



Development of an Electrolytic Pilot Plant for the Production of Chlorine Gas “In Situ” in the Disinfecting Water Process

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Abstract

Technology of gaseous chlorine in water treatment has shifted to the dosage of sodium hypochlorite or calcium hypochlorite because of their greater biocide power; require less contact time with the microorganisms in the pretreated water, and the pH of slightly affect water. The generation of chlorine gas in place is based on the principles of electrolytic dissociation and laws of electrolysis by Faraday. The equipment corresponds to an electrolytic cell of three (3) compartments, a central one (anode) where chlorine gas emerges, and two side (cathodes) where sodium hydroxide is produced in the central compartment must be refilled chloride solution sodium consumed due to the electrochemical reaction, the chlorine evolved being of gaseous nature ascends the column of sodium chloride and is captured by the vacuum venture system, who to put in direct contact with target that has been previously subjected to the processes of uptake, coagulation, sedimentation and filtration. Compartments anode-cathode - anode are physically separated by porous diaphragm, whose purpose is to permit selective flow of sodium and chloride ions, avoiding side reactions recombination, as additional product is the formation of sodium hydroxide in the cathode chambers, which should be removed from the cell through side pipes. Statistical models were used to optimize the performance and operation of the prototype.

Keywords: Saline electrolysis; chlorine gas; electrolytic reactor

1 A Headings are the primary heading type

One of the most common diseases among the population of Latin America are related to lack of water and contaminated water [1]; taking millions of lives per year [2]. In most large cities in the country with more than 10000 inhabitants, chlorination has been made reasonably reliable using gaseous chlorine dosing technology supplied in cylinders, however, chlorination of potable water supply systems that serve smaller populations usually remains unreliable and intermittent [3]. To help solving this problem, many technological alternatives applicable to disinfection have been investigated [4 and 5]. These include various technologies suitable for ozonation [6], iodine [7], ultraviolet radiation [8], as well as the use of various methods for performing gas chlorination, chloramines, chlorine dioxide and methods for generating disinfectants in situ [9 and 10]. The latter method being the one that has proven to be the most promising of all the technologies used [10].

2 Plant Development

2.1 CAD design and construction electrolytic pilot plant for gas chlorine production

For the purpose of evaluating the performance of chlorine-generating equipment in situ an electrolytic cell was designed and

built (1), which is loaded with concentrated brine (6) to which electric current supplied by a DC power rectifier source is applied (3), the mixture of gases generated in the anode are extracted by the suction created by the Venturi system (2), the pump (5) circulates a working volume (4) of 200 lt at a flow rate of 15,3 Lt/min (9,18 mt3/h), passing through the Venturi and the shunt (7). As seen in the Fig. 1 and Fig. 2.

2.2 CAD design and construction of an electrolyte cell with a capacity of 15 A

The reactor design used in the tests consists of an electrolyte cell loaded with 20 liters of brine at concentrations of 45 and 68 gr NaCl/lt respectively, which produces oxidizing gases (chlorine and oxygen species) in the anode compartment, and sodium hydroxide and hydrogen in the cathode compartment, which is shown in the fig. 4. These two compartments are separated by a semipermeable polysulfonated membrane usually made of Nafion®, General purpose material in the manufacture of car batteries, the prototype body was made of 5mm thick transparent acrylic sheets resistant to the action of chlorine, ozone and NaOH. The cathode of the unit is made of stainless steel and a matrix of 22 cylindrical graphite electrodes 10mm in diameter by 300mm of length.

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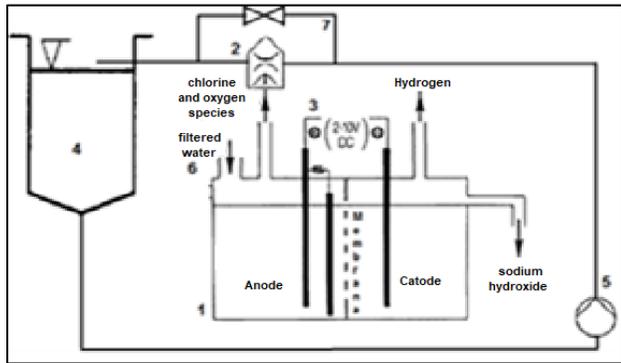


