ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

SCHOOL OF CHEMICAL AND BIO ENGINEERING

M.Sc. Program in Environmental Engineering

Electrolysis Sodium Hypochlorite Generation from Sodium Chloride Solution

A thesis submitted to Addis Ababa Institute of Technology, School of Chemical and Bio Engineering in partial fulfillment of the requirements for the Degree of Master of Science in Environmental Engineering

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Declaration

I declare that, this thesis work entitled “Electrolysis generation of sodium hypochlorite from sodium chloride solution” for the partial fulfillment of the requirements for the Degree of Master of Science in Environmental Engineering at Addis Ababa Institute of Technology, hereby submitted by me, is my original work and has not been previously submitted for a degree at this university or any other university, and that all resources of materials used for this thesis have been duly acknowledged.

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This thesis has been submitted for examination with my approval as a University Advisor.

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ABSTRACT

The scarcity of fresh-water has become one of humanity's most pressing problems today. Chlorine was chosen as the disinfection of choice because it was cost-efficient, effective, and easy to use. Chlorine was introduced to water as a gas or a liquid (NaOCl). For safety reasons, NaOCl was frequently used instead of chlorine gas. It was a viable disinfectant for controlling microbial growth and sanitizing inert surfaces. One of the alternate ways used as value-added disinfection was on-site electrolytic NaOCl production. To achieve the goal, a batch laboratory-scale reactor was used, with a Ti/IrO_2 anode and stainless-steel cathode.

To find the best optimal conditions, the RSM experimental design was used. The effects of the most important variables, the creation of optimal conditions, and the construction of the optimum sodium hypochlorite concentration were studied. The experiment was carried out with a variety of variables, including NaCl content, electric potential, electrode gap, and electrolysis time. At 32g NaCl, 7v, 2.5cm electrode gap, and 70min with pH of 11 and ambient temperature of 25°C, 7.2g Cl_2/l of NaOCl was created under the best-optimized circumstances for NaOCl generation. The results show that the NaOCl generated can disinfect water and inert surfaces.

***Keywords**: sodium hypochlorite, electrolysis, on-site, experimental design*

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List of Abbreviation

ANOVA	Analysis of Variance
APHA	American Public Health Association
AWWA	American Water Work Authority
BBD	Box Behnken design
DC	Direct current
FTIR	Fourier Transform Infrared Spectroscopy
NaOCl	Sodium hypochlorite
nm	Nanometer
OSEC	On-site electrochlorination
OSG	On-site generation
RSM	RESPONSE SURFACE METHODS
V	Voltage
WHO	World health organization

CHAPTER ONE
INTRODUCTION

1.1 Background

Chlorine is a chemical that is commonly used as an oxidizing agent in the disinfection of water and wastewater. This oxidizing agent comes in a variety of forms, including gaseous (Cl_2), liquid (NaOCl, OCl^-), and solid (OCl^-)[1]. For water treatment, sodium hypochlorite (NaOCl) is available as a transparent solution with a high chlorine concentration. For safety considerations, NaOCl was frequently used instead of gas disinfection. NaOCl is used for surface and water disinfection, fabric bleaching, and odor elimination on a wide scale[2]. For anodal oxidation of pigment particles and phenols in wastewater, on-site generation (OSG) of hypochlorite was used. As a bleach and disinfectant, it has a number of advantages, including simple dosing, secure storage and transportation, and no leftover flow.

Commercially, sodium hypochlorite is now made by electrolysis of sodium chloride (NaCl) solution. By putting an electric current across bound components and compounds, electrolysis could be used to isolate them. A dissolvable, such as purified water, is used to break down an ionic substance, in this case, salt, so that its particles are available inside the liquid. A power source is connected between a pair of dormant electrodes submerged within the fluid. The cathode is the negatively charged electrode, while the anode is the positively charged electrode. Each electrode draws in particles that are oppositely charged. Hence, positively charged particles (called cations) move towards the cathode, whereas adversely charged particles (named anions) move toward the anode. The energy required to isolate the ions, and cause them to assemble at the particular electrodes, is given by a coordinate electrical control supply. At the investigations, electrons are retained or discharged by the ions shaping a collection of the specified component or compound.

NaOCl can be generated by the electrolysis of NaCl aqueous solution (brine) solution in a completely blended unified cell that produces sodium hypochlorite and hydrogen gas. Hydrogen gas was bubbled up at the cathode. The principal reactions are as following:

$$Sodium\ chloride + Water + Electricity \rightarrow Hypochlorite + Hydrogen\ gas$$

$$NaCl + H_2O + E \rightarrow NaOCl + H_2 \qquad 1.1$$

The method has the advantage that the hypochlorite arrangement can be created on-site, in this way dodging the dangers of transporting, putting away, and dealing with fluid chlorine and gas

chlorine and the trouble of assembly all the safety-related activities required.

In this ponder, we have explored the electrolysis of sodium hypochlorite generation from the sodium chloride solution utilizing two distinctive electrodes (IrO_2/Ti as anode and stainless steel as a cathode). Hypochlorite is broadly utilized in sanitization, disinfection, making (plastics, solvents for dry cleaning and metal degreasing, textiles, dyestuffs, household cleaning items, etc.). The impact of distinctive process parameters (such as sodium chloride concentration, electric potential, electrode gap, and electrolysis time) on the sodium hypochlorite generation from aqueous sodium chloride solutions by electrolysis was considered.

1.2 Statement of the Problem

Treating water and wastewater is a vital issue to decrease environmental contaminants and problems. In addition to this, due to expanding population and growth in many regions of the world, wastewater recycling and reuse are critical to supplying water needs for irrigation, industry, and household employment. The final phase in the treatment of metropolitan wastewater was disinfection. Chlorination was the primary method of wastewater disinfection. Apart from its effectiveness, however, there are worries regarding the social and environmental consequences of its use[3].

Because the storing, shipping, and handling of any type of chlorine gas is hazardous to the public's health, safety rules have been abolished. When inhaled in small amounts, gas is a greenish-yellow gas that can irritate. According to the National Transportation Security Board (NTSB) and the Coast Guard, a chlorine gas spill on a board can move two miles in as little as ten minutes and do severe damage to nearly 20 miles away. The most advantage of the electrolysis handle is the generation of disinfectant on-site within the treatment device, hence maintaining a strategic distance from the drawbacks of common chlorination such as transportation and storing of perilous chlorine.

The purpose of OSG of NaOCl solution from sodium chloride solution is to financially and securely produce this capable biocide and disinfecting agent for utilizing in industrial plants and households anyplace we apply by utilizing alternative energy (like solar, geothermal, wind…etc.) sources. Considering this, electrolytic sodium hypochlorite generation is one of the elective strategies utilized as a value-added disinfectant to disinfect inert and water surfaces.

1.3 Objective

1.3.1 General objectives

The main objective of this study is to optimize sodium hypochlorite (NaOCl) generation from NaCl solution using the electrolysis process.

1.3.2 Specific Objective

- To determine the effects of NaCl concentration, electric potential, electrode gap, and electrolysis time on NaOCl generation.
- To analyze the residual chlorine concentration in the electrolysis generation of NaOCl.
- To optimize the NaOCl generation process by using an RSM Statistical experimental design.
- To validate and confirm optimization.

1.4 Significance of the study

The scarcity of fresh-water has become to be one of the greatest recent challenges to human creatures[4]. Chlorine is for the most part the disinfectant of choice because it is sensibly productive, cheap, and simple to handle. In all but the smallest water treatment plants, chlorine is included in water as either in liquid (sodium hypochlorite) or chlorine gas. Sodium hypochlorite is a successful disinfectant utilized to control the development of algae, devastate microscopic organisms, infections, and oxidize natural matter. It is produced on a huge scale by electrolysis of profoundly concentrated preparations of sodium chloride. Current chlorinator frameworks utilize a full or half-wave rectifier to change over primary AC to low voltage coordinate current (DC) to control the cell electrolysis plates. The sum of chlorine is corresponding to the charge passing within the electrode. One of the pivotal advantages of the electrolysis process is the on-site generation of liquid chlorine (NaOCl) on the solution. It is applied on an incredible scale for surface disinfecting, decolorizing, odor expulsion, and water disinfection.

It shows a wide extend of antimicrobial activity and is reasonable against some common pathogens at distinctive concentrations It is compelling against rotavirus at a concentration of 0.05% (500 ppm), in any case, higher concentrations of 0.5% (5000 ppm) are required for a few exceedingly secure pathogens inside the health-care setting.

The World Health Organization's suggestion of 0.1 percent (1000 ppm) in the COVID-19 context may be a preservationist concentration that will inactivate the ever-increasing range of other pathogens that will be found inside the healthcare setting[5].

The following are the key advantages of producing sodium hypochlorite solution on-site from sodium chloride solution:

- Better operator security
- Better quality chemicals
- Fewer by-products
- Environmentally friendly
- Cost-effective

1.5 Scope of the Study

This study focused on the NaOCl generation from NaCl solution using anelectrolytic process. Also, the effect of selected operating parameters such as sodium chloride concentration, electric potential, electrolysis time, and electrode gap and optimizing them for electrolysis NaOCl generation was investigated.

CHAPTER TWO

LITERATURE REVIEW

2.1 OVERVIEW OF DISINFECTANTS

Disinfectants are chemical compounds that are designed to kill or inactivate microorganisms on inert surfaces or in wastewater. Disinfectants act by damaging or interfering with an organism's cell wall or digesting system. It's also a purification framework, and it's defined as the process of reducing the number of pathogenic germs on a surface using physical or chemical means[6].

Chlorine could be a chemical commonly utilized as an oxidizing specialist for drinking-water sanitization and wastewater treatment. Chlorine has been utilized for applications, such as the deactivation of pathogens in drinking water, swimming pool water, and wastewater, for the cleansing of family regions, and material dying. This oxidizing specialist can be utilized in numerous shapes: vaporous (e.g., Cl_2, ClO_2, etc.), fluid (e.g., NaOCl, HOCl, NH_2Cl, etc.), and strong (e.g., Ca $(OCl)_2$).

Hypochlorite-based items incorporate fluid (sodium hypochlorite), strong, or powdered (calcium hypochlorite) definitions. These concentrations break down within the water to create a weakened liquid chlorine concentration in which undissociated hypochlorous corrosive (HOCl) is energetic as the antimicrobial compound. Hypochlorite shows a wide range of antimicrobial movement and is compelling against a few common pathogens at different concentrations [5], [7], [8].

2.2 Compounds of chlorine

A few chlorine compounds are utilized to purify water since of their capacity to inactivate microorganisms. However, due to high reactivity, these compounds also react quickly with inorganic-reducing substances (ferrous iron, Fe^{++}, manganous manganese, Mn^{++}, nitrites, NO_2^-, hydrogensulfide, H_2S), ammonia, and organic material commonly currently in normal water, generating chlorine disinfection by-products (DBP). Chlorine reduces to chloride and it is lost as a disinfectant[3].

Most compounds containing chlorine are briefly portrayed:

Chloride ion (Cl^-): is the normal frame in which chlorine is display within the environment. It is the foremost inexhaustible ion in seawater (concentration changes between 18,000 and 25,000 mg/l), where it shapes, with sodium, sodium chloride (common salt). It also occurs in freshwater (0-200 mg/l) due to the dissolutionof chloride-containing minerals from the soil (mainly sodium

and calcium chloride). The chloride particle has no disinfectant activity itself but is the compound utilized to create molecular chlorine by electrolysis.

Molecular chlorine (Cl_2): could be a noxious solvent overwhelming gas (dissolvability is 7,160 mg/L at 1 atm and 20°C), with solid oxidizing control. In water, it hydrolyzes readily, producing hypochlorous acid and hydrochloric acid, as shown below:

$$Cl_2 + H_2O \rightarrow HOCl + H^+ + Cl^- \quad 2.1$$

Hypochlorous acid (HOCH) and hypochlorite ion (OCl^-): - hypochlorous acid is a weak acid, which partially dissociates in water, forming a hypochlorite ion. The concentration of the two species is determined by the dissociation constant (pKa = 7.537 at 25°C) and depends on the pH and the total concentration of chlorine.

$$HOCl + H^+ + OCl^- \quad 2.2$$

Hypochlorous acid is the genuine disinfectant specialist, whereas the hypochlorite particle has primarily an oxidizing activity. However, due to the equilibrium, as soon as hypochlorous acid is consumed, new acid is generatedfrom the hypochlorite ion, and therefore, hypochlorite ion is considered to have an indirect disinfecting action.

Hypochlorite (Sodium hypochlorite (NaOCl) and Calcium hypochlorite ($Ca(OCl)_2$): are salts of hypochlorous acid, which hydrolyze in water, producing hypochlorite ion (and consequently hypochlorous acid), as described below:

$$NaOCl + H_2O \rightarrow Na^+ + OH^- + HOCl \quad 2.3$$

$$Ca(OCl)_2C + 2H_2O \rightarrow Ca^{++} + 2OH^- + 2HOCl \quad 2.4$$

Sodium hypochlorite is commercially available at different concentrations (0.5-16% w/v available chlorine 1, but usually between 0.5 and 15%) and is commonly known as bleach. Calcium hypochlorite isgenerally sold in the dry form (bleaching powder) and contains between 20% and 70 % of available chlorine.

2.3 Commercial forms of chlorine

In general, chlorine is used in a variety of water systems around the world. Chlorine gas, NaOCl, and calcium hypochlorite are the three main commercial forms of chlorine that can be used for treatment.

2.3.1 Chlorine gas

Chlorine gas is produced mechanically as a by-product of the Kellner-Solvay process, which is used to make caustic soda by electrolysis of brine. The chlorine is then compressed above its vapor

pressure, resulting in a 450-fold reduction in specific volume. Finally, it is shipped and sold in tanks ofsizes varying between 45.5 kg and 1000 kg.

$$2\mathrm{NaCl} + 2\mathrm{H_2O} \rightarrow Cl_2 + H_2 + 2\mathrm{NaOH} \quad (2.5)$$

2.3.2 Sodium hypochlorite

The interaction of chlorine gas with caustic soda produces commercial sodium hypochlorite in 5 to 16 percent (w/v) accessible chlorine solutions. Domestic consumers typically use solutions ranging from 1% to 5%, whereas industrial customers typically use solutions ranging from 5% to 16 percent. On-site generation is also possible, as follows:

$$2\mathrm{NaOH} + \mathrm{Cl_2} \rightarrow \mathrm{NaOCl} + \mathrm{NaCl} + \mathrm{H_2O} \quad (2.6)$$

The liquid NaOCl was a yellowish-green color with a subtle chlorine odor. Surface purification, water disinfection, clothing bleaching, and odor elimination are all popular applications [2]. The salts of hydrochloric acid are known as hypochlorite (HOCl). Because of the acid's instability, most operators would rather work with extra stable hypochlorite solutions. The most common way to state a NaOCl solution is to use household bleach. In larger quantities, NaOCl is used to decolorize paper, materials, and mush. Various applications for NaOCl include using it as a middle-of-the-road chemical in the manufacture of ordinary chemicals, on a massive scale for water sanitization, and in pharmaceuticals and fungicides. It's also used as a pool disinfectant and germicide in the commercial world. As of late consumption, water must be cleaned and screened at some point to avoid maladies.

2.3.3 Calcium hypochlorite

Calcium hypochlorite is industrially manufactured accordingly:

$$2\mathrm{Ca(OH)_2} + 2\mathrm{Cl_2} \rightarrow \mathrm{Ca(OCl)_2} + \mathrm{CaCl_2} + 2\mathrm{H_2O} \quad (2.7)$$

The final product is a white dry solid, in the form of powder, granules, compressed tablets, or pellets, with available chlorine content up to 70 % (wt.), but usually, for water disinfection purposes, between 25 and 28%. In the event that 100 g of powder at 25% accessible chlorine is included in 1 liter of unadulterated water, an arrangement with 25 g accessible chlorine per liter is shaped (2.5 % arrangement).

