

Electrosynthesis of Sodium Hypochlorite using ion Exchange Membrane

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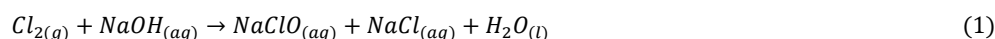
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In this article, we report on a newly designed *in situ* sodium hypochlorite generation process at laboratory scale. An anion exchange membrane was placed between two cation exchange membranes, dividing the cell into distinct compartments. The factorial design comprised three level variations for sodium chloride concentration as well as for electrical potential on the effect of sodium hypochlorite concentration and specific energy consumption. The most favorable operating conditions gave a concentration of 1800 mg/L of NaClO with a solution of 20 g/L NaCl and a voltage of 15 volts. The findings indicate an energy expenditure between (8.13 to 35.68 kWh/kg NaClO) associated with the production of a concentration between (906.9-1800 mg/L) of sodium hypochlorite. Based on the results achieved, it is possible to build an electrolyzer with an ion exchange membrane to obtain a sodium hypochlorite solution from low concentration sodium chloride.

1. Introduction

Access to safe drinking water is essential for sustaining humanity, and hypochlorite has become an important product for water purification due to its economy and effectiveness in various water systems, including natural waters and drinking water sources, in addition to its applications as a household bleach and antiseptic, especially in public health emergencies (Cheng et al., 2022; Pan et al., 2023). Sodium hypochlorite (NaClO) is widely used as a disinfectant in both residential and clinical settings to eradicate pathogens, including viruses and bacteria, from a variety of surfaces (Henwood, 2020). In addition, it is also used to treat hospital waste and wastewater (Wang et al., 2020). The electrolytic production of NaClO from NaCl solutions began in the early 20th century and, at present, the industrial production of hypochlorite is carried out by absorption of chlorine in a 21% sodium hydroxide solution, as illustrated in equation (1); both are produced by electrolysis of concentrated NaCl brine (Crook & Mousavi, 2016; Thangappan & Sampathkumaran, 2008).



The three predominant electrolysis technologies used for the synthesis of chlorine, sodium hydroxide and hydrogen include the mercury cell, the membrane cell and the diaphragm cell (Garcia-Herrero et al., 2017). Most of the free Cl₂ is produced electrochemically by electrolysis of water in equipment known as chlor-alkali cells. There are three most common cell configurations, one older type of system uses mercury cells, while the second relies primarily on asbestos diaphragm cells. The third, more modern type uses polymer electrolyte membranes (PEM) (Liu, 2023).

In situ production, so-called on-site generation (OSG) of hypochlorite by NaCl electrolysis might be a good alternative to avoid the use of hypochlorite or bulk chlorine gas due to its hazardous nature (Dénis et al., 2022). Generating sodium hypochlorite on-site is a simple and straightforward process using three common consumables: salt, water and electricity. Positively charged plates are called cathodes and negatively charged

plates are called anodes. Today, different types of cells are commercially used for the production of sodium hypochlorite, the most common being the non-split cells, where the cathode and anode operate in a single chamber and the final product consists of a solution containing sodium hypochlorite (NaClO) and other chlorine species, and the split cells, which incorporate an ion exchange membrane between the anode and cathode (Thangappan & Sampathkumaran, 2008). Undivided cells are used more frequently due to their cheaper and simpler operation compared to divided cells. To produce an uncontaminated and more concentrated hypochlorite solution, it is necessary to separate the cell from each electrode by means of a semi-permeable barrier such as an ion exchange membrane. Electrodialysis is an advanced separation technology that uses an electric current to move ions through semipermeable membranes (Kowalik et al., 2024). Ion exchange membranes are classified into cation exchange membranes (CEM) which are permeable to cations and anion exchange membranes (AEM) which are selectively permeable for anions (Kikuchi et al., 2024). Previous studies have reported on the newly designed hypochlorite ion generation process, which involves the installation of cation exchange membranes (CEM) (Kim et al., 2021). Jeon et al. (Jeon & Rhim, 2016) and Wu et al. (S. Wu et al., 2023) conducted experimental investigations on the production of sodium hypochlorite by the application of bipolar membranes inside an electrodialysis apparatus using sodium chloride solution. Both researchers have skillfully demonstrated that the synthesis of sodium hypochlorite is indeed feasible, resulting in an increase in NaClO concentration when subjected to an increase in electric potentials. Recently, the elctrosynthesis of sodium hypochlorite by bipolar membrane has been reported by Medina et al. (Medina Collana et al., 2025) reaching concentrations of 1040 mg/L NaClO at a potential of 5 volts in 120 minutes of electrolysis. The objective of this work was to investigate the performance of the newly constructed electrodialysis cell on the concentration of sodium hypochlorite produced from a model solution. To the best of the author's knowledge, this is the first work using this ion exchange membrane configuration to produce sodium hypochlorite. The effect of the Initial sodium chloride concentration and the applied electrical potential in the influence on sodium hypochlorite produced and the specific energy consumption was studied.