Figure 1: Schematic diagram of the test team

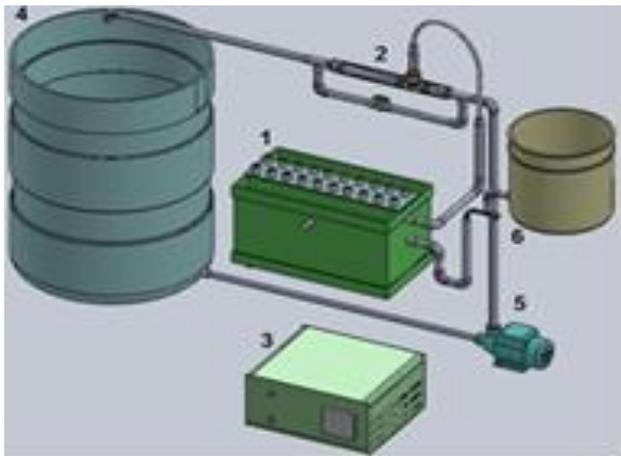


Figure 2: 3D design of the test team

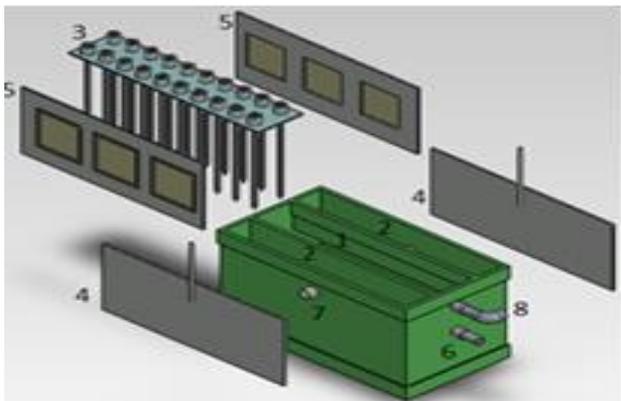


Figure 3: 3D design of the Electrolytic Cell generating oxidizing gas mixture with capacity of 15 A

2.3 CAD design and construction of a chlorine gas mixing system with water by Venturi principle

The chlorination system applied in the project was developed with Venturi tube which is able to generate a suction of emptying in the anode chamber, enough to extract the production of oxidizing gases including chlorine gas, and injecting it directly into the pump-driven water stream, while serving as a mixing chamber between chlorine and water. A shut-off valve was arranged by pass with the Venturi, as shown in the fig. 5, it allows

you to adjust the intensity of the vacuum and create an alternate path to water, decreasing the pump load and increasing its flow rate.

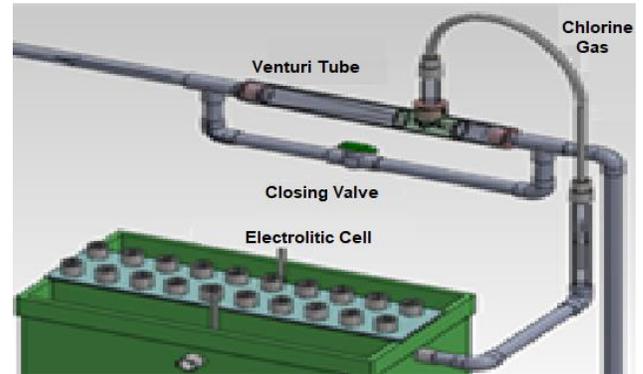


Figure 4: 3D design of chlorine gas mixing with Venturi

2.4 Design and construction of a 25A DC electric power rectifier source

For the supply of current to the electrolytic cell, a DC voltage source with a capacity of 25A was implemented, which is shown in Fig. 6. The source construction is part of the maximum current and voltage values required by the cell.