Among these chemicals, chlorine gas is the cheapest, in terms of cost per unit of accessible chlorine. Hence, it has been the winning framework received for water sanitization within the USA and Western Europe. Be that as it may, the dangers related to profoundly poisonous chlorine transportation and dealing with have decided, in a few cases, a predisposition for sodium

hypochlorite, in spite of the truth, its cost ranges from 150 to 200 % of the fetched of compressed chlorine gas.

Calcium hypochlorite is the foremost costly frame among the three fundamental chlorine disinfectants. It tends to crystallize, clogging metering pumps, channeling, and valves, and loses accessible quality on the off chance that not legitimately put away in a cool and dry area. Hence, its utility is restricted to little establishments, where chlorine gas and sodium hypochlorite frameworks are an inapplicable sense of their complexity. Because of these characteristics, sodium hypochlorite might be considered the most adequate form of chlorine for implementing point-of-use disinfection.

2.4 Generation of sodium hypochlorite from sodium chloride solution

A process that breaks down a chemical compound into its components or produces a new compound by the activity of an electrical current. The electrical current is passed through an electrolytic cell and oxidation /reduction reactions happen at the electrodes. Electrolysis of sodium chloride solution (seawater) requiresthe passage of direct current between an anode (positive pole) and a cathode (negative pole) to separate salt and water into their basic elements. Chlorine is created at the anode promptly goes through chemical reactions to make sodium hypochlorite. Hydrogen and hydroxide are shaped at the cathode, the hydrogen forms gas and the hydroxide help within the arrangement of sodium hypochlorite with zero pH move. The electrolyzer system is required to produce hypochlorite with a chlorine content of 7 to 9 g Cl2/l (0.7 to 0.9 percent w/v) in most cases[9], [10].

In comparison to bought and passed-on chlorine, OSG NaOCl could be a viable alternative. The invention was based on a NaCl electrolysis system that could deliver changeable NaOCl concentrations (perfect chlorine gas). The following are three of the best accessible advancements that should be evaluated for this application:

a. Direct electrolysis, which produces 0.8 percent fluid NaOCl by weight.
b. Electrolysis of membrane cells, yielding 12.5 percent fluid NaOCl by weight.
c. Electrolysis of membrane cells, which produces vaporized chlorine and caustic soda.

On-site electrochlorination (OSEC) is a technology for producing NaOCl as an effective disinfectant from the electrolysis of water and salt solution [11]–[13]. NaOCl OSG is a critical technique based on logical principles that have been used for decades. It uses NaCl that has condensed in water and delivers NaOCl and other oxidant species via an electrical current with a

low DC voltage. The OS-generated chlorine has a variety of applications in the commercial and mechanical worlds, including sanitization of swimming pools and cooling towers, among other things.

The most notable application of OSGs development is the purification of municipal drinking water. Many water districts have seen the benefits of using OSGs to replace traditional chlorine transport methods including chlorine gas, NaOCl concentrate, and bulk calcium hypochlorite with OSG systems. By running a NaCl arrangement through electrolytic cells while applying an electrical current, OS-generated NaOCl is formed.

Various communities have moved to use OSGs for filtering water systems due to the potential profits of the development; these benefits include high-quality sanitization, greener forms, and vital budgetary venture reserves. Despite its costs, OSGs have a low-risk level and provide security for directors because the concentration of the created hypochlorite is between 0.5 percent and 0.8 percent, which is below the risky classification threshold of 1 percent. This low concentration gives the hypochlorite more soundness and less scaling of mixture lines and fittings due to its low pH[14], [15].

Since the early 1970s, the OSG of NaOCl has been used for water purification in the United States. A few applications are now using NaOCl generation units to create a safe, consistent, and temperate disinfection for water. A proprietary system's usual salt consumption rate is 3kg of salt per kg of equivalent chlorine. Within the electrolysis cell is a matrix of plate-type electrodes made of metals resistant to the chemically hostile environment present during electrolysis.

The electrode reactions for the product are:

At anode: $2Cl^- - 2e^- \rightarrow Cl_2$ 2. 8

At cathode: $2H_2O + 2e^- \rightarrow 2OH^- + H_2$ 2. 9

Overall: $Cl^- + H_2O \rightarrow OCl^- + H_2$ 2. 10

The basic by and a large representation of the electrochemical reaction is:

Salt + Water + Energy ⟶ Sodium hypochlorite + Hydrogen

$NaCl + H_2O + \text{Energy} \rightarrow NaOCl + H_2$ 2. 11

2.5 Characteristics of sodium hypochlorite

Commercial bleach uses NaOCl as a dynamic fixer. It's commonly sold as a solution in concentrations of 6%, 12%, and 15%. Its somewhat short shelf life is influenced by a variety of conditions, including light, temperature, vibration, and concentration. Increasing these variables

will have a negative impact on the hypochlorite's shelf life. To maintain quality and soundness, it must also be stored in a specific condition.

All of these well-thought-out vital actions for excellent hypochlorite management include storing the container in a cold place and not agitating it during transportation. Higher concentration solutions are more likely to get contaminated sooner than lower concentration solutions. Higher concentration solutions tend to deteriorate faster than lower concentration solutions. However, solutions with a lower concentration require greater storage space within the region. Furthermore, if a solution with a 12 percent concentration rate is compared to a solution with a 6 percent concentration rate, the change in the stronger solution will be more obvious than it is in the lower concentration arrangement, but because it breaks down, the response will reduce as well. When 12 percent reaches the 6 percent level, it breaks down in the same way as the one that started at 6 percent did on the first day[16].

Because of its high quality as an oxidizing specialist, NaOCl is regarded as a wonderful disinfectant. Apart from other disinfection alternatives, NaOCl is regarded as one of the alternatives for sanitization. It has a high destructive character and a proclivity to rot over time while in use. Chlorine removes electrons from the outer shells of living organisms, weakening the structure until the organism is no longer alive. Free chlorine is the disinfectant used in most municipal water treatment facilities. It is made by mixing NaOCl with water to form hypochlorite (OCl^- ion), which is also known as free chlorine.

The anodic oxidation of pigment particles and phenols in the wastewater was the reason for using hypochlorite made on-site[17], [18]. Using NaOCl as a disinfectant has several advantages, including safe handling, ease of dosing, reduced shipping, low weakening rate, and no residual spewing. For eliminating stains from garments, NaOCl at a concentration of 5% is employed as a bleach.

1-5 dilutions of home bleach will kill or control the majority of microorganisms. This bleach is also used to clean surfaces in hospitals. In other words, NaOCl is one of the most important disinfectants for drinking water, with one litter disinfecting 4000 liters. It is also used to sterilize swimming pools and eliminate cyanide wastes. One of the most important features of NaOCl is that microorganisms have no resistance to it. Because high concentrations of NaOCl are considered harmful, most bleach solutions on the market contain concentrations of 3-5 percent sodium hypochlorite electrolysis.

NaOCl is well-known today and is commonly used for a variety of plants, with some of them producing hypochlorite using undivided electrolytic cells and coordinated electrolysis of weak brine or table salt. The generation capacity necessary should be considered when selecting a cell. After generation, the quality of NaOCl continues to deteriorate. The main issue with using NaOCl as a disinfectant is that it degrades. In a clear preparation, the concentration of hypochlorous acid and hypochlorite ion in water decreases with time, depending on temperature, light, pH, the initial concentration of accessible chlorine and natural matter, and the proximity of metal ions. To increase NaOCl stability in the solution, the storage strategy should include keeping the pH between 11.5 and 13, keeping the NaOCl content low, keeping the temperature around 303K, keeping the solutions free of graphite and metallic particles, and providing light-resistant holders. [2], [19].

The measured crystallized salts are electrolyzed by the electrolysis frameworks within the handle of OSG of NaOCl. The electrolysis cell was designed to handle a low brine stream rate with limited dispersion between cathodes, resulting in sodium hypochlorite concentrations of less than 1%. NaOCl OSG could be a more affordable and safer alternative to chlorination. It is regarded as one of the more reasonable options because it can be produced with the volume and grade of disinfection required in the time frame specified. The capital has taken a toll on technology, and the most significant shortcomings of these outlines are the amount of energy obtained and the presence of energy. Furthermore, the electrode's replacement could be a major impediment because it is made using expensive materials such as titanium-coated with iridium, ruthenium, titanium, or platinum oxides, mixed in prohibitive units with a typical shelf life of 5-8 years. The major focuses of the systems in partitioned are their simplicity and ease of upkeep and operation, which are deemed unpretentious.

2.6 Advantage and Disadvantage of electrolysis processes

The degree of reagents generation in a pollutant treatment reactor can be productivity attached to the request forced by the degree of contamination of the process stream, and the degree of reagents generation can be productivity attached to the request forced by the degree of contamination of the process stream[3], [16].

Electrolysis forms, in general, provide various specific vital characteristics in comparison to other advancements. The following is a list of some of the advantages of electrochemical techniques to contamination control:

a. **Environmentally friendly:** the electron is the primary reagent used, and it is a clean reagent, thus there is usually no need to count other chemicals.
b. **Versatility:** electrochemical forms based on coordinate or roundabout oxidation and reduction can produce neutral, positively or negatively charged inorganic, organic, or biological species. They can also work with solids and liquids, and they can simulate the era of quickens, vaporous species, pH shifts, and charge neutralization. Things that result from the electrolysis of poisons regularly are unquestionably vital. A wide range of reactor and cathode materials, geometries, and settings are available. Because the adjustments were modest, the same reactor could be used for a variety of electrochemical reactions. Electrochemical techniques also advocate the idea of using chemicals to generate energy (e.g., for water sanitization). Finally, liquid quantities ranging from microliters to liters can be processed.
c. **Energy efficiency:** electrochemical forms often have lower temperature and weight requirements than non-electrochemical counterparts (e.g., burning, supercritical oxidation). The connected possibilities can be controlled, and electrodes and cells can be designed to reduce control errors caused by insufficient current flow and voltage dips.
d. **Safety**: electrochemical forms are secure since the mellow conditions are more often than not utilized and the little amount and harmless nature of the included chemicals.
e. **Choosiness:** in many circumstances, the connected potential can be regulated to a certain attack, bonds, and thereby ambiguity the creation of by-products.
f. **Computerization**: The electrical variables used in electrochemical shapes are well-suited to facilitating data acquisition, preparation computerization, and control.
g. **Cost-effectiveness**: the essential equipment and activities are typically straightforward and, if well set out, may be made relatively inexpensive.

Despite these preferences, there are a few obstacles to overcome:

a. Electrode materials are susceptible to crumbling, complexation, oxidation, wear, and inactivation.
b. The majority of electrochemical forms are performed in fluid arrangements, which are soluble and disintegration (water oxidation/ lessening) is frequently difficult to avoid, resulting in essential waste.
c. Gases produced by excess decay (hydrogen and oxygen) may form hazardous combinations.
d. The best terminal materials in terms of toughness and torpidity integrate valuable metals on a

regular basis, increasing the expenses.

e. Power is prohibitively expensive in many areas.
f. There's a chance you'll lose a lot of money in the beginning.
g. The lack of knowledge or comprehension of electrochemistry may be the most significant barrier to its application.

2.7 Electrode Materials for Electrolysis

For electrolysis planning, issues such as fetching, openness, consistent quality at the needed feasible outcomes, selectivity, composition, and pH of the reaction medium, nature of intermediate substances and things, and common compatibility must be considered. Since most electrodes are lucrative since it was an obligatory run of potential and pH, care should be made in a true blue guarantee.

The electrolysis process' terminal materials change dramatically. Stainless steel, copper, graphite, carbon, and reticulated vitreous carbon are common cathode materials, while platinized titanium or niobium, tantalum, graphite, carbon, metal oxides, silver, copper, nickel, dimensionally stable anodes (DSA), and combinations of these materials are common anode materials[4].

2.7.1 Types of anode materials

Electrode materials are divided into four types based on their receptive response when exposed to an electric current.

1. Metals and alloys that are fully consumable:

Metals like magnesium, zinc, and aluminum, as well as media, are used to break up under-connected currents. The link between current thickness (A/cm^2) and the total amount of metal that enters into the solution can be depicted using Faraday's law. Electrode materials can be classified into four diverse sorts based on their receptive behavior beneath the electric current application. [16]:

$$W = \frac{I \times t \times M}{Z \times F} \qquad 2.12$$

Where, I= current density (A/cm^2), w = metal dissolving (g), M = molecular weight of the metal, F = Faraday's Constant (96500) and Z = number of electrons involved in the oxidation/reduction reaction.

2. Bulk non-metallic conductors:

Graphite and magnetite anodes are examples of bulk non-metallic conductors. The basic reaction

at the anode is the development of chlorine and oxygen gas. Cathode degeneration occurs as a result of a chemical reaction between the electrolyte and the anode. The rate of anode deterioration for this type of anode is around a hundred times lower than that of completely consumable metal anodes**.**

3. Metals and alloys that are partially inactive:

Lead and tall silicon media amalgam anodes are consolidated in this category. The method of an electrically conductivity thick oxide film over the metal surface is used to finish resistance to the anode's assist breakdown. Micropores or metal-combination particles embedded inside the oxide film provide conductivity through this type of thick oxide sheet. By a factor of ten to one hundred, the rate of deterioration is slower than that of fully consumable metal anodes.

4. Completely inactive anodes:

Platinum is the most common metal used in this type of anode. An electrically conductive thin film a few atomic layers thick forms on the surface of platinum anodes. The film is conductive as well as non-reactive. Gas progression is the reaction at the anode. In addition to this category, the DSA^{TM} (Dimensionally Consistent Anode) is made of titanium with a thin film coating of RuO_2 and IrO_3. For the most part, the residual rate of deterioration is one-millionth of that of completely consumable metal anodes.

2.7.2 Characteristics of Specific Electrode Materials

The following are a few important subtle aspects of various anode materials used in electrochemical forms[3]:

a. **Ferrous metals:** This electrode material has a real problem with extremely high utilization frequency. The potential degradation rate is almost 1 kilogram per kiloamp-hour. Because of its simple and inexpensive openness as scrap material, this type of anode can be explored for electrochemical forms.

b. **Aluminum**: - This solves the problem of ferrous metals quickly corroding. Even though more chemically liberal, degeneration occurs. For cost considerations, the use of aluminum in electrochemical applications isn't very frequent.

c. **Magnetite**: - a common mineral formed by the accumulation of media and oxygen in a non-stoichiometric precious stone cross-section. Dissolved and delivered at 1540°C. The disadvantage of magnetite anodes is that they are mechanically sensitive and have lower electrical conductivity than metal anodes. When compared to ordinary metal anodes, the rate

of deterioration is almost 2×10^{-4}kg/amp-hour. this offers the quick deterioration issue that ferrous metals have. The deterioration things indeed even though more chemically liberal. The utilization of aluminum in electrochemical applications isn't outstandingly common for financial reasons. The oxygen generation over-voltage of magnetite is said to be lower than that of chlorine gas generation, which could be useful in reducing chlorine gas by-products.

d. **Carbon**: - Carbon, in the form of graphite, is one of the most often used anode materials. Because graphite is very permeable, gas production inside the pores at high current densities can cause anode mechanical degradation. The degradation reactions are chemical reactions with the electrolyte, not oxidation events. The oxidation of carbon into CO_2 is the dominant process in low chloride, molecular conditions. Anode crumbling in seawater is induced by more complex processes involving chlorine gas and chloride particles. The pace of crumbling is proportional to the existing thickness. The reported degradation rate in seawater is roughly 0.005kg/kiloamp-hour at current densities of 4-10amps/m^2.

