E_{cel}\int_0^t Idt}{C_{NaClO}V_s} \quad (5)$$

where: $SEC$ is the specific energy consumption (kWh/kg), $Ecell$ is the applied electric potential (V), $I$ is the applied current (A), t is the electrolysis time (h), $Vs$ is the solution volume (L) equal to 0.8 L and $C_{NaClO}$ is the concentration of sodium hypochlorite in (kg/L)

## Electrosynthesis cell

A laboratory-scale electrosynthesis unit, recently designed and built by the author, was used for the experiments. Figure 2 shows the filter press type electrosynthesis cell with two compartments divided by a bipolar membrane, four pieces of synthetic rubber separators were used to seal the electrolyzer and prevent liquid

**Table 2.** Factorial design variables

| No. | Factors | Unit | Notation | Levels | | |
|---|---|---|---|---|---|---|
| | | | | Low | Medium | High |
| 1 | Sodium chloride concentration | g/L | X1 | 10 | 30 | – |
| 2 | Electric potential | V | X2 | 3 | 4 | 5 |

**Figure 2.** Schematic diagram of experimental equipment

spillage and gas leakage (hydrogen and chlorine). The distance between cathode and anode is 10 mm. Two polypropylene plates with eight holes for the entry of the crossbars with nuts were placed on the outside to prevent leakage, mixing and spillage of the electrolyte. The magnitude of the electric current was initially recorded on the power supply indicator at one-minute intervals and at longer intervals thereafter.

## Experimental equipment

Figure 3 shows the diagram of the experimental equipment. The system contains a current rectifier, a bipolar membrane, pumps (P1 y P2), a two-compartment cell, two acrylic vessels with a capacity of 1.5 L and two electrodes acting as anode and cathode (50 × 100 mm). The solutions were recirculated through the two compartments (cathode and anode) by the pumps with a flow rate of 350 mL/min. A volume of 0.8 L of sodium chloride solution was used in each compartment. The anode and cathode were connected to the positive and negative terminals of a digital power supply (DC 0–32 V).

## RESULTS AND DISCUSSION

Figure 4 shows the obtaining of sodium hypochlorite, before electrolysis, milliliters of potassium iodide solution, three drops of starch solution and milliliters of sodium chloride solution were placed in two flasks (A and B), after two minutes of electrolysis, 2 mL were taken from the anolyte compartment and poured into flask B, changing its color from colorless to yellow as shown in Figure 4B. This qualitative analysis confirms that generation of sodium hypochlorite (NaClO) was successful. Previous studies have confirmed this result (Shen et al., 2023).