2. Materials and methods

2.1 Chemicals

The chemicals used were sodium chloride (NaCl), hydrochloric acid (HCl), potassium iodide (KI), sodium thiosulfate (Na₂S₂O₃) and analytically pure starch. The high purity chemical compounds were supplied by the company CIMATEC S.A.C. The synthetic solution was prepared with distilled water containing sodium chloride (NaCl).

2.2 Analysis de hypochlorite de sodium

The concentration of hypochlorite ions in the product solution was measured by titration with a 0.1 N solution of sodium thiosulfate (Na₂S₂O₃), using a mixture of potassium iodide, hydrochloric acid and aqueous starch solution as an indicator. To calculate the concentration of hypochlorite ions is determined by the following equation.

$$\text{Conc. Hypochlorites} \left(\frac{\text{mg}}{\text{L}} \right) = \frac{AxBx37.22x1000}{C} \quad (2)$$

where A is the volume of the sodium thiosulfate solution used (mL), B is the normality of the sodium thiosulfate solution (0.1 N), C is the weight of the sample used (g).

2.3 Ion exchange membranes

Heterogeneous cation exchange (MK-40) and anion exchange (MA-41) membranes (Shchekinoazot, Pervomaisky, Russia) is their manufacturer. The main characteristics of the two membranes are summarized in Table 1.

Table 1: Physical-chemical characteristics of the membranes used adapted from

Membrane / Characteristic	MK-40	MA-41
Electric resistance (Ω .cm ²)	2	<11
Ion-exchange capacity (meq/g dry membrane)	2	1.6
Transport number	>0.98	0.98 \pm 0.02 in 1 M NaCl
Thickness (μ m)	520	530 \pm 20

2.4 Design of experiment by full factorial method

The design contemplated the effect of two factors at three levels for each variable to evaluate their effect on sodium hypochlorite concentration and energy consumption. The two operational variables and the levels considered are presented in Table 2. The effect of the initial concentration of sodium chloride (A) at three levels (7, 10 and 15 g/L) and the electric potential applied to the cell (B) at three levels (8, 12 and 15 volts) has been studied. applied to the cell at three levels (8, 12 and 15 volts). A total of 18 experiments, including replicates, were performed.

Table 2: Experimental process parameters and levels

Factors	Notation	Units	Levels		
			Low	Medium	High
Initial sodium chloride concentration	A	g. L ⁻¹	7	10	15
Electric potential	B	V	8	12	15

2.5 Calculation of Energy Consumption (SEC)

To measure energy consumption, the specific energy consumption (SEC), which is defined as the energy consumed to produce one unit of product (NaClO), is commonly adopted. The consumption of electrical energy is an economic parameter of great importance in the electro dialysis process. Is directly proportional to the applied electric potential, to the treatment time, to the electric current intensity and inversely to the volume used. In this study, the specific electrical energy consumed (SEC) is defined as the amount of electrical energy per cubic meter, was calculated by (Gherasim et al., 2014; X. Wu et al., 2022) (Bejjany et al., 2017) was estimated by equation (3)

$$SEC \left(\frac{kW.h}{kg NaClO} \right) = \frac{U_{celda} \int_0^t I dt}{C_{NaClO} x V_s} \quad (3)$$

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).

2.6 Electrodialysis cell configuration

The membrane configuration of the electro dialysis cell used in this study is shown in Figure 1 (a). An anion exchange membrane was placed between two cation exchange membranes, dividing the cell into four distinct compartments. A compartment for anode wash solution (I), a concentrated solution (II), dilute solution (III) and cathode wash solution (IV). Sodium ions migrate to the cathode through a cation exchange membrane (CEM) but are blocked by the exchange membrane anionic. Chloride ions move towards the anode through the anionic membrane (AEM) but are blocked by the cationic membrane. Throughout the treatment, compartment III is gradually depleted of ions; this compartment is called dilute while compartment (II) is increased with sodium and chloride ions and is called concentrated solution.