   Surface oxygenated useful bunches in carbonaceous materials (carbon felt, reticulated vitreous carbon, and sparkling carbon) are known to enable electron trade with normal substances and are safe from a typical point of view. In any event, high-temperature carbonization and graphitization phases are required to transform polymer compositions into conductive carbon cathodes, making composite conductive carbon materials expensive to produce. The erosion resistance of three-dimensional carbonaceous cathodes made primarily of graphitized unclear carbon and graphite felts has been demonstrated (attack by radical species or intercalation of particles between its basal planes causing breaks). The graphitization ensures adequate responsiveness update, while the indistinct structure allows for planar cross-linking.

e. **Lead and lead amalgams: -** When used as an anode material, lead has complicated electrochemistry. At certain current densities, clean lead disintegrates into Pb^{+2} particles. Under these conditions, the measured crumbling rate for perfect lead is 5kg/kiloamp-hour. Lead chloride ($PbCl_2$) forms at the surface of saltwater with greater current densities. Because of the IR voltage loss that occurs over the $PbCl_2$ film, $PbCl_2$ may be a separator that considerably reduces control adequacy. In any scenario, an epitaxial film layer of lead oxide (PbO_2) can be generated over the surface of the anode under perfect conditions. PbO_2 is an electrical conductor that can protect the surface against both weakening and the formation of $PbCl_2$. A PbO_2 secure lead anode can have a deterioration rate as moo as 0.001kg/kiloamp-

hour. By alloying and embedding surface micro-electrode clusters, lead can be conditioned to form oxide films.

When silver and antimony are alloyed together, the result is a surface with localized silver grains that shape nucleation targets for PbO_2 to form and spread across the entire surface. Lead anodes with platinum micro-electrodes (0.5mm remove over) inserted in the surface have shown a comparable effect. By embedding magnetite particles in lead, a lower-cost process for embedding micro-electrodes was completed. A handful of manufacturers considered surface passivated lead amalgam anodes to be eight times more down to earth than silicon steel.

f. **Platinum**: - Platinum cathodes resist corrosion not only because of their respectable metal status in the galvanic process but also because they form an electrically conductive passivating thin surface plate. Platinum anodes deteriorate at a rate of roughly 2×10^{-6} kg per kiloamp-hour in seawater. Platinum's high cost, combined with the fact that it has a particularly low rate of wear, has led to it being used as a thin-film plating over less expensive metals. Platinum and titanium have been shown to be the most cost-effective combo. Other metals that have been successfully used as substrate metals for the production of platinum anodes include niobium and tantalum.

When used as an anode, titanium has the basic properties of base metal in that it forms a non-conducting oxide over any cracks within the platinum thin plate. This substrate metal's anodic inert oxide handling is particularly important for platinum's compelling use as a lean plate anode. Any cracks inside the platinum thin plate would cause a concentration of current flow to break inside the thin plate, leading to the rapid erosion of the less reputable magnesium substrate multiplying from that point if platinum was coupled over magnesium for the outline. By electrification, platinum is usually connected with the substrate metal.

Platinum is most commonly associated with the substrate metal by electro-deposition in a plating shower. Coatings ranging from 1 to 15 microns are often used. If a long working life is to be maintained, these platinum thin plates have a few limits in terms of operating circumstances. The electrical swell in the control supply couples with the passivating lean plate, eroding the anodes quickly. A well-filtered DC supply is required. Furthermore, using a platinum-coated terminal as a cathode results in rapid platinum coating failure due to hydrogen entry into the titanium substrate. Any facilitated metallic contact between magnesium and a platinum-coated electrode in an electrolyte will result in the platinum cathode acting as a

cathode inside the platinum cathode. This also prevents platinum-coated anodes from acting as both an anode and a cathode when used as a substitute for extreme.

2.8 Literature Review on Electrolysis Generation of Sodium Hypochlorite

Rengarajan, et al. (1996) investigated the effects of current density, temperature, and NaCl concentration on the productivity of producing NaOCl, as well as the proportion of NaOCl and chloride and the vitality consumed. In addition, tests were conducted to investigate the effect of temperature in the range of 303-333K on the formation of NaOCl. The studies revealed that the highest current productivity for the formation of NaOCl was achieved at a current thickness of 5-10A.dm^2, 313K, and a NaCl concentration of 40-50gpl. With the passage of time, the present productivity for the production of NaCl has decreased[20].

Morganti, (2002), the ponder, conducted a series of tests in Nepal to assess and consider the execution of the generating process as well as the quality of the NaOCl solution developed for household water sanitization. The specialized and organizational criteria that point to a successful NaOCl generation handle commissioning have been identified. With the true-blue producing and adequate capacity conditions maintained, the average rot rate will be 230mg/l (0.023 percent) per month throughout the required three and half months, according to the NaOCl solution with 0.56 percent concentration given by the method handle. Distant better; A much better; A higher; A stronger; An improved and A distant better decay rate (0.10% per month) can happen with coincidental defilement with the iron, by utilizing of an iron nozzle, along these lines supplanted by a plastic spigot.

As a result, it is critical to ensure that the process is free of media contamination in order to maintain enough rack life for the process. Alkalinization of the solution is also indicated to increase its strength. “Tap water chlorine demand in Kathmandu was discovered to be approximately 0.5mg/l, and thus a NaOCl estimation of three drops per liter (roughly 1.5ml/l) illustrated palatable to supply a free remaining chlorine level over 0.2mg/l,” he added. Analyst examination of the genuine chlorine demand in a few seasons and various regions of the country is recommended in any instance[15].

Hooper, (2005) investigated the advancements made within the equipment used for producing 0.8 percent NaOCl on-site (at the point of use), as well as the benefits and drawbacks associated with this method. He concluded that producing NaOCl on-site can be a commonsense, cost-effective,

and secure alternative to 12.5 percent commercial survey NaOCl and chlorine tablets, among other cleaning options that include chlorine gas, commercial-grade 12.5 percent, calcium hypochlorite tablets, and an OSG of NaOCl. It is more expensive per kilogram than chlorine gas, but if security concerns are taken into account, it can still be a cost-effective option[21].

Rezeq, Mohammed Rashad Al Dalou (2017) looked at the challenges surrounding brine or wastewater generated during the desalination process. They've been throwing light on the business opportunities associated with brine squander by using the brine for on-site NaOCl generation. The experiments resulted in the creation of NaOCl, which was then examined using the HAACH tables. Hydrogen was also given to the anode, which was checked by a start. The cathode terminal was stocked with NaCl, whereas the foot was stocked with brine salts (carbonates and hydroxides). The total amount of hypochlorite formed appears to be dependent on the reaction time and the DC voltage applied[4].

Abdul-Wahab and Al-Weshai (2009) looked at the challenges surrounding brine or wastewater produced during the desalination process. They've been throwing light on the business opportunities associated with brine squander by using the brine for NaOCl OSG. The tests yielded NaOCl, which was reviewed by HAACH tables. In addition, hydrogen was created at the anode and inspected by a start. At the cathode electrode, NaCl was accumulated, while brine salts (carbonates and hydroxides) were retained at the foot. They also demonstrated that the total amount of hypochlorite supplied is dependent on the reaction time and the DC voltage applied[22].

For the OSG of NaOCl, Asokan and Subramanian (2009) set up a tank electrolyzer. The perfect current thickness, electrolysis time, working cell temperature, and hold up or current concentration were all tested. As a result, it has been discovered that as current thickness is increased, hypochlorite formation increases as well, but cell temperature increases as well. NaOCl tends to chemically break down to sodium chlorate at temperatures over 308K.

$$3NaOCl \rightarrow NaClO_3 + 2NaCl \qquad 2.13$$

The concentration of NaOCl rises to 50Ma.cm^2, however as the current density grows, the concentration falls as the temperature rises. The stream rate, on the other hand, has been demonstrated to be connected to both the NaOCl concentration and the current proficiency. Increased stream rate slows the degradation process while simultaneously lowering the NaOCl concentration. The optimal stream rate is 3.6Lh^{-1}, which results in the highest concentration of hypochlorite at a reasonable current capacity. They discovered that increasing the temperature

reduces NaOCl concentration and the current viability of the response by evaluating the ideal working temperature. Where the electrolyzer must be kept at the same temperature as the surrounding environment in order to produce a very high NaOCl concentration of roughly 8gpl[2].

Nasser Abu Ghalwa et al. (2012) looked into the NaOCl period. As a result, it appears that NaCl is the most productive conductive electrolyte for producing NaOCl. At the most extreme hypochlorite concentration, the optimal current density is $1A.cm^{-2}$. The electrolyzer must be kept at the same temperature as the surrounding environment (10°C) in order to get the highest possible NaOCl concentration. The effect of temperature on the creation of NaOCl revealed that increasing the time of electrolysis to 90 and 60 minutes, respectively, increases hypochlorite generation for Pb/PbO_2 and C/PbO_2 terminals. Aside from that, when evaluating the effect of NaCl concentration, it was discovered that a concentration of 20 gpl NaCl is deemed ideal[17].

Chakrabarti et al. (2012) investigated the OSE generation of NaOCl disinfectant for a seawater control plant. They took into account the effects of various electrode types, current densities, electrode gap, and electrode surface zone. The results almost showed up that titanium cathode coated with DSA outlined astounding relentlessness when compared to other attempted cathode materials, with NaOCl generation reaching perfect regard of around 6315mg/l; current thickness of $72.4mA/cm^2$ was found to be promising regard for a capable generation of NaOCl and an anode to cathode surface extend the proportion of solidarity was found to be. A 7cm inter-electrode spacing was discovered to be better[23].

2.9 Factor affecting electrolysis sodium hypochlorite generation

2.9.1 Sodium chloride (NaCl) concentration

NaCl is the most basic salt in water, and it can be found in crystalline form in nature. In numerous studies, researchers [4] found that NaCl was the best electrolyte for the production of NaOCl. They concluded that the most important high concentration of NaCl for the formation of NaOCl was 40-50gpl, with a high current proficiency (56.3-59%) and essentiality usage compared to (4.8KWh kg^{-1}). A high concentration of NaCl can result in a chlorate solution. Low concentrations reduce current productivity by driving oxygen progressions through water output, and the chlorate arrangement was 7.6 times higher than expected.

2.9.2 Electric potential

The framework's most important constraint is electric potential; the higher the cell potential, the

greater the sum of electric current that travels through the framework and the greater the current thickness. The electric potential has a considerable impact on the current density of the electrolysis system.

2.9.3 Electrode gap/inter-electrode spacing

The ions must travel through the crack between the anodes to reach the electrolyte. The inter-electrode spacing is a critical parameter in the electrochemical production of NaOCl. Increased electrode spacing lowers the capital cost of generation but lowers the efficiency of generation. As a result, optimizing this value is crucial. As a function of inter-electrode distance, the formation of sodium hypochlorite varies with time. As the inter-electrode separation shrank, the rate of NaOCl production increased.

The sodium (Na^+) and chloride (Cl^-) ions are evenly distributed throughout the brine solution where electrolysis is taking place. The sodium (Na^+) and chloride (Cl^-) ions flow towards the oppositely charged electrodes when two oppositely charged electrodes are immersed in the solution. To ensure a critical uniform distance between the electrodes, they are separated consistently. In the case of NaOCl production, this critical distance is very small. This distance must be maintained for optimal performance and rapid hydrogen removal from the cell.

2.9.4 Current Density

Current thickness is used to assess the pace of an electrochemical reaction. A current density is defined as the amount of current that travels through one square meter of the electrode. It's a crucial parameter in electrolysis because it's the only one that can be directly manipulated. With increasing current density, the rate of sodium hypochlorite production increases.

Due to the requirement for a good current thickness to synthesize NaOCl, the current thickness has a fundamental effect on the formation of NaOCl. In most electrochemical processes, the chemical change is determined by the total current transmitted, according to Faraday's law of electrolysis. In industry, the current passed is measured in terms of current thickness, which indicates how much current is passed per unit electrode. They came to the conclusion that as current thickness increases, so does NaOCl production and the temperature of the cell. NaOCl tends to chemically break down to sodium chlorate when the temperature rises over 308K.

2.9.5 Electrode material

The electrode material may play a significant role in the efficiency with which NaOCl is produced. Various studies have looked into the relationship between the type of terminal and the total amount

of NaOCl produced. The true success of electrochemical innovation is the fitting assurance of electrode material. The cathode fabric must have a viable electrocatalytic activity and selectivity since the anode fabric will influence the course of action and ideals of the NaOCl period. The anode material's durability under open-circuit circumstances, as well as its accessibility at a reasonable cost, are important factors to consider when selecting an anode material.

For the most part, a compromise between activity, selectivity, and toll must be made in order to ensure the fabric's integrity. A cloth with high soundness and moo electrocatalytic properties is often more curious than one with low chemical permanency and a high electrocatalyst.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials and Chemicals

To make sodium hypochlorite from sodium chloride, the following materials and chemicals were used in the electrolysis process.

Table 3. 1The following materials and chemicals were used;

Materials/apparatus	**Chemicals/reagents**
Magnetic stirrer	Sodium chloride (99.9%)
Digital balance	Potassium iodide
Flasks 1000 ml	Distilled water
Conical flask 500ml	Sodium thiosulfate
Cylinders 100 ml and 250ml	Acetic acid, glacial
Plastic sample bottles 100 ml	Starch indicator
Burette and Burette stand	Ethanol
Digital pH meter	Acetone
Double beam UV-Vis Spectrophotometer	
Electrodes (Ti/IrO_2 and Stainless-steel plate)	
DC supply system	

3.2 Laboratory-Scale Batch Electrolysis Unit

An electrolysis unit was used in this experiment (Figure 3.1). This experiment was carried out in a batch mode with overwhelming agitation in a laboratory-scale undivided cell. This unit consists of an electrolysis chamber made of rectangular PVC (which can hold up to 1 liter of electrolyte) and a direct current (DC) control supply (with a typical voltage range of 3-9 V). The tests were conducted in a 1000ml rectangular PVC cell with IrO_2/Ti kind terminals as anodes and stainless-steel plates as cathodes. The current inlet can be controlled using the DC control. Electrode gaps were drawn to ensure that the electrode plates were flush with one another, maximizing ion exchange between terminals. The space between the electrodes can range from 1 to 6 cm. During the electrolysis process, this region permits the electrodes to remain fixed and preserve the separate space.

Figure 3. 1 Experimental setup

3.3 Electrode Preparation

Titanium and stainless steel were used in this project. The anode materials were chosen based on several parameters, including improved electrical conductivity, erosion resistivity, degradation resistivity, the ability to catalyze electrode reactions, reasonableness, and market openness[24]. The electrodes were immersed in 1% acetone for 3 hours. At that moment, the electrodes were rinsed with distilled water. Following that, the cathodes were immersed in 1% ethanol for 10 minutes. The cathodes were then cleaned three times with purified water before being dried for 24 hours. Before the electrolysis experiments, the cathodes were placed in a desiccator. The anode was made of titanium, and the cathode was made of stainless steel.

3.4 Electrolysis Experiment

Sodium chloride (Blulux Research Facilities Ltd – 121005, India) was weakened at different concentrations with purified water (10, 30, and 50g NaCl). At that time, the weaker salt solution was transported to the electrolysis chambers. The electrodes for the cathode and anode were

collected and fitted into the electrolysis chambers. The cathode was made of stainless steel, and the anode was made of titanium (Ti/IrO_2). The electrodes were coupled to three different channel voltages (3, 6, and 9 V). During the electrolysis preparation, plugs were used to keep the wires connecting the terminals and the DC control supply stationery. The gaps were also well-organized to keep the anode and cathode electrodes facing each other and from moving. The electrolysis procedure was carried out under certain electrolyzing conditions (10, 50, and 90 min). Sodium hypochlorite was recovered from the chambers once the electrolysis handle was removed.

The following conditions were used for electrolysis: a sodium chloride concentration of 10-50g/l, an electric potential of 3-9V, a time of electrolysis ranging from 10 to 90 minutes, and a distance between the two terminals (anode and cathode) ranging from 1-6cm. With the use of an indoor regulator water bath, the electrolyte was brought to room temperature (200°C).

3.5 Experimental design

The following variables were used in the experiment to see if the generation of NaOCl was viable under the following conditions: change in contact time, change in the interspace between electrodes, and change in electric potential.

In this study, a Box–Behnken (BBD) test plan for the response surface method (RSM) was connected to the Design-Expert program (Stat-Ease version 12). As a result, BBD is sufficient for determining the fitted model's measurable validity as well as the model's lack of fit[25]–[28]. A total of 29 tests were conducted for this four-factorial BBD (24 tests (each factor on three levels) plus five central tests).