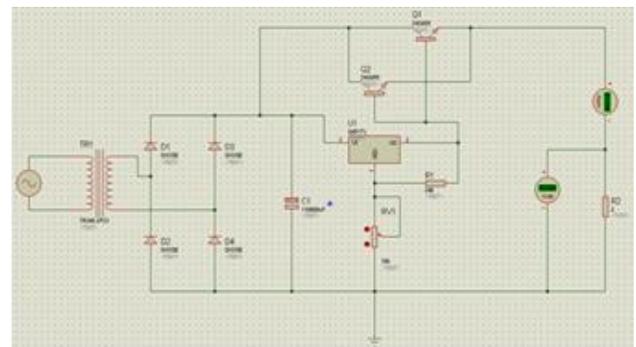


Figure 5: DC source electronic design

2.5 Construction of the sodium chloride solution dosing system

The level control in the reactor brine is based on the application of an operational amplifier working as a voltage comparator circuit, this is shown in the fig. 7.

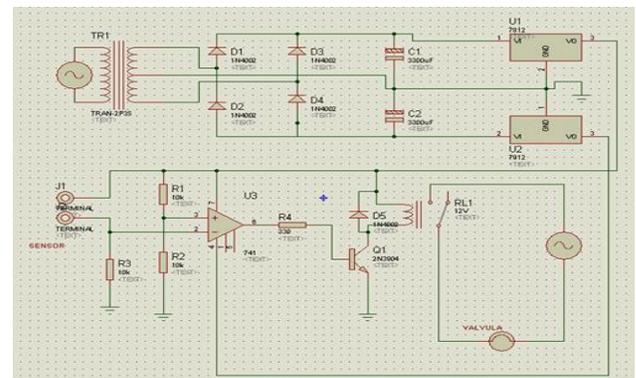


Figure 6: Electronic design of sodium chloride solution dosing system

2.6 Grafset program for the automation of the chlorination plant

The control of the equipment is clearly sequential, as shown in fig. 8, which objective is to turn outputs on and off (DC Source, Pump and Valve) coordinately, depending on the information of the entries (Start, Stop, Level R, Level T). While in its initial stage the program moves towards simultaneous activation of stages 2 and 3 that activate the DC Source and the pump after checking the minimum level in the tank and a call to start with the memory %M5.

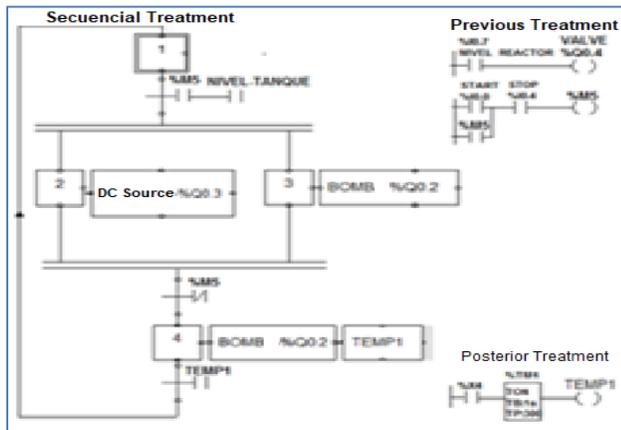


Figure 7: For the automation of the chlorination plant

3 Checking and Verifying the Proposed System Using Statistical Models

The previous study for the design of an electrolyte cell to produce chlorine gas in situ assumes that the most important factors affecting chlorine gas production and its Cf concentration (ppm) in the chlorine gas dissolution tank are:

- **Current:** It is the energy supplied by the DC voltage source (continuous current) and which can break down the molecular structure of the NaCl sodium chloride and thus produce the chlorine gas.
- **Time:** Duration of the process being tested and measured in minutes.
- **Concentration:** Number of grams of sodium chloride dissolved per liter of NaCl grams/liter solution.
- **PH:** It is a measure of acidity or alkalinity of a dissolution. The PH indicates the concentration of hydronium ions (H₃O⁺).

Table 1:

Factors	Low Level	High Level
Current	10A	15A
Time	20 min	40 min
Concentration	45 gr/lit	68 gr/lit
PH:	6.5(H ₃ O ₊)	7.5(H ₃ O ₊)

3.1 Selecting the response variable

The response variable needed by the designer of an electrolyte cell for chlorine gas production is the final concentration of chlorine dissolved in parts per million of Chlorine (PPM Cl₂).