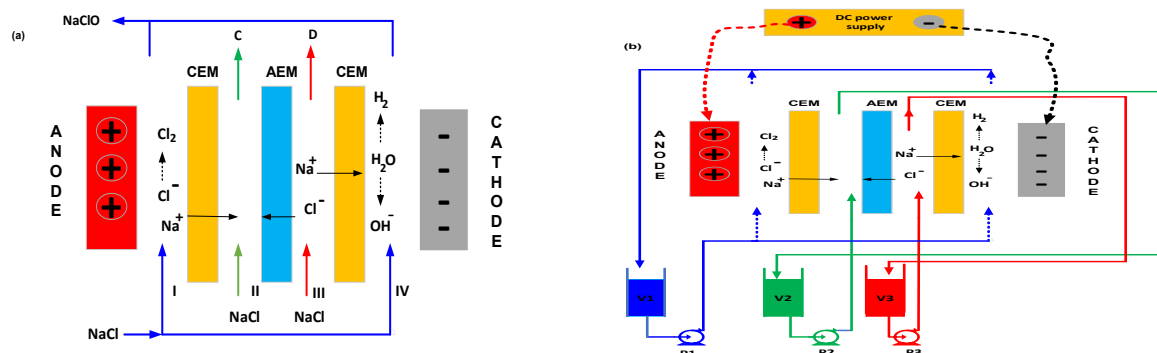


Figure 1: (a) Configuration of the membrane. (b) Experimental module

In the cathode compartment (IV), the electric current decomposes water into hydroxyl ions and hydrogen gas, according to reaction (2).



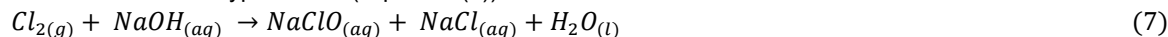
In this process, sodium ions passing through the cationic membrane from compartment (III) to compartment IV and hydroxide combine to produce sodium hydroxide (caustic soda).



In the anodic (I) compartment, chloride ions are oxidized to produce chlorine gas (equation (3)).



Chlorine generated at the anode reacts with NaOH produced from the cathode compartment and results in the formation of sodium hypochlorite (Equation (4)).



2.7 Experimental unit

Figure 1(b) shows the schematic of the experimental electro dialysis system used in the sodium hypochlorite synthesis trials, which includes two cation exchange membranes and one anion exchange membrane. The effective membrane area of each membrane is (80 mm × 120 mm). The anodes were made of titanium coated with ruthenium oxide and iridium oxide (Ti-RuO₂ -IrO₂), the cathode was of pure titanium(Ti), both electrodes have an effective area of 50 cm² (5 cm×10 cm). The concentrated, diluted and rinsing solutions of the electrodes were placed in three cylindrical containers of acrylic material with a volume capacity of 1000 mL (V1, V2 and V3). The solutions were recirculated through the diluted, concentrated and electrode wash compartments by pumps (P1, P2 and P3) at a flow rate of 1100 mL·min⁻¹. The anode and cathode were connected to the output (negative and positive) of a digital power supply (DC 0-30 V, 5 A UNI-T UTP). The intensity of the electric current was read on the power supply indicator every 5 minutes. Sodium hypochlorite (NaClO) concentration and electrical conductivity were analyzed every 30 min. The temperature of the solutions was maintained at 18 ± 2 °C in all experiments.

3. Results of model solution

The experimental design created according to the factorial design and the sodium hypochlorite concentrations and energy consumption obtained in the trials are listed in Table 3. A total of nine replicate experiments were performed and the average is reported.