As shown in table 3.2, the variables in this experiment were NaCl concentrations (10, 30, and 50g), an electric potential (3, 6, and 9 V), anode crevice (1, 3.5, and 6cm), and electrolysis periods (10, 50, and 90 minutes).

Table 3. 2 Box–Behnken plan experiment variables and levels.

Factor	Name	Units	Coded Low (-1)	Coded High (+1)	Mean	Std. Dev.
A	NaCl concentration	g	10.00	50.00	30.00	13.09
B	Electric potential	volt	3.00	9.00	6.00	1.96
C	Electrode gap	cm	1.00	6.00	3.50	1.64
D	Electrolysis time	min	10.00	90.00	50.00	26.19

3.6 Analytical methods

3.6.1 Iodometric methods

Iodometric titration was used to determine the concentration of sodium hypochlorite in this study. This technique is more precise than the colorimetric strategy when the remaining chlorine concentration exceeds 1mg/l, but it is less accurate at lower concentrations. The method of assessing and testing, which is based on established methodologies for water and wastewater analysis[29], [30].

a. Preparation of reagents

Transfer 1.581g sodium thiosulphate to the beaker and dissolve it in 100ml bubbled purified water. Fill a regular flask halfway with water and make it up to 1000ml.

b. Sample analysis

1. Fill the burette with $Na_2S_2O_3$ after rinsing it with the substance.
2. Secure the burette in place on the stand.
3. Using a conical flask to collect 20ml of a specific test.
4. Adding 2 mL acetic acid to the solution to lower the pH to 3 - 4.
5. Dilute 1g potassium iodide (KI) by combing it all together with a mixing pole.
6. Titrate the arrangement rapidly with normal $Na_2S_2O_3$ until the yellow hue of released iodine is almost completely blurred (pale yellow color appears).
7. Adding 1ml of starch arrangement and proceed with the titration until the blue color disappears.
8. Make a mental note of the burette perusing (to know the volume of $Na_2S_2O_3$ devoured). Calculating the residual chlorine using iodometric methods at that point;

$$\text{Residual chlorine(g/L)} = \frac{V_{Na_2S_2O_3} \times N \times eq_{wt\ of\ Cl_2}}{V_{sample}} \qquad 3.1$$

The iodide ion is added to the solution in the form of potassium iodide (KI), and the resulting iodide (I_2) was titrated using a solution of sodium thiosulfate ($Na_2S_2O_3$) in the presence of starch as an indicator.

The examination takes put in a series of steps:

$$ClO^- + 2I^- + 2H^+ \rightarrow Cl^- + I_2 + H_2O$$

$$I_{2(aq)} + I^-_{(aq)} \rightarrow I^-_{3(aq)}$$

A conventional arrangement of thiosulfate particles was used to titrate the triiodide, which reduces the iodine back to iodide ions:

$$I_{3(aq)}^{-} + 2S_2O_3^{2-} \rightarrow 3I_{(aq)}^{-} + S_4O_{6(aq)}^{2-}$$

3.6.2 Spectrophotometric methods

Is a method for determining how much light a chemical substance absorbs by measuring the intensity of light as it passes through a sample solution. The basic concept is that any compound absorbs or transmits light across a specific wavelength range. The sample absorbance at 292nm, which is the wavelength that corresponds to the maximal absorbance in the visible region, was measured using a Spectro UV-VIS 3200 double beam pc spectrophotometer to determine the degree of the NaOCl concentration that had happened during electrolysis generation[31].

Experimental procedure

- Before running any sample, the spectrophotometer was turned on for at least 15 minutes to warm up.
- The cuvettes were cleaned and distilled water was washed completely.
- The test's legitimate volume is packed into the cuvette.
- A control solution (blank solution) was made using simply a chemical solvent in which the examined solute was dissolved.
- To reduce measuring error, the outside of the cuvette was cleaned before being placed into the spectrophotometer.

When running the experiment:

- To evaluate sodium hypochlorite, a wavelength of 292nm was chosen, which is in the preferred range of (200-400nm). The testing is more effective when a single wavelength of light is used (monochromatic Color).
- A blank solution was used to calibrate the equipment.
- The blank was removed once the machine was calibrated, and the calibration was verified to ensure it was at 0. At a wavelength of 292nm, the absorbance of NaOCl was measured.
- The test was repeated with each wavelength of light to determine which wavelength was the best.
- The graph was then plotted using the absorbance value Vs wavelength.

CHAPTER FOUR

RESULT AND DISCUSSION

4.1 Introduction

This chapter examines, considers, and concludes the test results. Looking at numerous aspects that influence the generation process of NaOCl, the generation of NaOCl using NaCl was chosen. NaCl content, electric potential (voltage), electrode gap, and electrolysis time are the variables.

4.2 UV/Vis Analysis (UV-Visible Absorption Spectrum)

Figure 4.1 shows the UV-vis absorption spectra for a test between 200 and 400nm. The peak of the test is 292nm. As a result, this data indicates that sodium hypochlorite is activated in the UV area. It is clear that when the particle size decreases, the peaks get sharper, and the absorbance increases.

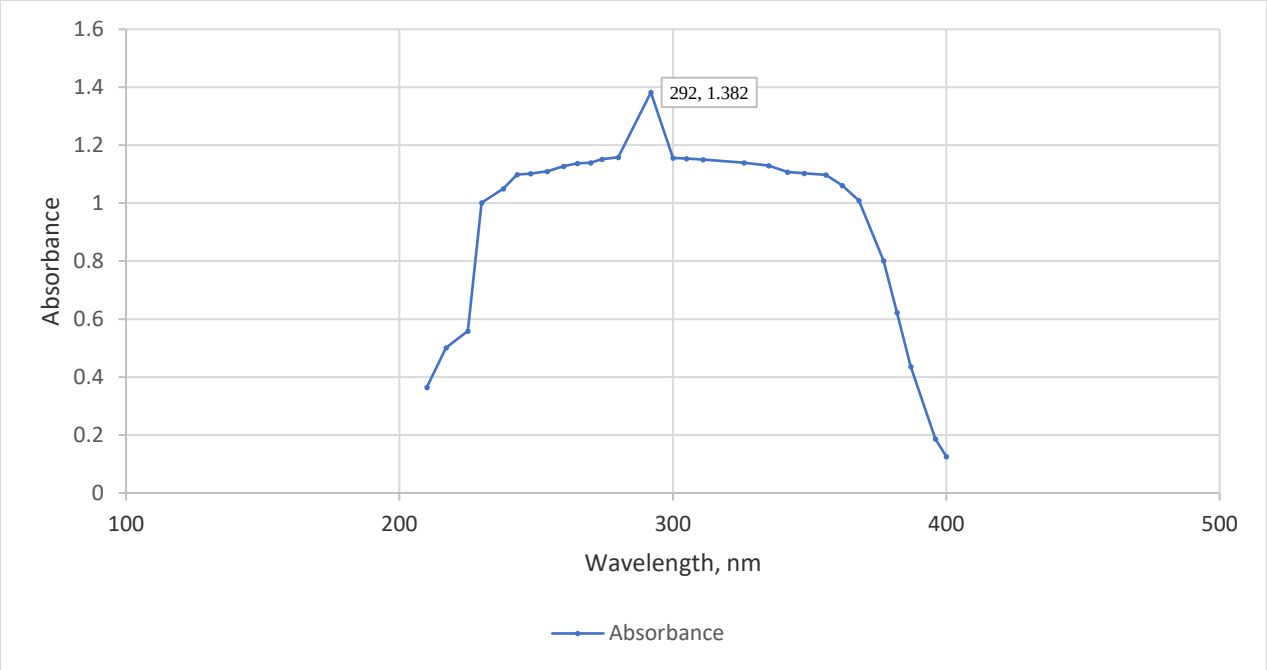

Figure 4. 1 Absorbance vs wavelength

To add a twist to the calibration, an iodometric titration to standardize the stock sodium hypochlorite arrangement is required[32]. Sodium hypochlorite in the amount of 8g (0.8%) was discovered. The calibration courses of action were made in a volume of 50 ml to cover a range of 0.1–0.8 percent NaOCl (2, 4, 6, and 8g or 0.2 percent, 0.4 percent, 0.6, and 0.8 percent). The calibration twist was achieved by graphing the absorbance at 292nm against a sodium hypochlorite concentration. The R^2 value was discovered to be 0.9957, indicating a high degree of linearity.

There will be more discussion on the attached.

4.3 FTIR Analysis

Infrared Spectroscopy with Fourier Transform (FTIR). Figure 4.3 depicts sodium hypochlorite spectra in the 400–4000 cm^{-1} region, which were used to identify the functional groups present in the sample[33], [34]. Several assimilation groups, including O-H and alkane, were discovered in natural bunches. Between the peaks 3693 and 2895 cm^{-1}, broadband was found in the sample NaOCl, which was associated with stretching hydroxyl (O-H) groups, indicating water as moisture[35], [36]. Furthermore, the peaks at 1643 cm^{-1} and 686 cm^{-1} correspond to the Na-O-Cl stretching band in anatase morphology. The adsorption peaks around 32895-3693 and 1442-1771cm^{-1} are attributed to stretching and bending vibrations of the O-H bond in the hydroxyl group, as well as absorbed water on the sodium hypochlorite surface. The peak absorbance was found to be broad and weak in this study, with values of 3276 cm^{-1} and 574 cm^{-1}. The adsorbed OH groups aid in the improvement of sodium hypochlorite's disinfection action by acting as an oxidant for the destruction of microorganisms and providing greater charge transfer by interacting with water[34], [37], [38].

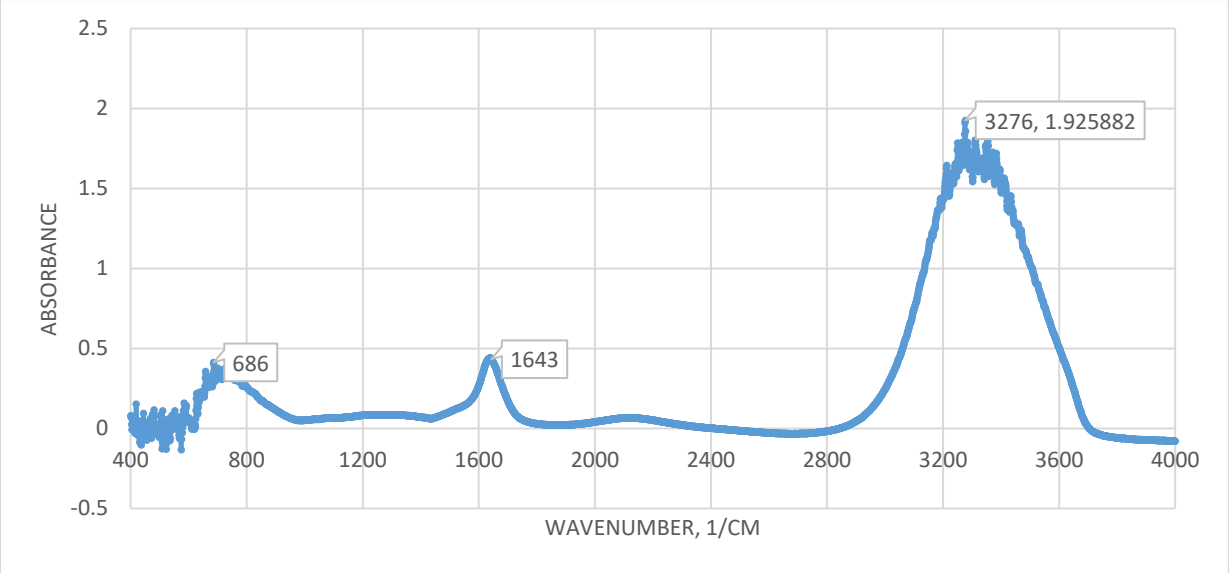

Figure 4. 2 FTIR-ART analysis curve

4.4 Analysis of NaOCl generation with Response Surface Methodology

Design-Expert software can determine the scientific link between the response and the independent factors NaCl concentration (A), an electric potential (B), electrode gap (C), and electrolysis time (D) in terms of coded and real variables (version 12). Within the condition, the model condition

was presented that ties the response (chlorine concentration) to the generation process factors in terms of coded components after removing the inconsequential terms. The analysis of the variance model was assumed to be quadratic, and ANOVA was performed using a fractional sum of squares methods with a lambda value of 1. Model A: NaCl concentration, B: electric potential, C: electrode gap, and D: electrolysis time and quadratic model factors; pure quadratic terms (A^2, B^2, C^2, D^2) and quadratic interaction terms (AB, AC, AD, BC, BD, CD) dependent on F and P values.

Table 4. 1 Box–Behnken RS design arrangement and response for NaOCl;

		Factor 1	**Factor 2**	**Factor 3**	**Factor 4**	**Response 1**
Std	Run	A: NaCl concentration	B: Electric potential	C: Electrode gap	D: Electrolysis time	Chlorine concentration
		g	volt	cm	min	g/l
4	1	50	9	3.5	50	5.81
22	2	30	9	3.5	10	4.28
29	3	30	6	3.5	50	6.52
27	4	30	6	3.5	50	6.29
2	5	50	3	3.5	50	2.86
25	6	30	6	3.5	50	6.61
17	7	10	6	1	50	4.56
6	8	30	6	6	10	3.17
10	9	50	6	3.5	10	4.33
7	10	30	6	1	90	7.25
19	11	10	6	6	50	3.92
9	12	10	6	3.5	10	1.72
23	13	30	3	3.5	90	3.76
12	14	50	6	3.5	90	4.96
18	15	50	6	1	50	5.79
3	16	10	9	3.5	50	4.68
14	17	30	9	1	50	5.2
5	18	30	6	1	10	4.61
15	19	30	3	6	50	1.91
11	20	10	6	3.5	90	5.41
8	21	30	6	6	90	5.86
13	22	30	3	1	50	3.45
24	23	30	9	3.5	90	5.31
20	24	50	6	6	50	5.46
16	25	30	9	6	50	5.6
28	26	30	6	3.5	50	6.24

21	27	30	3	3.5	10	1.21
1	28	10	3	3.5	50	0.9
26	29	30	6	3.5	50	6.65

4.4.1 Development of the RS model

ANOVA was used to test the constructed model for predicting hypochlorite chemical characteristics, which are listed in Tables 4.2. (chlorine concentration). The significance and suitability of the RS models were determined using the F-value test and the requirement of fit test. The F-value and p-value determine the significance of the RS models. To determine the centrality of the event, the F-value must be bigger than the p-value. The F-value for all of the RS models in this study was greater than the p-value (less than 0.05). All other models are substantially simpler as a result of this. The lack of fit for all RS models was not basic, demonstrating the models' ampleness. Typically appeared in Table 4.2.

The four process factors (NaCl concentration, electric potential, electrode gap, and electrolysis time) indicated on the RSM by BBD were used to optimize the response for sodium hypochlorite formation. Analysis of variance can be used to see the impact of the independent components and their interactions on the efficiency of NaOCl production (ANOVA).

Table 4.2 ANOVA for (Quadratic model) chlorine concentration

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	78.63	14	5.62	36.46	< 0.0001	significant
A-NaCl concentration	5.36	1	5.36	34.80	< 0.0001	
B-Electric potential	23.49	1	23.49	152.52	< 0.0001	
C-Electrode gap	2.03	1	2.03	13.20	0.0027	
D-Electrolysis time	14.59	1	14.59	94.70	< 0.0001	
AB	0.1722	1	0.1722	1.12	0.3082	
AC	0.0240	1	0.0240	0.1560	0.088	
AD	2.34	1	2.34	15.20	0.0016	
BC	0.9409	1	0.9409	6.11	0.0269	
BD	0.5776	1	0.5776	3.75	0.0733	
CD	0.0006	1	0.0006	0.0041	0.9501	
A^2	9.05	1	9.05	58.78	< 0.0001	
B^2	22.44	1	22.44	145.72	< 0.0001	
C^2	0.9561	1	0.9561	6.21	0.0259	
D^2	6.46	1	6.46	41.92	< 0.0001	
Residual	2.16	14	0.1540			

Lack of Fit	2.02	10	0.2017	5.78	0.0527	not significant
Pure Error	0.1395	4	0.0349			
Cor Total	80.78	28				

The 36.46 Model F-value indicates that the model is significant. An F-value this large appears to arise by chance only 0.01 percent of the time. Terms with P-values less than 0.0500 appear to be significant. A, B, C, D, AD, BC, A^2, B^2, $C^{2,}$ and D^2 are important model terms in this situation. The model terms are not important if the value is bigger than 0.1000. Model diminishment may make strides in your model if there are numerous inconsequential model terms (not including those necessary to support progression).