3.2 Experimentation methodology

This study case falls within the analysis of experiment 2 through 4, in which there are 4 main factors, with 2 possible levels for each, the main interest of these tests being to determine which factors have the greatest significance in the response of the model studied, and to rule out the effects of non-significant factors, and is evidenced in Table II. Statistical model 2 through 4 includes 16 possible combinations of treatments for which the response may vary, each combination of treatment indicates the behavior of the output of the system or variable evaluated against random changes in the inputs. The tests were carried out in a Bach-like system. The production of free chlorine in the tank with 200Lt was estimated by applying the titration technique with chemical reagents, using the HTH product KIT. Measurements were made by extracting 8 cm³ of the treated water every 5 minutes and adding 5 drops of the ortho-tolidine reagent HTH® which causes the sample to take yellow hues, from the clearest to 0.5 PPM Cl₂ to the most intense with 5 PPM Cl₂ determining concentration by visual comparison.

Table 2:

CORRIDA	FACTOR				Treat. Comb	FACTOR				Response Variable Free Chlorine PPM Cl ₂
	A	B	C	D		A: CURRENT (AMP)	B: TIME (MIN)	C: Concentration (gr NaCl/lit)	D: PH (H ⁺)	
1	-	-	-	-	[1]	10	20	45	6,5	2,5
2	+	-	-	-	a	15	20	45	6,5	16
3	-	+	-	-	b	10	40	45	6,5	6
4	+	+	-	-	ab	15	40	45	6,5	20
5	-	-	+	-	c	10	20	68	6,5	12
6	+	-	+	-	ac	15	20	68	6,5	18
7	-	+	+	-	bc	10	40	68	6,5	20
8	+	+	+	-	abc	15	40	68	6,5	25
9	-	-	-	+	d	10	20	45	7,5	2
10	+	-	-	+	ad	15	20	45	7,5	15
11	-	+	-	+	bd	10	40	45	7,5	5,5
12	+	+	-	+	abd	15	40	45	7,5	18
13	-	-	+	+	cd	10	20	68	7,5	10
14	+	-	+	+	acd	15	20	68	7,5	17
15	-	+	+	+	bcd	10	40	68	7,5	18
16	+	+	+	+	abcd	15	40	68	7,5	24

Multifactor variance analysis of data was developed with Statgraphics Centurion XV Version 16.1.11 Software. The Anova Multifactorial result for a Maximum Order of Effect of 2 (considering the effect of The Main Factors and Interactions only) shows the results in Table III. In this case, 3 effects and two interactions have a P-value less than 0.05, indicating that they are significantly different from zero with a confidence level of 95.0%. The R-Square statistic indicates that the model explains 98.503% of the variability in Cf (PPM). The adjusted R-square statistic, which is best suited for comparing models with different number of independent variables, is 98.4254%. The Pareto diagram shows graphically in Fig. 9, the magnitude of the effects of factors on the final concentration Cf (ppm) in this case Factor A: Current;

Factor B: Time; Factors C: Concentration; and factor A interactions with C and the interaction of factor B with C. The ANOVA result is confirmed by the Normal Probability chart for the Cf (ppm) response where it is observed that Factor A: Current; Factor B: Time; Factor C: Concentration; and the interactions of A and C; B and C are the most relevant in the system, because they move away from the normal probability line. The distribution of errors is normal indicating that Fig. 10 is a straight line.

Table 3:

Source	Sum of Squares	Df	Mean Square	F-Ratio	P-Value
A: Current	370,563	1	370,563	449,17	0,0000
B: Time	121,0	1	121,0	146,67	0,0000
C: Concentration	217,563	1	217,563	263,71	0,0000
AC	52,5625	1	52,5625	63,71	0,0000
BC	16,0	1	16,0	19,39	0,0013
Total error	8,25	10	0,825		
Total (corr.)	785,938	15			

R-squared = 98,9503 percent; R-squared (adjusted for d.f.) = 98,4254 percent
Standard Error of Est. = 0,908295; Mean absolute error = 0,625