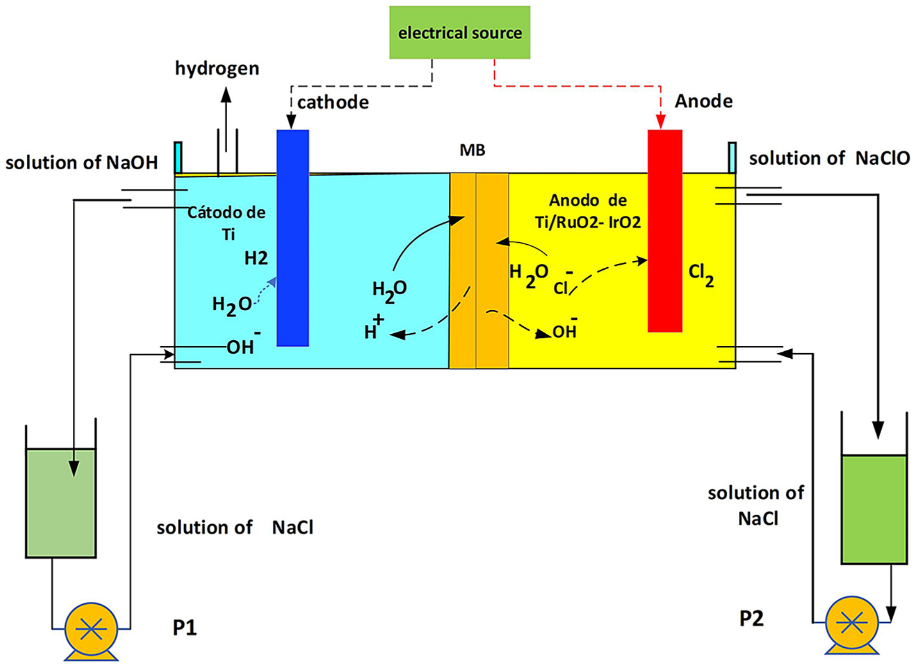


**Figure 3.** Schematic representation of the electrosynthesis with bipolar membranes

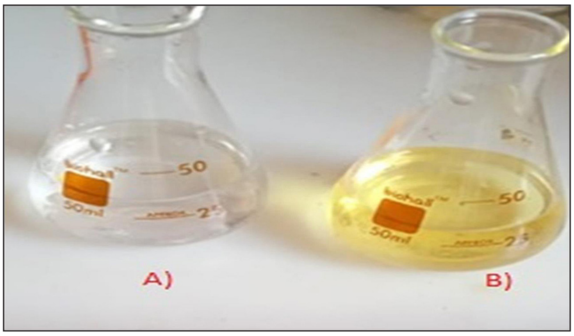


**Figure 4.** Image showing the volumes (milliliters) of the potassium iodide, sodium chloride and starch solutions A) and B) together with the sodium hypochlorite solution generated

Table 3 shows the matrix of experiments, results of sodium hypochlorite concentration, and specific energy consumption.

Table 4 shows the descriptive statistical results for sodium hypochlorite concentration and specific energy consumption. It is observed that the average sodium hypochlorite concentration is of 771.667 mg/L and energy consumption is 3.723 kWh/kg of NaClO

Figure 5 shows the sodium hypochlorite concentrations reached after 120 minutes of electrolysis, applying electric potentials of 3, 4 and 5 volts with electrolyte solution concentrations of 10 and 30 g/L of sodium chloride, for the six tests carried out. It can be seen that in all the tests there is an increase in the concentrations of sodium hypochlorite as the electrolysis time elapses, as well as the increases of the electric potential and concentration of sodium chloride. It was found that the highest concentration of sodium hypochlorite (1030 mg/L) is reached when the electric potential applied to the cell is 5 volts and with a sodium chloride concentration of (30 g/L). Similarly, the lowest concentration of sodium hypochlorite is achieved at 3 volts and a sodium chloride level of 10 g/L. These findings are corroborated by the previous research conducted by (Shen et al., 2024) (Ye et al., 2015).

Figure 6 shows the effect of electrolysis time, sodium chloride concentrations and application of an electric potential held constant at 5 volts on the electric current. The intensity of the electric current increases as the concentration of sodium chloride increases. It is also evident that during the first 20 minutes, the increase in intensity is more pronounced and then the increase is not very significant. These results are supported by previous studies developed by the researchers (Shen et al., 2023).

## Effect of the operating parameters

Figure 7 shows the effect of electric potential and sodium chloride concentration on the concentration of sodium hypochlorite. From the figure, it can be seen that the most influential factor is the electric potential applied to the cell. The most appropriate level to reach the highest level of NaClO concentration was at 5 V, achieving a concentration of 1040 mg/L. It is also observed that the sodium chloride concentration shows a slight improvement at the value of 30 g/L.

Figure 8 shows the effect of electric potential and sodium chloride concentration on average specific energy consumption. From the figure, it can be seen that the factor with the

**Table 3.** Factorial design matrix and responses

| N° tests | Electric potential (V) | Concentration of NaCl (g/L) | Concentration of NaClO (mg/L) | Energy consumption (kW-h/kg NaClO |
|---|---|---|---|---|
| 1 | 3 | 10 | 580 | 2.1 |
| 2 | 3 | 30 | 620 | 1.86 |
| 3 | 4 | 10 | 690 | 4.15 |
| 4 | 4 | 30 | 670 | 4.86 |
| 5 | 5 | 10 | 980 | 4.02 |
| 6 | 5 | 30 | 1040 | 5.35 |

**Table 4.** Descriptive results

| Variable | N | Average | Dev.est | Variance | Minimum | Maximum |
|---|---|---|---|---|---|---|
| Concentration of NaClO (mg/L) | 6 | 771.667 | 192.085 | 36896.7 | 580 | 1040 |
| Energy consumption (kWh/kg of NaClO) | 6 | 3.72333 | 1.43634 | 2.06307 | 1.86 | 5.35 |