The F-value of 5.78 for the Lack of Fit indicates that it is not significant in comparison to the pure error. Due to noise, a significant Lack of Fit F-value has a 5.27 percent chance of occurring. It's bad if the show doesn't fit; we need it to. This low chance (less than 10%) is concerning.

Table 4. 2 Fit Statistics

Std. Dev.	0.3925	R^2	0.9733
Mean	4.63	Adjusted R^2	0.9466
C.V. %	8.47	Predicted R^2	0.8535
		Adeq Precision	20.7597

The Adjusted R^2 of 0.9466 is reasonably close to the Predicted R^2 of 0.8535; that is, the difference is less than 0.2. The signal-to-noise ratio is measured by Adeq precision. It is preferable to have a ratio of more than four. Your signal strength is adequate at 20.760. This demonstration can be used to learn more about the design process.

Final Equation in Terms of Coded Factors

$$\text{Chlorine concentration} = +6.46 + 0.6683 * A + 1.40 * B - 0.4117 * C + 1.10 * D - 0.2075 * AB + 0.0775 * AC - 0.7650 * AD + 0.4850 * BC - 0.3800 * BD + 0.0125 * CD - 1.18 * A^2 - 1.86 * B^2 - 0.3839 * C^2 - 0.9977 * D^2 \quad (4.2)$$

For certain levels of each figure, the condition in terms of coded variables can be used to form expectations about the reaction. The high levels of the components are coded as +1 by default, whereas the low levels are coded as -1 by default. By comparing the factor coefficients, the coded

condition is useful for recognizing the relative effect of the components.

Final Equation in Terms of Actual Factors

$$\begin{aligned}\text{Chlorine concentration} = {} & -12.01506 + 0.273767 * \text{NaCl concentration} + 2.98236 \\ & * \text{Electric potential} - 0.175430 * \text{Electrode ga} + 0.137167 \\ & * \text{Electrolysis time} - 0.003458 * \text{NaCl concentration} \\ & * \text{Electric potential} + 0.001550 * \text{NaCl concentration} \\ & * \text{Electrode gap} - 0.000956 * \text{NaCl concentration} \\ & * \text{Electrolysis time} + 0.064667 * \text{Electric potential} \\ & * \text{Electrode gap} - 0.003167 * \text{Electric potential} \\ & * \text{Electrolysis time} + 0.000125 * \text{Electrode gap} \\ & * \text{Electrolysis time} - 0.002954 * \text{NaCl concentration}^2 \\ & - 0.206685 * \text{Electric potential}^2 - 0.061427 * \text{Electrode gap}^2 \\ & - 0.000624 * \text{Electrolysis time}^2\end{aligned} \quad 4.3$$

For certain levels of each calculate, the condition in terms of real components can be used to create predictions about the reaction. The levels should be written in the appropriate units for each figure. Because the coefficients are scaled to suit the units of each calculate and the captured isn't at the center of the design space, this condition shouldn't be used to determine the relative effect of each calculate.

4.4.2 Diagnostics plots and Model graph of response chlorine concentration on the Experimental Variables

Figure 4.3 depicts a normal probability map of residuals. When test data is used to frame typical dispersion, the residuals fall on a straight line, implying that the errors are spaced out in a typical dispersion. A leftover here denotes a difference between the observed esteem (as determined by the test) and the expected or fitted value (esteem that can be anticipated by the created model condition). As a result, looking at this typical figure, all of the focuses line up nicely, and the variation of focuses from normalcy is negligible, implying that the chlorine concentration is modeled).

As a result, the model does not require any adjustment because it is well fitted, as illustrated by the Box-cox plots in Figure 4.5. If the data is normal, it doesn't need to be transformed; instead, it can recommend the optimum model transformation. Figure 4.4, on the other hand, depicts the plot

of residuals versus the test run arrangement. It looks for hidden elements that may have influenced the test's outcome. Because the plot reveals a random scatter, no more blocking or other solutions are required. Figure 4.4 shows residual vs. any chosen factor to see if the model can account for the variation; the graph represents a random scatter.

Another essential diagnostic plot to consider is the expected versus actual (Figure 4.7), which is a graph of the predicted response values versus the actual response values. The goal is to find a value, or a collection of values, that the model can't predict easily. Looking at the graph, all of the data fall along a straight line, demonstrating the model's strong predictive power.

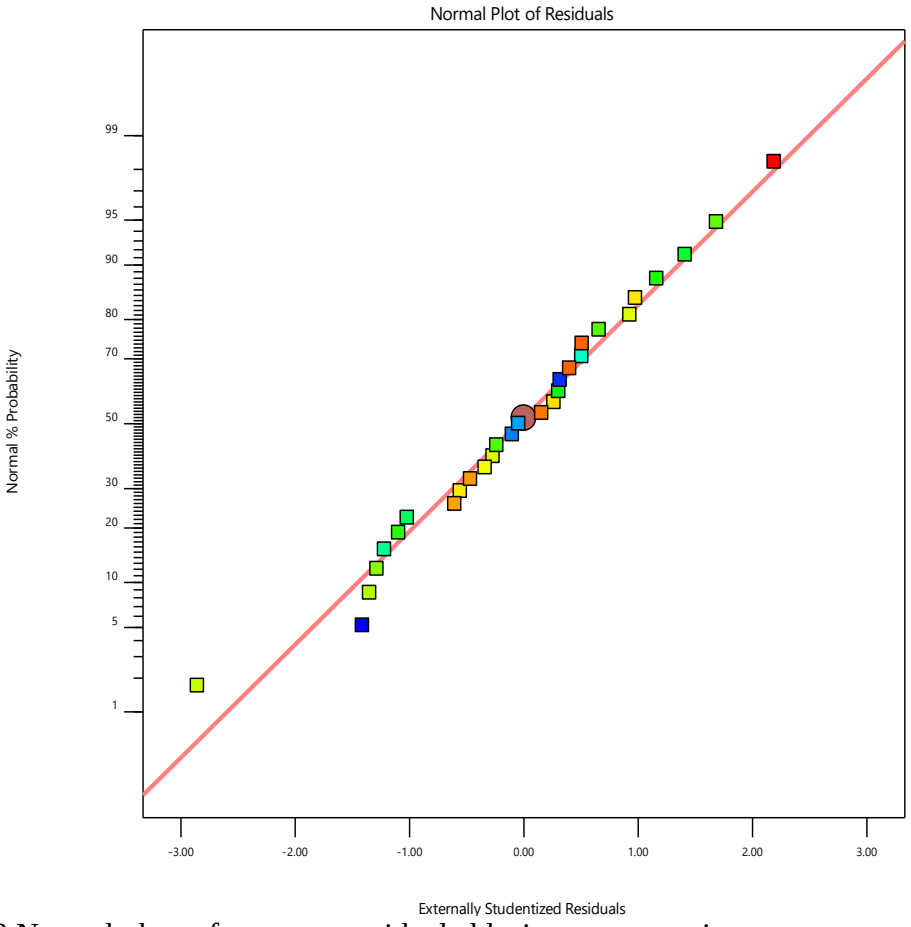

Figure 4. 3 Normal plots of response residual chlorine concentration

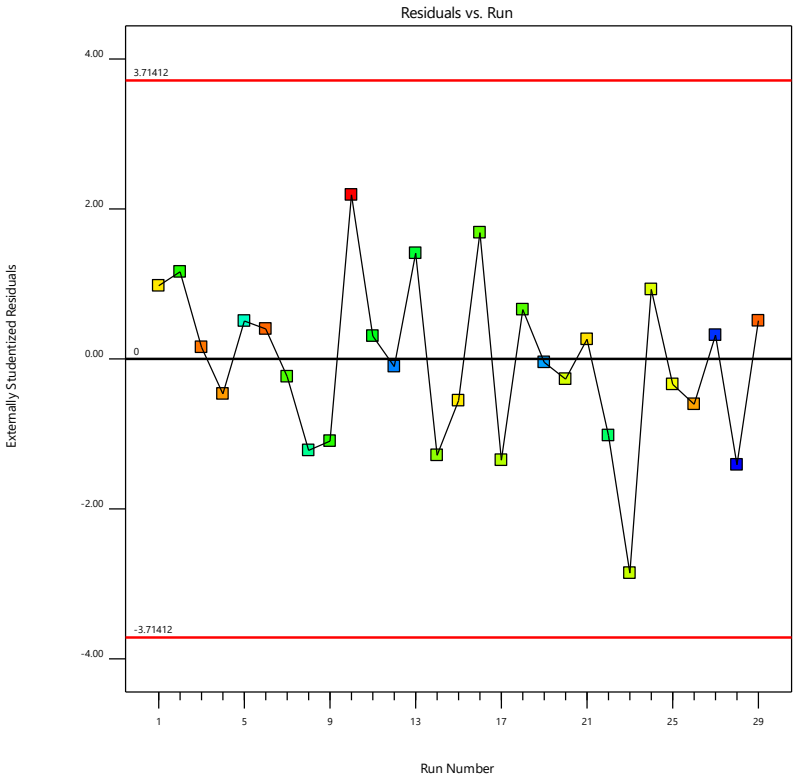

Figure 4. 4 Residual vs run plot of response chlorine concentration

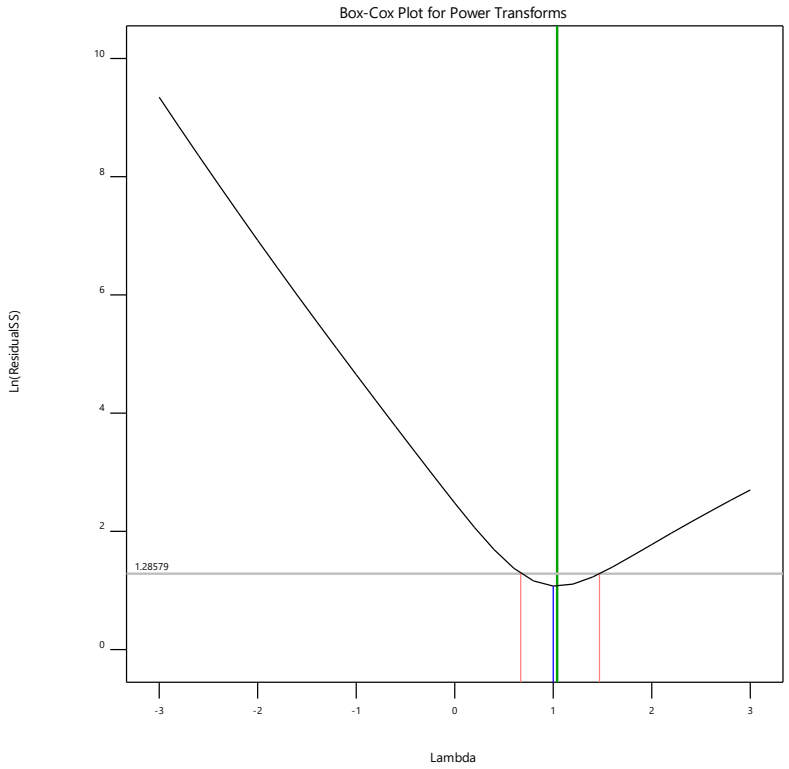

Figure 4. 5 Box-Cox plot of response chlorine concentration

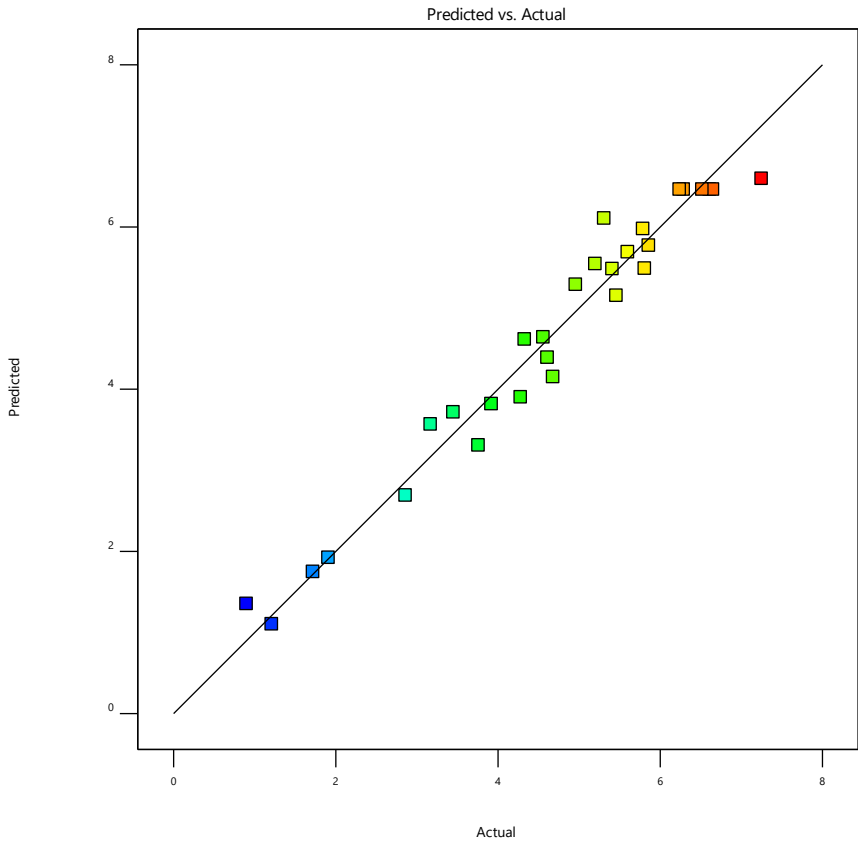

Figure 4. 6 Predicted vs Actual plot of response chlorine concentration

4.5 Effects of parameters on sodium hypochlorite generation

According to the data obtained from the design expert software version 12, we'll attempt to evaluate the several process factors (variables) that are causing the generation of hypochlorite in this section. Based on the required condition, these parameters can be NaCl concentration, electric potential, electrode gap, and electrolysis time.

4.5.1 Influence of NaCl concentration and electric potential

The analyses show that increasing sodium chloride concentration causes an increase in sodium hypochlorite production of up to 32 g/l. The formation of chlorate can be aided by a higher concentration of NaCl. The current thickness has increased as the electrical potential has increased. By raising the current thickness, the electron exchange rate is raised. The efficacy of NaOCl production is extremely dependent on a proper electric potential. The effect of voltage on the formation of NaOCl using titanium coated with iridium oxide and stainless-steel electrodes was investigated in this study. In 29 tests, the electric potential was changed from 3 to 9 volts.

As shown in Figure 4.7, the interaction effects of NaCl concentration and electric potential over

time show that increasing the electric potential increases the generation of NaOCl concentrations, while increasing the NaCl concentration up to the optimum point increases the generation of NaOCl concentrations. The generation of NaOCl will be affected by an advanced increase in electric potential.