Normal Probability Plot for Cf

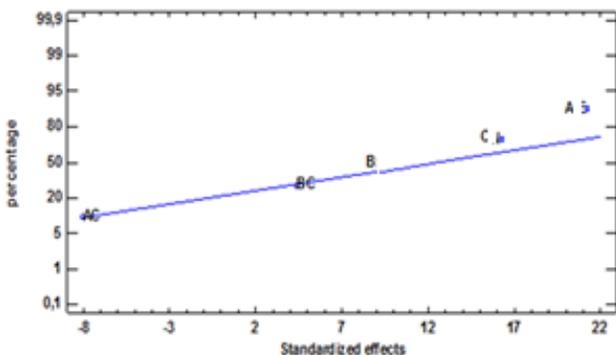


Figure 8: Normal Probability Chart

Table 4:

Coefficient	Estimate
constant	-65,9022
A: CURRENT	5,48696
B: TIME	-0,216304
C: CONCENTRATION	0,847826
AC	-0,0630435
BC	0,00869565

This analysis will only consider FactorS A: Current; Factor B: Time; Factor C: Concentration and interactions of A with C and B with C, shown in Table IV. The No.1 regression equation obtained is: Cf (ppm) = - 65,9022 + 5,48696* Current - 0,216304* Time + 0,847826* Concentration - 0,0630435* Current* Concentration + 0,00869565* Time* Concentration. (See table V). It also yields Cf (PPM) values close to the model and

experiment.

Table 5:

TRATAMIENTO	X1: Current (AMP)	X2: TIME (MIN)	X3: CONCENTRATION N (MOLARIDAD)	PH	RESPONSE VARIABLE (Cf:PPM) EXPERIMENTAL	RESPONSE VARIABLE (Cf:PPM) REGRESSION
1	10	20	45	6,5	2,5	2,8
2	10	20	45	7,5	2	2,8
3	10	20	68	6,5	12	11,2
4	10	20	68	7,5	10	11,2
5	10	40	45	6,5	6	5,8
6	10	40	45	7,5	5,5	5,8
7	10	40	68	6,5	20	18,7
8	10	40	68	7,5	18	18,7
9	15	20	45	6,5	16	15,5
10	15	20	45	7,5	15	15,5
11	15	20	68	6,5	18	17,3
12	15	20	68	7,5	17	17,3
13	15	40	45	6,5	20	19,0
14	15	40	45	7,5	18	19,0
15	15	40	68	6,5	25	24,8
16	15	40	68	7,5	24	24,8

Figure 9 reveals the good correlation between the results of free chlorine measurements against regression values.

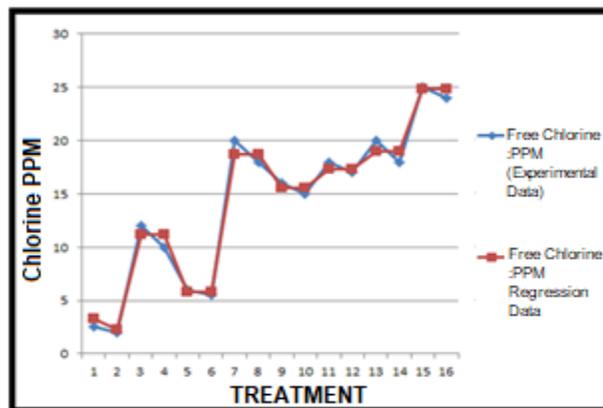


Figure 9: Experimental free chlorine Vs estimated (regression No1)

The number of levels was expanded to three (3) for each significant factor in order to obtain a more reliable regression curve tailored to the model, with a factorial design 33 of 27 treatments, as recorded in TABLE VI. As a result of regression analysis No2, applied to Table 5, a curve is shown in Tables 7 and 8, a curve that only contemplates the most important factors (Current-Time and Concentration).

Regression Model N°2

$$Cf(I, T, C) = - 0,56052632 + 0,27397661 * Current + 0,13055556 * Time + 0,02202643 * Concentration.$$

Noting that the regression coefficient associated with the Concentration factor is very low (0.02202643), a new regression was found that only considers the most significant factors

(Current and Time) resulting in a regression model N°3 simpler of the process, which is evidenced in the tables 9 y 10, fig. 12.