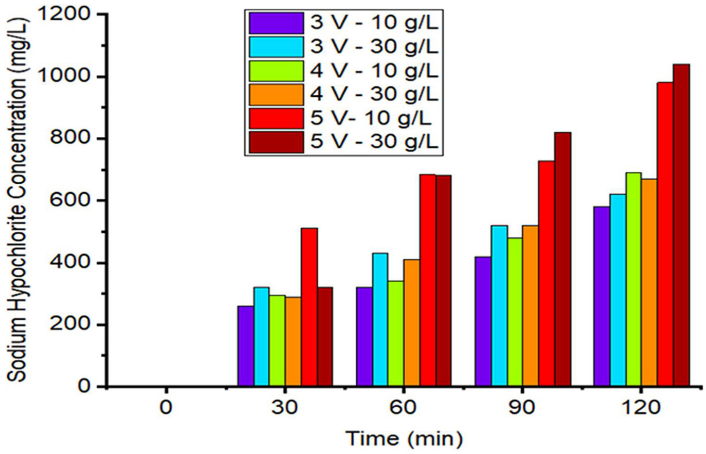


**Figure 5.** Evolution of sodium hypochlorite concentration as a function of time

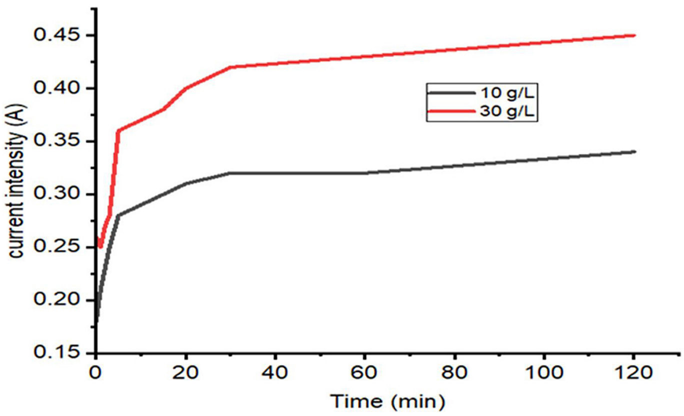


**Figure 6.** Current intensity - time electrolysis curve in 10 y 30 g/L of sodium chloride

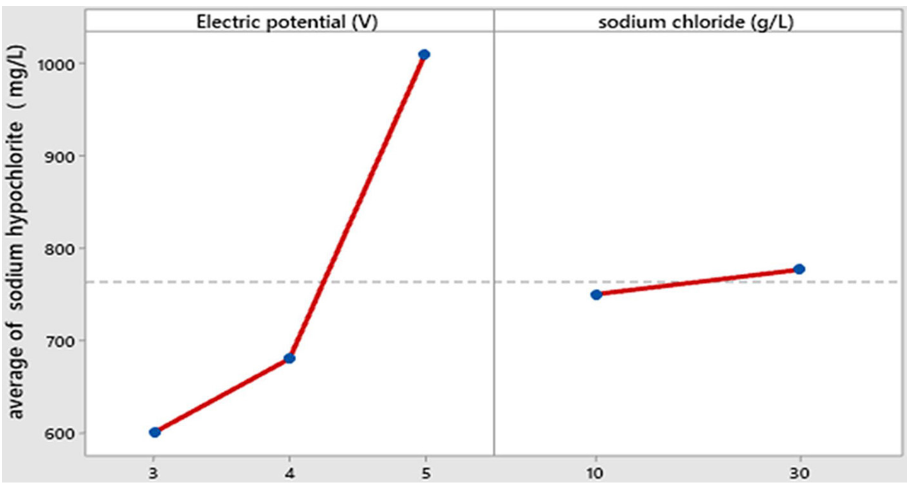


**Figure 7.** Mean of sodium hypochlorite concentration

greatest influence on energy consumption is the electric potential, at the high level (5 V) reaching a consumption of 5.35 kWh/kg NaClO and the lowest energy consumption at an electric potential of 3 V, the value of which is 1.86 kWh/kg NaClO. It is also observed that the sodium chloride concentration shows a slight increase in the value of 30 g/L on energy consumption. At the concentration of 10 and 30 g/L (average consumption 3.42 and 4.02 kW/kg NaClO).