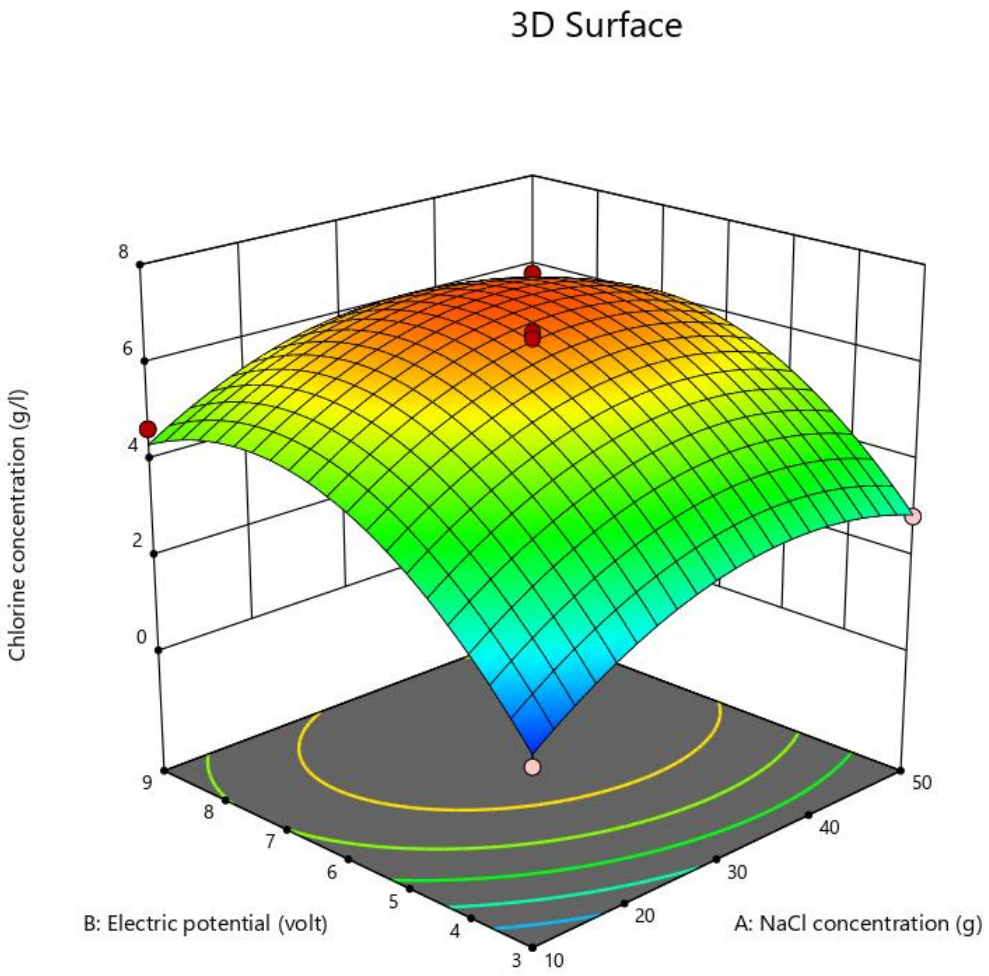

Figure 4. 7 3-D interaction effects of NaCl concentration and electric potential on NaOCl generation

4.5.2 Influence of NaCl concentration and electrode gap

An increase in sodium chloride concentration until it reaches the optimum level causes an increase in sodium hypochlorite production. Chlorate can be formed if the concentration of NaCl is increased rapidly. The influence of the gap (space) between the cell's two electrodes was taken into account. The results show that there was an increase in sodium hypochlorite formation as the gap between the two electrodes shrank.

As observed in Figure 4.8, the interaction impact of NaCl concentration and electrode gap over time demonstrates that as the amount of NaCl concentration grows up to optimal, so does the formation of NaOCl. However, as the electrode gap increases, so does the efficacy of NaOCl generation. This could be because the number of ion exchange (electrolysis) active sites available to generate NaOCl at the tiny electrode gap is large.

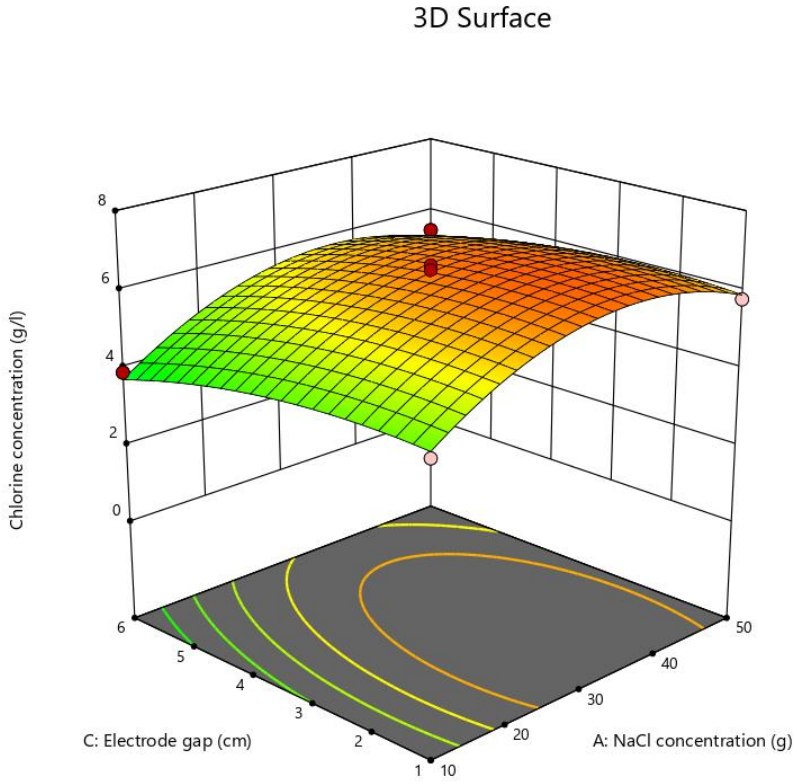

Figure 4. 8 3-D interaction effects of NaCl concentration and electrode gap on NaOCl generation

4.5.3 Influence of NaCl concentration and electrolysis time

The generation of NaOCl is affected by the method's electrolysis time. The effect of electrolysis time on the formation of NaOCl was investigated using two-terminal materials: titanium coated with iridium oxide and stainless steel. The duration of electrolysis varied between 10 and 90 minutes. Expanding the term time creates an arranged relationship with the NaOCl era until it reaches an ideal point, and when the NaOCl generation reaches an immersion point, electrolysis time has no fundamental impact on NaOCl production. These connections can be seen in the diagrams below.

A rise in both NaCl concentration and electrolysis time up to the optimum point, as illustrated in Figure 4.9, increases NaOCl. The arrangement of chlorate can be influenced by the next

concentration of NaCl. The production of sodium hypochlorite is unaffected by increasing the electrolysis time.

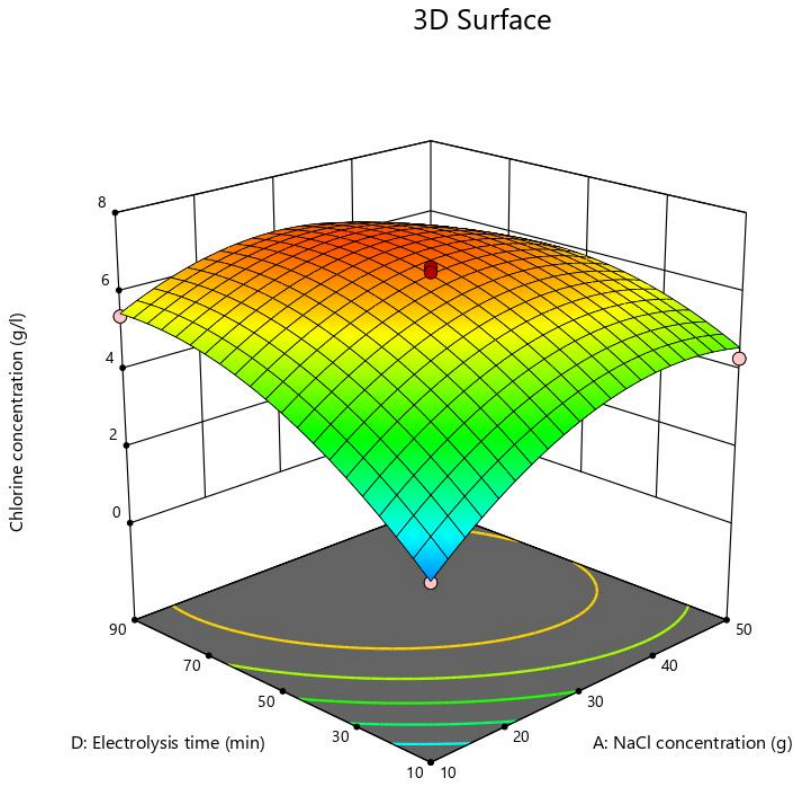

Figure 4. 9 3-D interaction effects of NaCl concentration and electrolysis time on NaOCl generation

4.5.4 Influence of electric potential and electrode gap

The results demonstrate that when the space between the two electrodes narrowed, sodium hypochlorite production increased. Figure 4.10 depicts the interaction impact of electric potential and electrode gap over time. As the electric potential rises to the optimum, the generation of NaOCl rises as well, but as the electrode gap rises, the effectiveness of NaOCl creation decreases. This could be due to a large number of ion exchange (electrolysis) active sites available to make NaOCl at the tiny gap between electrodes and the rapid rise in electric potential required to convert the desired product to sodium hypochlorate.

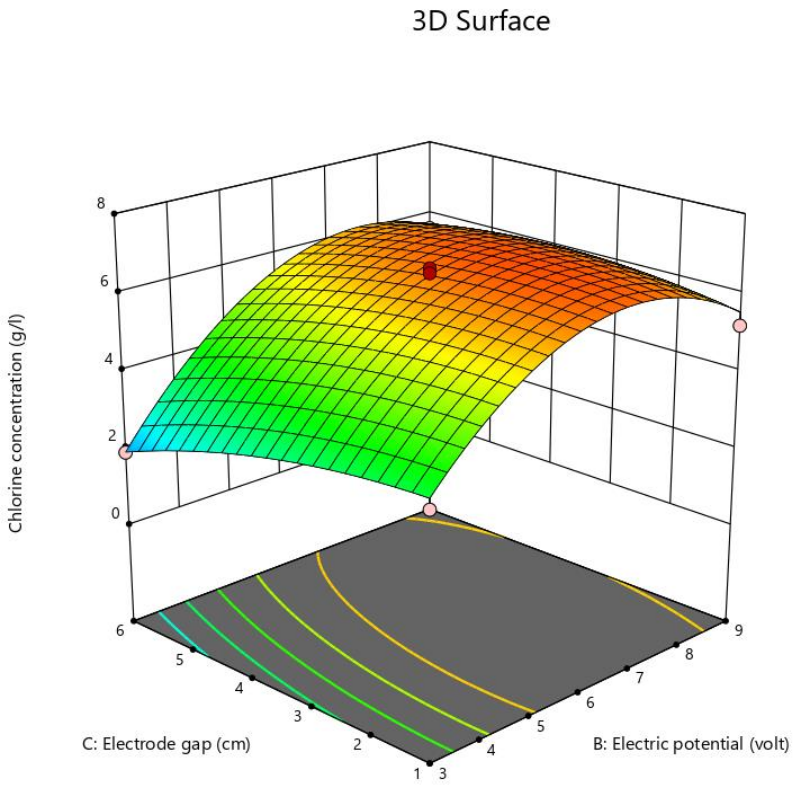

Figure 4. 10 3-D interaction effects of electric potential and electric gap on NaOCl generation

4.5.5 Influence of electric potential and electrolysis time

The analysis reveals that increasing the length of time creates a coordinate link with the era of NaOCl until it reaches an immersion point and that when hypochlorite generation reaches a saturation point, electrolysis duration has no effect on hypochlorite creation. An increase in both electric potential and electrolysis time up to the optimum point, as illustrated in Figure 4.11, increases NaOCl. An advanced increase in electric potential over the optimum has a major effect on NaOCl generation, i.e., it causes an increase in product temperature, which causes hypochlorite to be converted to hypochlorate.

4.5.6 Influence of electrolysis time and electrode gap

On the other hand, as shown in Figure 4.12, increasing the electric potential up to the optimum increases the generation of NaOCl; however, increasing the electrode gap reduces the effectiveness of NaOCl creation. This could be because there are a lot of ion exchange (electrolysis) active sites available to make NaOCl in the little area between the electrodes and the electric potential rises quickly enough to convert the desired product to hypochlorate.

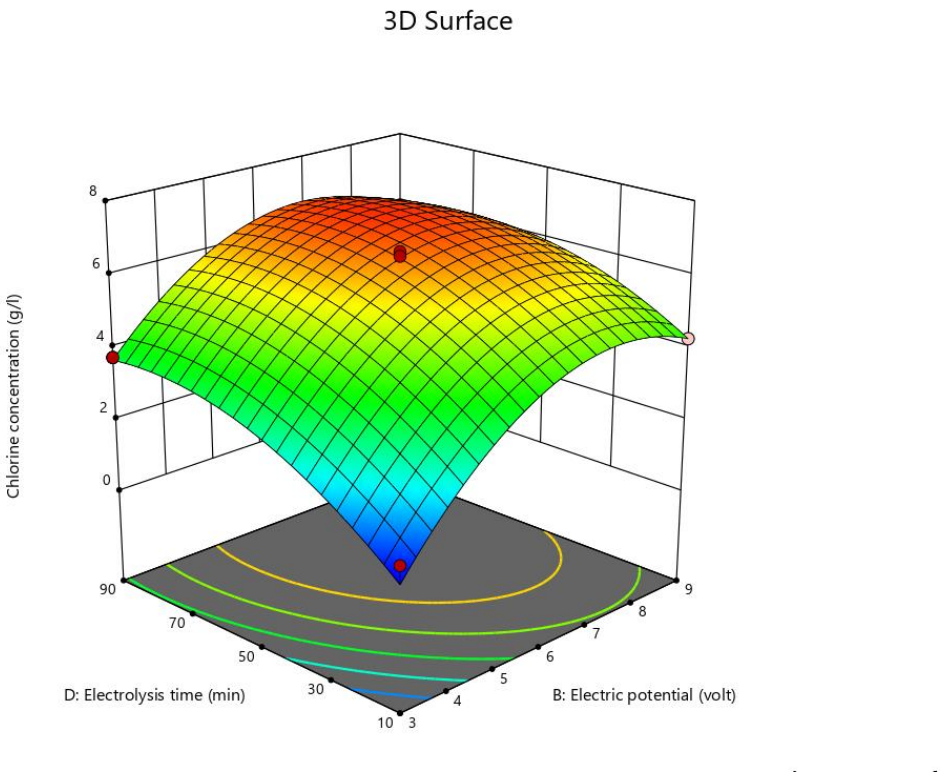

Figure 4. 11 3-D interaction effects of electric potential and electrolysis time on NaOCl generation

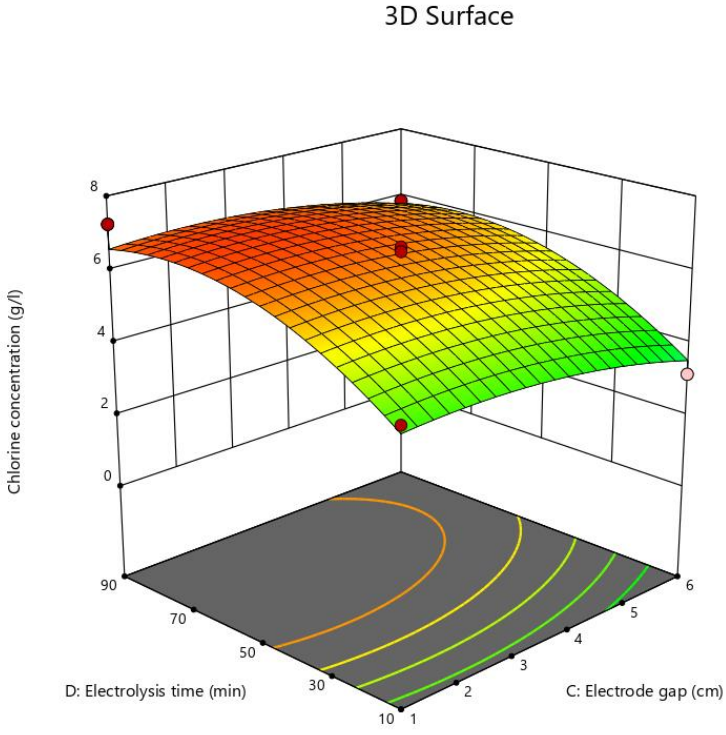

Figure 4. 12 3-D interaction effects of electrode gap and electrolysis time on NaOCl generation

4.6 Optimization of the Model

Manipulation of the process parameters can improve the electrolysis generation of sodium hypochlorite. Four elements (such as NaCl concentration, electric potential, electrode gap, and electrolysis time) were chosen for the optimization operation and are listed in Table 4.4, each with the lowest and maximum values that defined the domains of variables that influence NaOCl formation. Though it is frustrating to find the optimal point for the response due to the interaction and unclear effect of the parameters, using the numerical optimization procedure resulted in high-quality work.