Table 6:

Treatment	FACTOR			VARIABLE DE RESPUESTA		
	I CURRENT (A)	T TIME (MIN)	C Concentration (gr NaCl/litros)	Experimental	Variable	
				Measure Residual Chlorine Cf (PPM)	Response Regression Cf (PPM)	Response Regression Cf (PPM)
1	10	20	45.4	2	1.79	2.04
2	10	20	56.75	2.2	2.04	2.04
3	10	20	68.1	2.5	2.29	2.04
4	10	30	45.4	3	3.10	3.35
5	10	30	56.75	3.2	3.35	3.35
6	10	30	68.1	3.5	3.60	3.35
7	10	40	45.4	4.5	4.40	4.65
8	10	40	56.75	4.6	4.65	4.65
9	10	40	68.1	5	4.90	4.65
10	12	20	45.4	2.4	2.34	2.59
11	12	20	56.75	2.5	2.59	2.59
12	12	20	68.1	3	2.84	2.59
13	12	30	45.4	3.5	3.64	3.89
14	12	30	56.75	4	3.89	3.89
15	12	30	68.1	4	4.14	3.89
16	12	40	45.4	4.8	4.95	5.20
17	12	40	56.75	5	5.20	5.20
18	12	40	68.1	5.2	5.45	5.20
19	15	20	45.4	3	3.16	3.41
20	15	20	56.75	3.2	3.41	3.41
21	15	20	68.1	3.5	3.66	3.41
22	15	30	45.4	4.5	4.47	4.72
23	15	30	56.75	4.8	4.72	4.72
24	15	30	68.1	5	4.97	4.72
25	15	40	45.4	6	5.77	6.02
26	15	40	56.75	6.2	6.02	6.02
27	15	40	68.1	6.5	6.27	6.02

Table 7: Regression statistics No2

Regression statistics No2	
Multiple correlation coefficient	0.992270038
Determination coefficient R ²	0.984599828
Adjusted R ²	0.9825111
Typical error	0.165675926
Observations	27

Table 8: Regression statistics No2

Regression Model No. 2		
	Coefficients	P Value Probability
Interception	-4.5605263	1.62817E-13
Variable X 1 (Current)	0.27397661	7.17884E-15
Variable X 2 (Time)	0.13055556	5.27966E-21
Variable X 3 (Concentration)	0.02202643	1.56206E-06

Regression Model N°3

$$Cf = -3,31052632 + 0,27397661 * \text{Current} + 0,13055556 * \text{Time}$$

Table 9: Regression statistics No3

Regression statistics No3	
Multiple correlation coefficient	0.978343926
Determination coefficient R ²	0.957156837
Adjusted R ²	0.953586574
Typical error	0.27517697
Observations	27

Table 10:

Regression Model No. 3		
	Coefficients	P Value Probability
Interception	-3.310526316	1.62817E-13
Variable X 1 (Current)	0.273976608	1.04431E-10
Variable X 2 (Time)	0.130555556	5.27966E-21
Variable X 3 (Concentration)	0.130555556	1.56206E-06

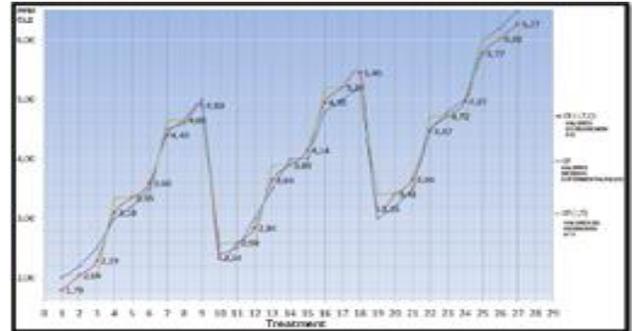


Figure 10: Regression graphs (Experimental, N°2 y N°3)

3.3 Residual analysis

There is no characteristic pattern, this indicates that the residues are independent or randomly distributed. Fig. 13 does not see any pattern or funnel shape, so the data can be considered to satisfy the linearity criterion. The debris fits quite well to a straight. The points (errors) fall close to the straight line, distributed both above and below it, i.e. there is no characteristic pattern, indicating that the residues are independent and randomly distributed, in conclusion, there is normality in the errors.