Table 3. Results of the experimental design

N°	A(g/L)	B(V)	Concentration of NaClO (ppm)	Energy consumption kW-h/kgNaClO
1	10	8	971.1	8.13
2	10	12	906.9	23.91
3	10	15	1030.2	32,57
4	15	8	716.5	15.07
5	15	12	1167.7	24.87
6	15	15	1380.6	32.45
7	20	8	885.3	14.33
8	20	12	1330.0	21.55
9	20	15	1800.0	35.68

Table 4 presents results of descriptive statistics on the parts per million sodium hypochlorite produced. Shows the mean of the 9 tests for sodium hypochlorite produced is 1122.1 ppm and the standard deviation was 328.2

Table 4. Mean parts per million NaOCl produced

Response	N	Mean concentration(ppm)	Standard error	Desv.Est	Variance	Minimum	Maximum
sodium hypochlorite produced	9	1122.1	77.4	328.2	107739.6	691.0	1810.0

Figure 2 shows the sodium hypochlorite concentration at 30, 60 and 90 minutes of electrolysis for the nine experimental tests, according to the factorial design. From the figure it can be clearly observed how the concentration of sodium hypochlorite increases as the electrolysis time elapses throughout all the tests performed. Similarly, it can be observed in test nine at the levels of (20 g/L NaCl and 15 Volts) the increase in the concentration of NaClO is more accentuated over time, achieving a concentration of 1800 mg/L at the end of the test. Previous studies show that the performance of sodium hypochlorite electrosynthesis increases with increasing current density and NaCl concentration in the system (Baydum and Sarubbo, 2022).

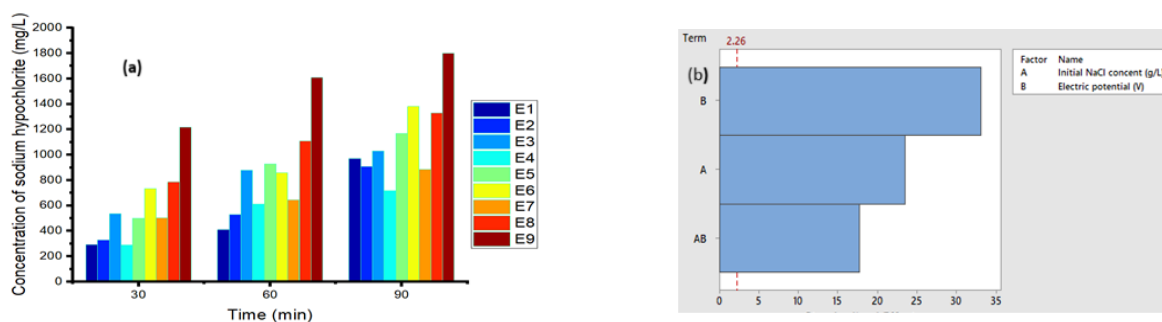


Figure 2: (a) evolution of NaClO concentration with time, (b) Pareto diagram for concentration of NaClO

The Pareto diagram is an exceptionally effective tool for the visual delineation of the comparative importance of various factors within a system, employing the use of bars and a vertical line (Ikegwu et al., 2021). The vertical line indicates the minimum degree of effect for a confidence level of 95% (Saleh et al., 2018). According to the Pareto diagram, horizontal bars that exceed the vertical reference line (2.26) are statistically significant. According to the Pareto diagram shown in Fig. 2(b), with a confidence level of 95%, the effects of the operating variables of electrical potential and initial sodium chloride concentration and the interactions between them were significant, with electrical potential being the variable with the greatest influence in achieving the highest sodium hypochlorite concentration. Cuesta-Parra et al. reported that the hypochlorite obtained is significantly enhanced at high chloride concentrations, high current intensities and prolonged times, using a 22-liter electrolysis cell with graphite electrodes (Cuesta-Parra et al., 2023). According to previous studies by the current density is strongly influenced by the electric potential, increasing the voltage and increasing the current density improved the production rate of sodium hypochlorite, as mentioned in previous works (Cuesta-Parra et al., 2024). Afify et al. (Afify et al., 2023) have mentioned that as sodium chloride concentration increased, there was a corresponding increase in sodium hypochlorite concentration.

4. Conclusions

A new sodium hypochlorite production system based on electrolysis using a combination of cation and anion exchange membranes has been proposed. The most suitable parameters were the sodium chloride concentration of 20 g/L and the electrical potential of 15 V applied to the cell, resulting in a sodium hypochlorite concentration of 1800 g/L and an electrical energy consumption of 35.68 kWh/kg NaClO produced, clearly demonstrating that in situ electro-synthesis of sodium hypochlorite can be used as a method of NaClO production.

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