Numerical optimization is a comprehensive advanced explanation of the most successful process optimization method. The model predicts the upper and lower restrict values of each parameter, as well as its response, based on the ideal objective. The ultimate goal of this optimization was to obtain the highest response (chlorine concentration) while also meeting all of the components' requirements at their respective levels or values.

Table 4. 3 Data for optimization operation

Name	Goal	Lower limit	Upper limit
A: NaCl concentration	In range	10	50
B: electric potential	In range	3	9
C: electrode gap	In range	1	6
D: electrolysis time	In range	10	90

Figure 4.13 depicts the optimum value for sodium hypochlorite electrolysis generation, in which the selected factors NaCl concentration, electric potential, electrode gap, and electrolysis time are 32.36g, 7v, 2.5cm, and 70 minutes, respectively, resulting in the highest sodium hypochlorite concentration.

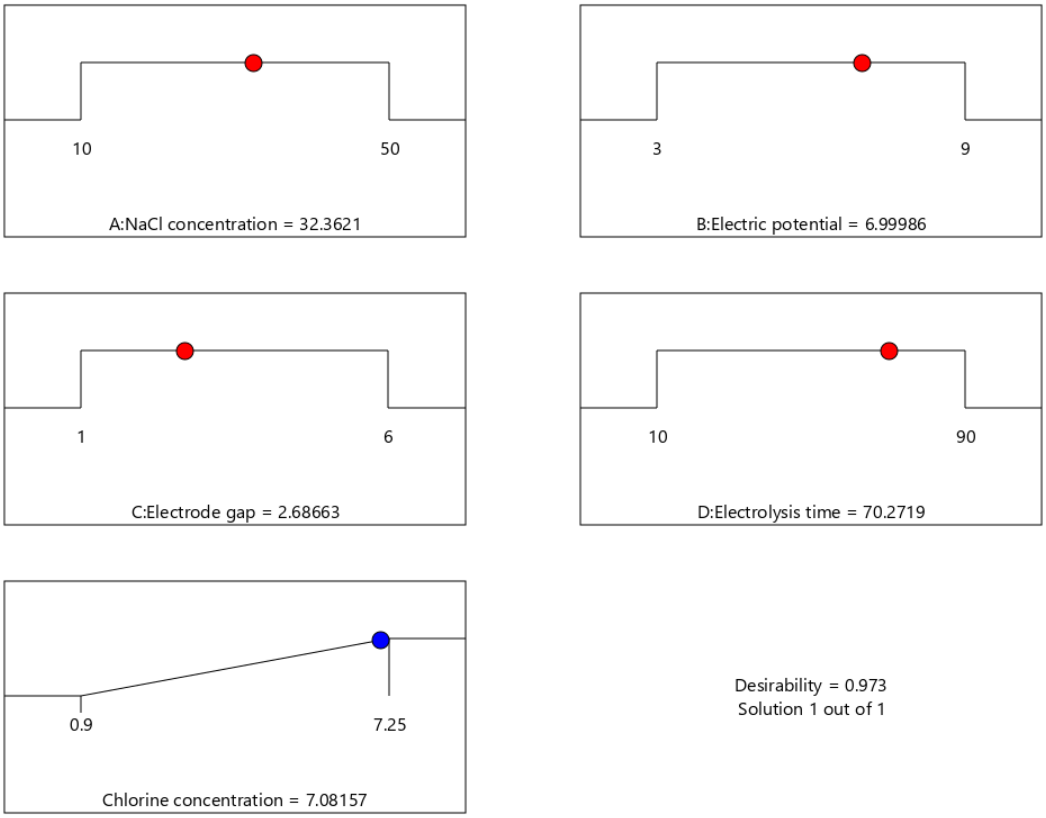

Figure 4. 13 Optimization value for the response of chlorine concentration.

4.7 Experimental validation

The best-optimized settings for NaOCl generation were 32.36g NaCl, 7v, 2.5cm electrode gap, and 70 min with a PH of 11, 7.2g Cl_2/L, based on data from statistical optimization and using titanium coated with iridium oxide and stainless-steel electrode pairs.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The goal of this thesis was to look into the electrolysis generation of NaOCl for water and inert surface disinfection using sodium chloride solution. The effects of several process factors, such as NaCl concentration, electric potential, electrode gap, and electrolysis time, were investigated.

The following centers can be concluded as a result of the research:

1. It is possible to use electrolyzing sodium chloride to produce sodium hypochlorite on-site at a low concentration (10gpl or 1%), which can be used as a water and idle surface disinfectant.
2. Using RSM, the optimal electrolysis conditions for the production of NaOCl were discovered. It reveals that quadratic models with R^2 and R^2 adj values more than 0.90 accurately portrayed and predicted the responses (chlorine concentration) under test conditions.
3. The optimal conditions for the formation of NaOCl were discovered using titanium coated with iridium oxide and stainless-steel electrodes, a NaCl concentration of 32g, a voltage of 7v, a spacing between the electrodes of 2.5cm, and an electrolysis time of 70 minutes at a temperature of 25°C. In this investigation, the most excellent effective concentration value of NaOCl was 0.72 percent (7.2gpl) of chlorine concentration with a pH of 11.
4. Comparing various research facility perspectives, including security, environmental, health, financial, and political concerns. It was evident that the NaOCl produced on-site was the most cost-effective solution for maintaining acceptable security and success concerns while also overcoming political and operational hurdles.

5.2 Recommendation

It is critical to examine the issue of electrolysis NaOCl formation from NaCl for water and inert surface disinfection. Based on the findings of this thesis, the following suggestions for further research are made (study).

1. Future research should concentrate on using continuous flow experiments to generate sodium hypochlorite plus therapy (disinfection).
2. It is proposed that electrolysis cells manufactured by international companies be used, since they may provide better and more conservative results for NaOCl generation than batch mode experiments.
3. Extending the surface area of the electrodes is more feasible since it will produce more NaOCl and will be more cost-effective than using an electrical current.
4. It was discovered through research facility testing that the security of using and caring for NaOCl solution in comparison to market NaOCl is high, and the achievability of using NaCl in making NaOCl is high, so I recommend that the government prioritize this issue.

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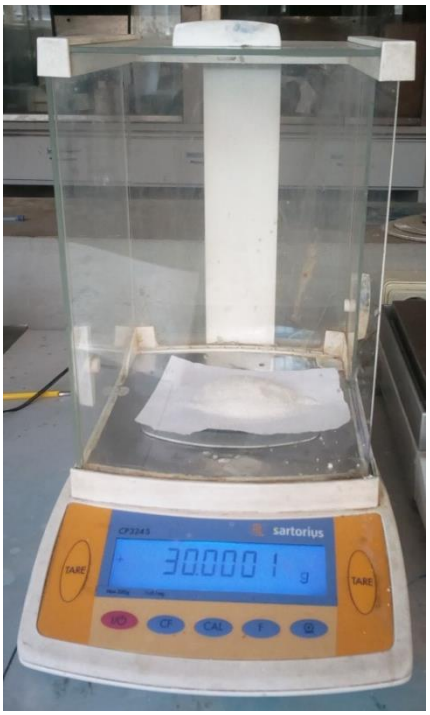
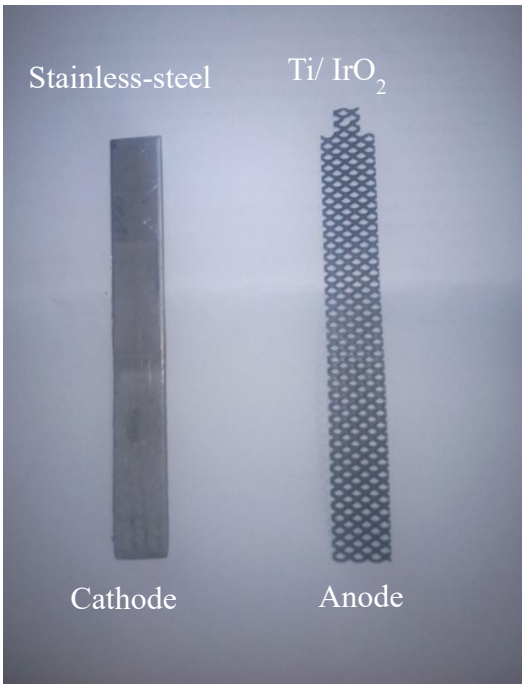
ANNEXES

Annex 1. The material utilized in the generation of NaOCl and experimental set up



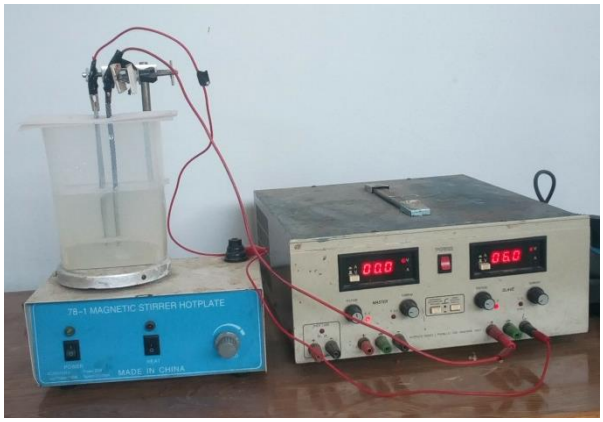
Digital balance



Electrodes utilized

DC supply(250v)

Chemical utilized



Experimental set up

UV-VIS spectrophotometer

Iodometric titration/NaOCl characterization

Annex 2. UV-VIS absorbance measurement

RUN	Absorbance	K*Abs
1	0.364	3.64
2	0.501	5.01
3	0.559	5.59
4	1.001	10.01
5	1.050	10.50
6	1.099	10.99
7	1.102	11.02
8	1.110	11.10
9	1.127	11.27
10	1.382	13.82
11	1.140	11.40
12	1.152	11.52
13	1.158	11.58
14	1.137	11.37
15	1.156	11.56
16	1.154	11.54
17	1.150	11.50
18	1.139	11.39
19	1.130	11.30
20	1.108	11.08
21	1.103	11.03
22	1.097	10.97
23	1.061	10.61
24	1.009	10.09
25	0.802	8.02
26	0.623	6.23
27	0.436	4.36
28	0.187	1.87
29	0.126	1.26

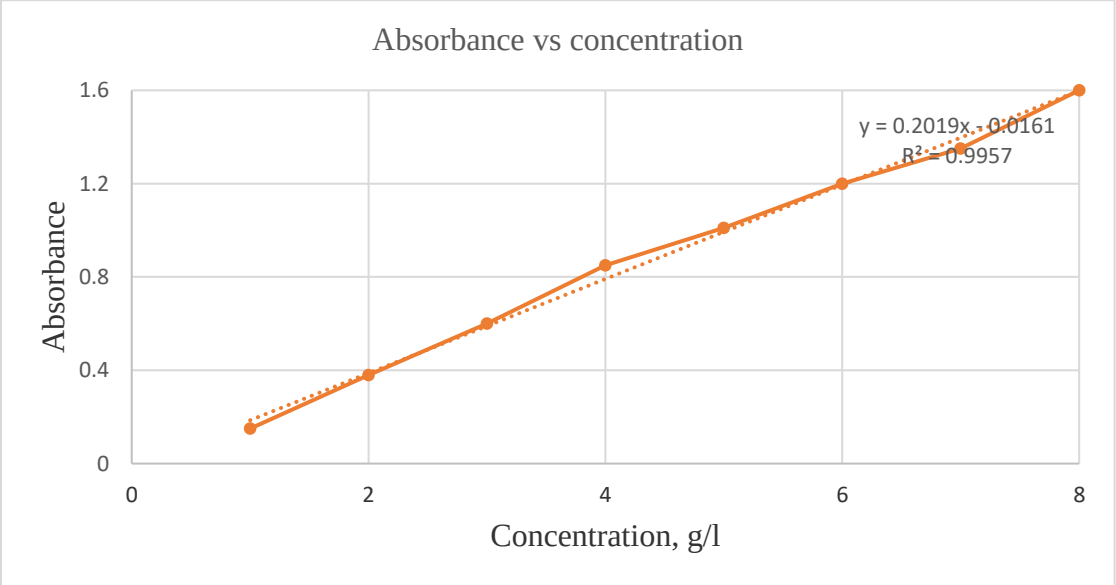
The Beer Lamber law for the assurance of the concentration of A given chlorine in NaOCl whose absorbance is known:

$$C = \frac{A - \text{Intercept}}{\text{Slope}} \qquad 4.1$$

Where A is the intercept for the standard calibration plot of chlorine in NaOCl.

C is the concentration of unknown chlorine in NaOCl

Slope and intercepts are from the standard calibration plot as appeared within the fig below.

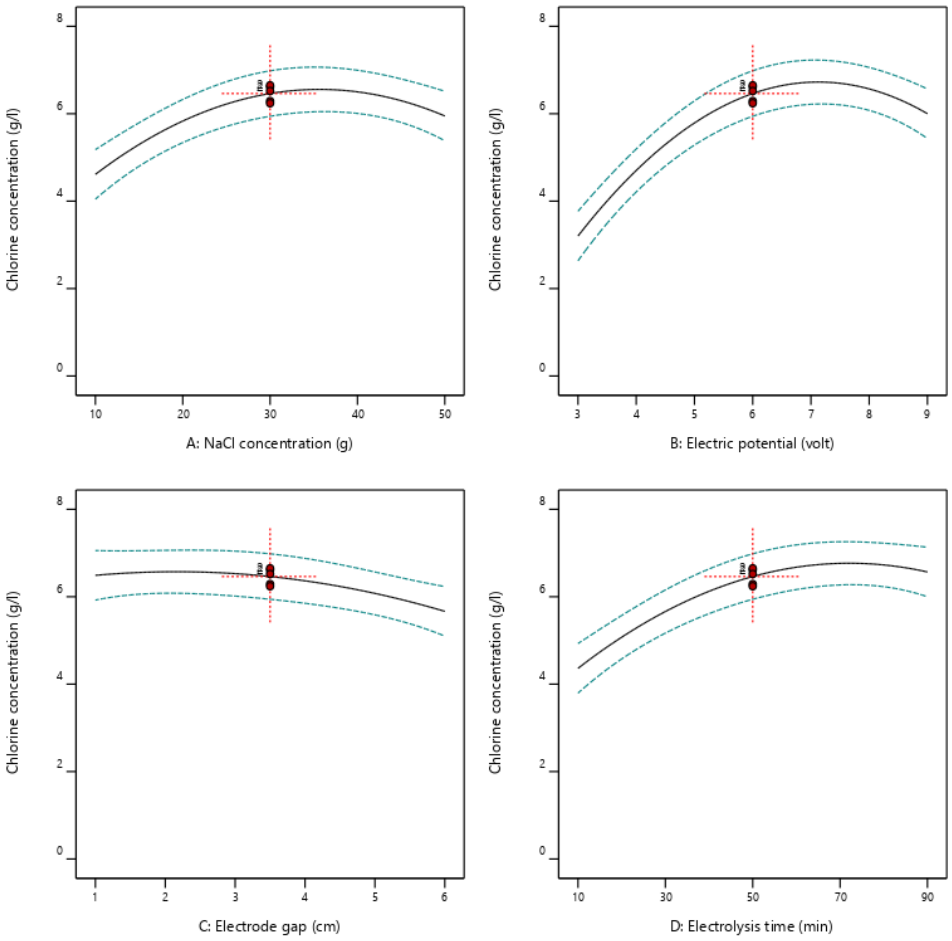


Annex 3. Statistical report of the diagnostic table for the actual and predicted value of the response of chlorine concentration in NaOCl.