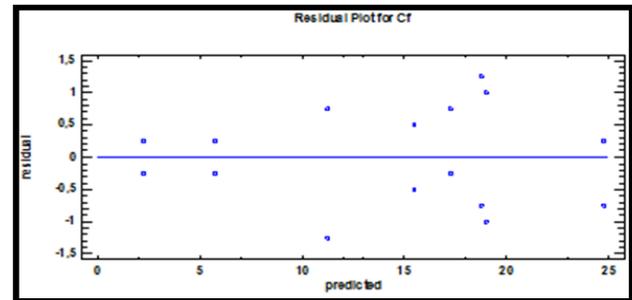


Figure 11: Residual analysis

3.4 Response surface

Three-dimensional response surface where cf (PPM) production of gaseous chlorine is observed based on current and time. In this case there are two influence factors (k-2), the response surface is displayed in a three-dimensional space in which the third dimension represents the expected production of chlorine gas Cf (ppm) on the two-dimensional plane defined by the combinations of the current and time factor levels, shown in Figure 12.

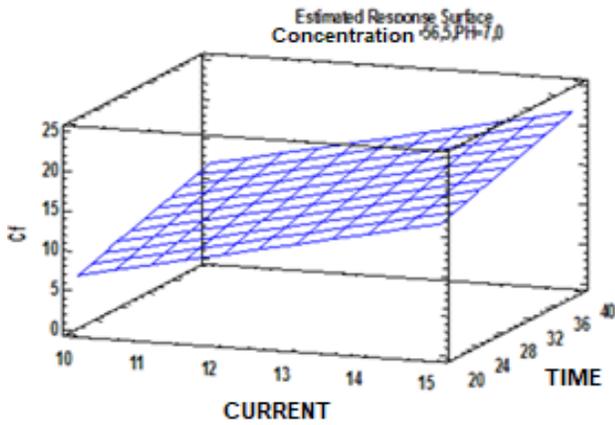


Figure. 12: Response surface

3.5 Equipment performance in closed-circuit recirculation

The team's behaviour was assessed by measuring the increase in Cl₂ concentration by a volume of 200lt that was recirculated at a flow rate of 15.3Lt/min, incrementally capturing the gases generated through the Venturi. And it is evidenced in Table 11 and fig. 15. For this test, the cell was loaded with 20 Lt of brine at a concentration of 45 gr of NaCl/Lt and a current of 8A in the source. It should be noted that these current and concentration levels can be considered as the minimum adjustment levels of the equipment, being able to work at concentrations of 68 gr of NaCl/Lt and 15 A.

Table 11:

TIME (Min)	Free Chlorine: PPM Current I: 8A Concentration: 45grNaCl/Lt
0	0.0
5	1.0
10	1.5
15	2.0
20	3.0
25	4.0
30	5.0
35	6.0
40	8.0
45	9.0
50	9.5
55	10.0
60	10

This test reveals that even at minimum adjustment levels the equipment could leave a 10 PPM footprint on 200lt of water which would amount to saying it would take 2m3 of water to 1 PPM in an hour.

3.6 Open circuit - Performance without recirculation

For this test, the cell was loaded with 20 Lt of brine at a concentration of 45 gr of NaCl/Lt and a current of 10A in the source. The total time of the experiment was 360 min, during this time it is observed the increase in the concentration of the output flow from a value of 1 ppm to the five minutes of the test to a value of 14 ppm at 360 min of the test, keeping this value

containing after this time, this data is shown in TABLE 12 and Fig. 16.