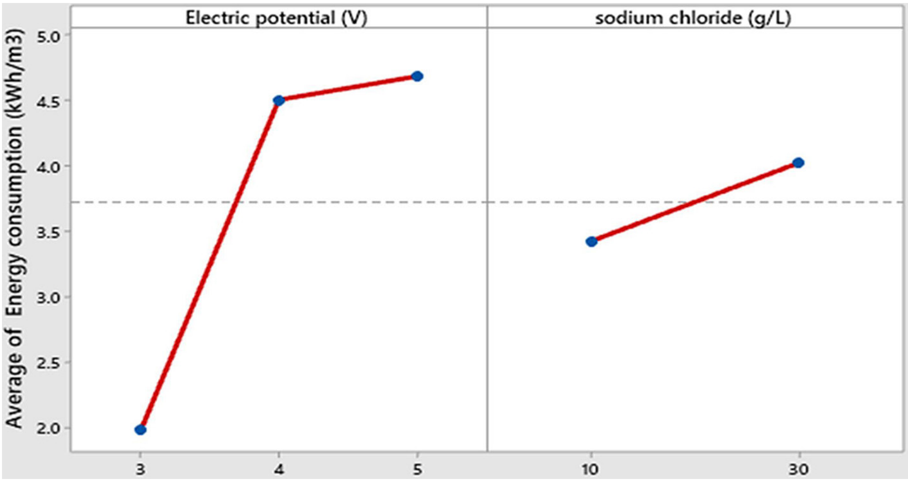


**Figure 8.** Mean of energy consumption

## Analysis of variance

An analysis of variance (ANOVA) is a statistical tool used to investigate and evaluate experimental results. ANOVA was performed to study the impact of individual factors on the results. Table 5 shows the results of the analysis of variance for sodium hypochlorite concentration and specific energy consumption in 120 minutes of electrolysis. The validity of the model was justified by Fisher's test (F value) and the probability test (p-value). The F-value of 89.23 and 11.78 indicates that the model is significant. The p-value of 0.002 and 0.038 indicates that the model is adequate, since it is much lower than the value at 0.05 for the electric potential factor and the response variables of sodium hypochlorite concentration and energy consumption. The electric potential applied to the cell contributes the most to the hypochlorite concentration versus energy consumption if the p-values obtained are compared, as shown in Table 5.

Previous studies by (Wu et al., 2023), designed a bipolar membrane electrolyzer for NaCl electrolysis to produce high quality NaClO; after 3 h of electrolysis, sodium hypochlorite reached a concentration of 531.8 mg/L. The research work conducted in the presented paper corroborates these results, after 120 minutes of electrolysis a sodium hypochlorite concentration of the order of (580 to 1040 mg/L) was reached. Previous studies by (Jeon and Rhim, 2016) entitled "Study on hypochlorite production using newly synthesized bipolar membranes for the electrolysis process" reported the concentration of sodium hypochlorite as a function of electric potential over a time period of 240 min for two types of membranes, as shown in Table 6. The presented research work corroborates these

**Table 5.** Analyses of variance (ANOVA) for concentration of NaClO and energy consumption

| Response | Source of data | Degree of freedom(DF) | Sum of squares | MC ajust. | F-value | p-value |
|---|---|---|---|---|---|---|
| Concentration of NaClO (mg/L) | Electric potential (V) | 2 | 181433 | 90717 | 89.23 | 0.002 |
| | Error | 3 | 3050 | 1017 | | |
| | Total | 5 | 184483 | | | |
| Energy consumption (kWh/kg | Electric potential (V) | 2 | 9.150 | 4.5750 | 11.78 | 0.038 |
| | Error | 3 | 1.165 | 0.3884 | | |
| | Total | 5 | 10.315 | | | |

**Table 6.** Concentration of sodium hypochlorite – electric potential (Jeon and Rhim, 2016)

| Membrane | Electric potential (V) | Concentration of sodium hypochlorite (mg/L) |
|---|---|---|
| SPEEK/APSf 2:1 | 4 | 72.3 |
| SPEEK/APSf 3:1 | 6 | 123.4 |

results, since after 120 minutes of electrolysis a sodium hypochlorite concentration of the order of (580 to 1040 mg/L) was reached. In this work, a commercial membrane was used; it is likely that the difference in sodium hypochlorite concentration produced is due to this factor.

## CONCLUSIONS

A new bipolar membrane electrolysis cell was built for the production of sodium hypochlorite. During the electrolysis process, a concentration of 1030 mg/L NaClO was achieved by applying an electrical potential of 5 volts and a sodium chloride concentration of 30 g/L in an electrolysis time of 120 minutes. The ANOVA results revealed that the contribution of the applied electric potential is the highest with a p-value (0.002). It was observed that the lower the electrical potential (3 V) applied to the cell and the lower the sodium chloride concentration (10 g/L), the lower the energy consumption (2.1 kWh/kg of NaClO). Likewise, the highest energy consumption corresponds to the electrical potential levels of 5 V and NaCl concentration of 30 g/L, obtaining 5.35 kWh/kg of hypochlorite. In conclusion, the research has confirmed that this technology is very promising, especially when low concentrations of sodium hypochlorite are used.

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