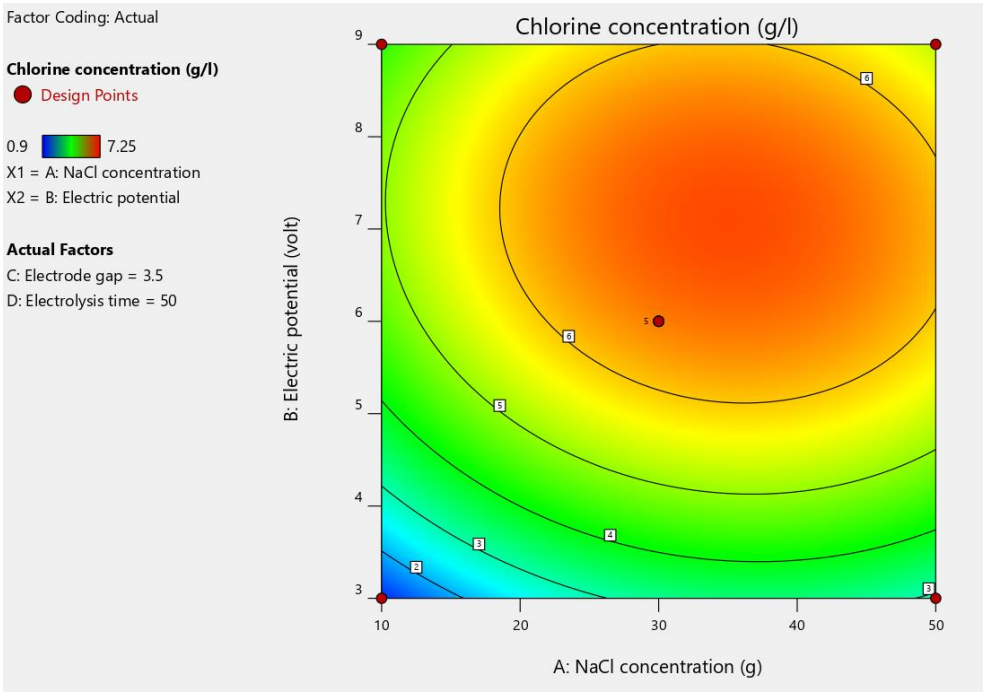
Run Order	Actual Value	Predicted Value
1	5.81	5.49
2	4.28	3.90
3	6.52	6.46
4	6.29	6.46
5	2.86	2.69
6	6.61	6.46
7	4.56	4.64
8	3.17	3.57
9	4.33	4.61
10	7.25	6.59
11	3.92	3.82
12	1.72	1.75
13	3.76	3.31
14	4.96	5.29
15	5.79	5.98
16	4.68	4.15
17	5.20	5.54
18	4.61	4.39
19	1.91	1.92
20	5.41	5.48
21	5.86	5.77
22	3.45	3.72
23	5.31	6.11

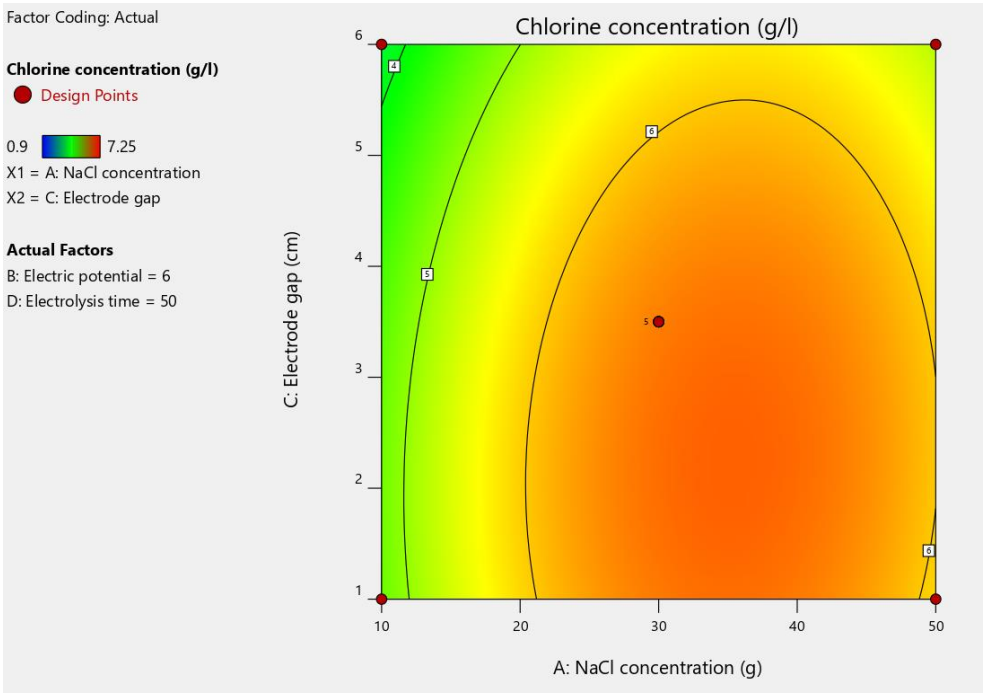
24	5.46	5.15
25	5.60	5.69
26	6.24	6.46
27	1.21	1.10
28	0.9000	1.35
29	6.65	6.46

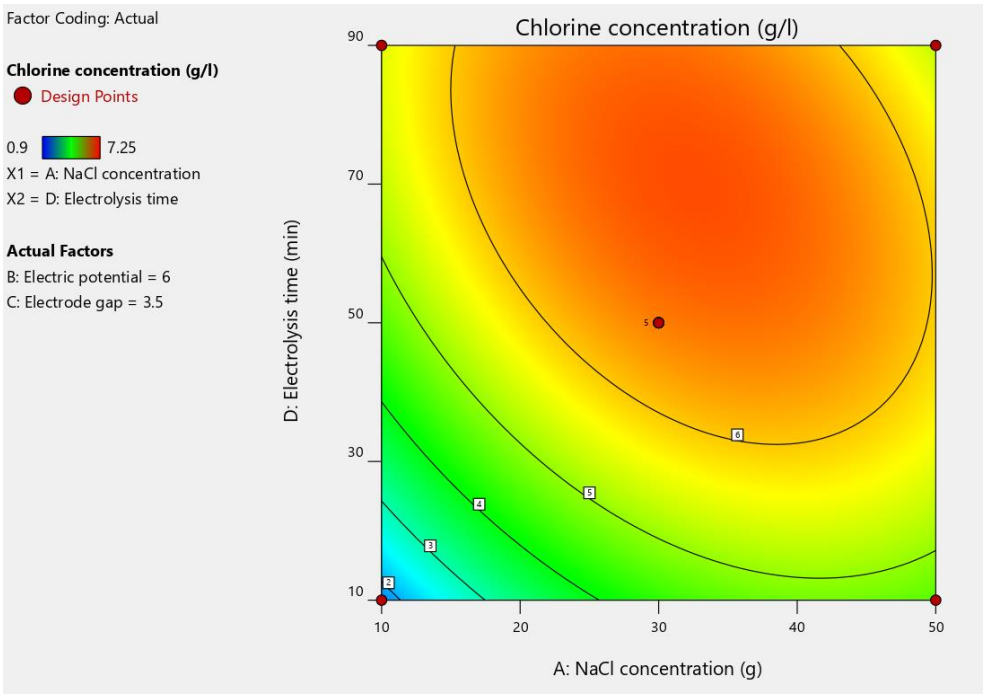
Annex 4. One factorial plot for the response chlorine concentration

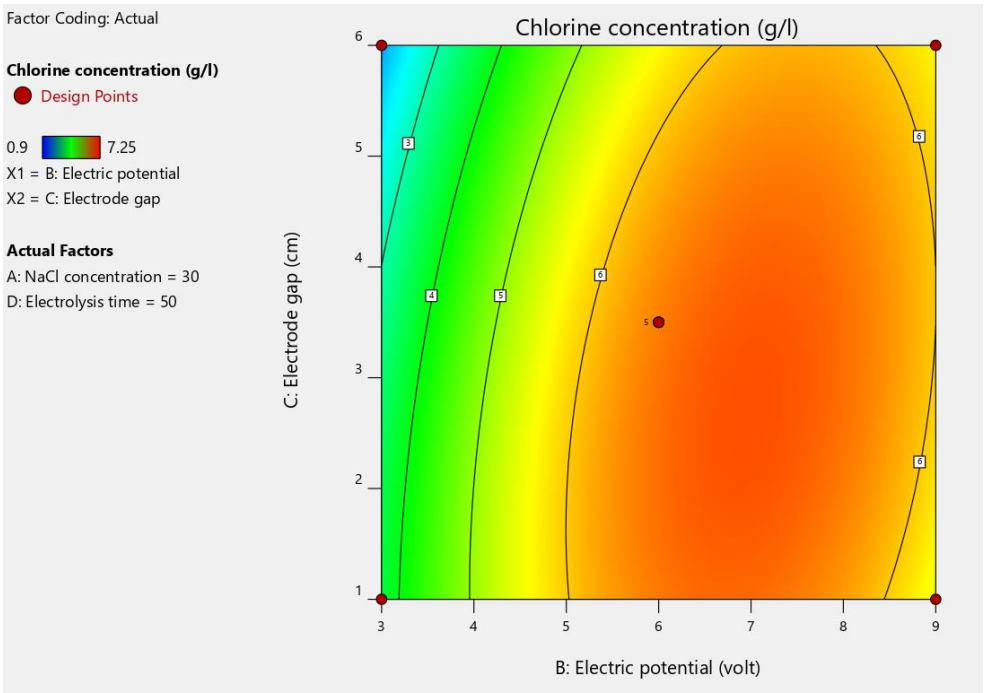


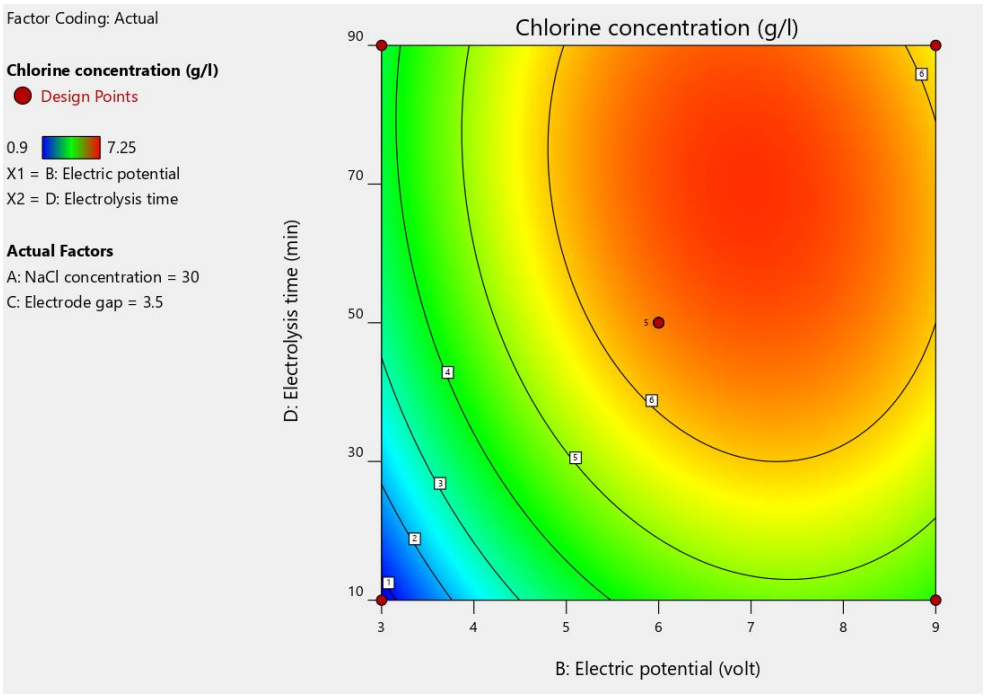
Annex 5. Contour plot for the response chlorine concentration

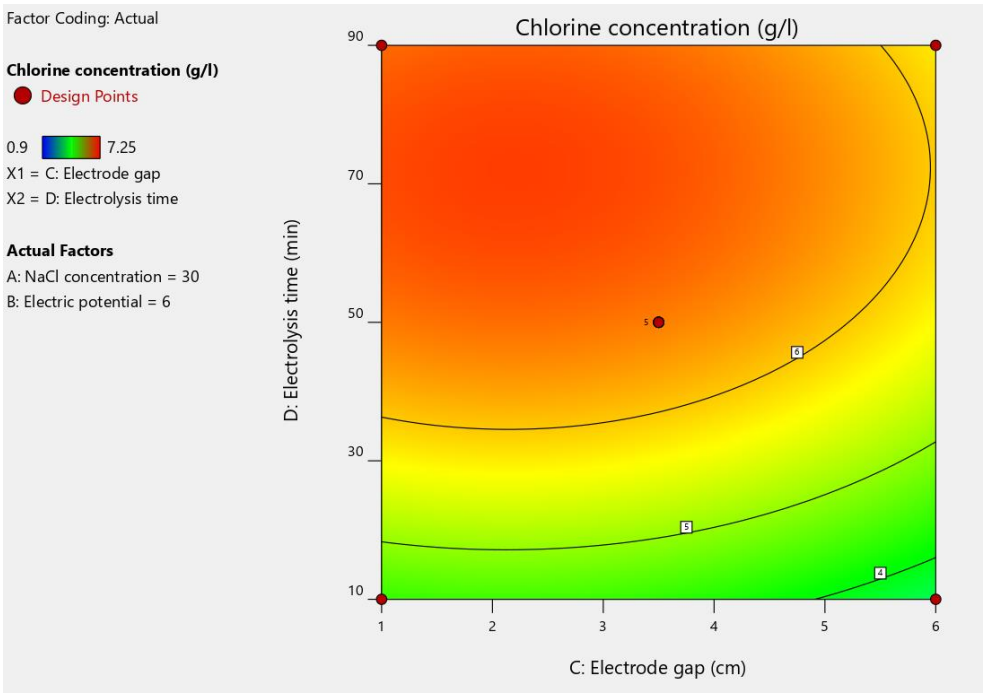





Factor Coding: Actual
Chlorine concentration (g/l)
Design Points
0.9
7.25
X1 = A: NaCl concentration
X2 = D: Electrolysis time
Actual Factors
B: Electric potential = 6
C: Electrode gap = 3.5
Chlorine concentration (g/l)
D: Electrolysis time (min)
A: NaCl concentration (g)
90
70
50
30
10
10
20
30
40
50


Factor Coding: Actual
Chlorine concentration (g/l)
Design Points
0.9
7.25
X1 = B: Electric potential
X2 = C: Electrode gap
Actual Factors
A: NaCl concentration = 30
D: Electrolysis time = 50
Chlorine concentration (g/l)
C: Electrode gap (cm)
B: Electric potential (volt)
6
5
4
3
2
1
3
4
5
6
7
8
9


Factor Coding: Actual
Chlorine concentration (g/l)
Design Points
0.9
7.25
X1 = B: Electric potential
X2 = D: Electrolysis time
Actual Factors
A: NaCl concentration = 30
C: Electrode gap = 3.5
Chlorine concentration (g/l)
D: Electrolysis time (min)
B: Electric potential (volt)


Factor Coding: Actual
Chlorine concentration (g/l)
Design Points
0.9
7.25
X1 = C: Electrode gap
X2 = D: Electrolysis time
Actual Factors
A: NaCl concentration = 30
B: Electric potential = 6
Chlorine concentration (g/l)
D: Electrolysis time (min)
C: Electrode gap (cm)