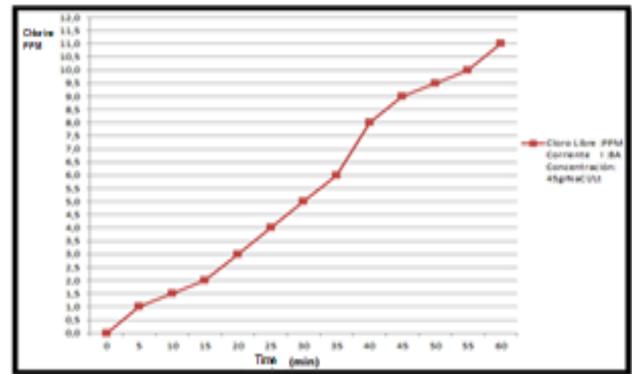


Figure. 13: Closed circuit - Recirculation performance

Table 12:

Chlorine accumulation in a 200Lt water Volume			
Time (Min)	Free Chlorine :PPM Current I:10A Concentration: 45grNaCl/Lt	Time (Min)	Free Chlorine :PPM Current I:10A Concentration: 45grNaCl/Lt
0	0.0	195	8.5
15	1.0	210	9.0
30	1.5	225	9.5
45	2.0	240	10.0
60	3.0	255	11.0
75	3.5	270	11.5
90	4.5	285	12.0
105	5.0	300	12.5
120	6.0	315	13.0
135	6.5	330	13.5
150	7.0	345	14.0
165	7.0	360	14.0
180	8		

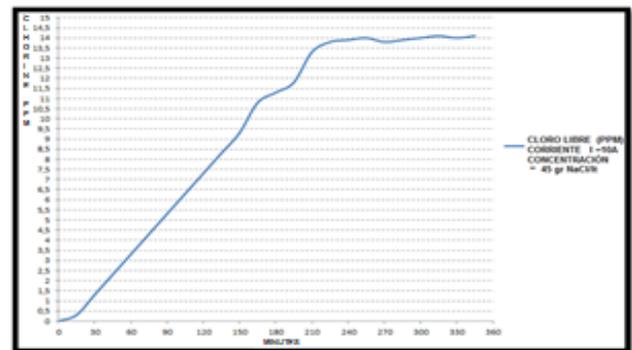


Figure. 14: Open circuit - Performance without recirculation

With these concentration and flow values the equipment can carry a flow rate of 12.8 m3 to 1 ppm of chlorine in one hour. The measurement of equivalent residual chlorine was carried out as follows:

- Water samples were taken every 15 minutes in a specimen, added to the Cl₂ meter by comparison.
- Adding 5 drops of Orthotolidine Hydrochloride.
- The color of the sample was compared to the meter standards

by colorimetric comparison of the sample. 0,5 – 5 PPM.

When the concentration exceeded 5 PPM dilutions were made in which 10 ml of the sample was taken and taken to 20ml, 30ml, 40ml, 50ml, 60ml, with distilled water and this solution samples were taken for comparison. Dilutions were taken into account to calculate the total amount of residual chlorine in treated water.

4 Conclusions

- The prototype meets expected expectations with an efficiency greater than 85%.
- The operation and maintenance of the equipment is very simple. The steps for this procedure would be: Cell washing after each operation, brine supply for a new chlorine gas production, recirculation pump ignition and level control system, automated controller ignition.
- The equipment can work with batteries powered by solar cells. This would be applied in regions where there is no power supply of the electric fluid.
- The equipment can treat flow rates greater than 300 m³/day with a concentration of 1 ppm; this is possible since the electrolyte cell produces other oxidizing gases such as ozone, this increases the ability to water to almost double what is calculated.
- If we assume a consumption of 100 liters per person per day; 3000 people can be served, at a cost of \$27.40 person/month.
- The equipment avoids the need to handle and import cylinders with chlorine gas or liquid sodium hypochlorite drums.

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Ethical issue

Authors are aware of, and comply with, best practice in publication ethics specifically about authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests and compliance with policies on research ethics. Authors adhere to publication requirements that submitted work is original and has not been published elsewhere in any language.

Competing interests

The authors declare that there is no conflict of interest that would prejudice the impartiality of this scientific work of Development of an electrolytic pilot plant to produce chlorine gas “in situ” in the disinfecting water process.

Authors' contribution

All authors of this study have a complete contribution for data collection, data analyses, manuscript writing and translate process.

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