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Research Article

Experimental Study by the Plan Composite Centered Method, Modeling and Optimization of Hydrogen Production by Alkaline Electrolysis

Ahmed Brahmia [1], **Zina Meddeb** [1*]

[1] Mechanical, Modeling, Energy and Materials Research Unit (M2EM), National Engineering School, Gabes University, Rue Omar Ibn Elkhattab Zrig, 6072, Gabes, Tunisia

*** Correspondence:** Zina Meddeb , Mechanical, Modeling, Energy and Materials Research Unit (M2EM), National Engineering School, Gabes University, Rue Omar Ibn Elkhattab Zrig, 6072, Gabes , Tunisia

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Abstract

A planned experimental study on the production of green hydrogen by alkaline electrolysis is carried out by the Plan Composite Centered (PCC) method. The parameters studied are the concentration of the electrolyte, the distance between electrodes, the height of the electrodes, the total supply voltage of the electrolyser, temperature, and the electrolyte type. The results show that the effects of concentration, height, voltage and temperature are positive. However, the effect of the distance between the electrodes is negative. Electrolysis with potassium hydroxide (KOH) is more efficient than with sodium hydroxide (NaOH). The second-order interactions are weak, except for the voltage-temperature interaction which is significant. The results of the experimental study conducted in this work are in agreement with previous studies. Two polynomial models (with KOH and with NaOH) suitable for predicting the flow of hydrogen produced are presented. Three optimizations of ascending constraints on the operating parameters to have a maximum hydrogen production and with a minimum of electrical energy and a minimum of concentration consumed are carried out.

Keywords:
Green hydrogen; alkaline electrolysis; plan of experiments; modelling; optimization

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Introduction

Energy production across the world depends primarily on fossil fuels. This leads to contamination of the environment. An effective alternative to this serious danger is the rapid substitution of fossil fuels, carbon energy sources, by clean renewable energy sources that cause no emissions [1]-[3]. The acceleration of the energy transition is necessary [4]-[7]. It is the gradual transition from carbon-based, polluting energies to clean, renewable, and safe energies. These clean energies are solar, wind, geothermal, hydraulic energy, etc., which meet a series of complementary challenges: the reduction of greenhouse gas emissions; decentralization and redevelopment of infrastructure; reducing inequalities in access to energy and protecting the health of populations. But with all these advantages unfortunately renewable energies also have limitations such as the initial investment being very expensive and the intermittency. Hydrogen as an energy carrier presents itself as a promising solution. There are several sources of hydrogen production with different cleanliness and different colors. The electrolyte is one of the central factors in the electrolyser. This electrolyte must be carefully chosen to reduce the ohmic drops. For industries that need hydrogen in their production process, there is the possibility of producing hydrogen of renewable origin by electrolysis [8]. The principle is to install an electrolyser on site, supplied with green electricity (solar and wind), as well as a storage unit. There are several methods and several types of hydrogen production by electrolysis. However, the yield of hydrogen production by electrolysis remains more or less low and further research on it should be carried out [9]-[11]. The work of this article is articulated in this context.

The studies carried out on alkaline electrolysis are numerous but none of them are planned; each author studies one parameter while keeping the others constant.

In this work a planned parametric study using the centered experimental design method is conducted. To predict the response, we have to do a model. To increase the efficiency of hydrogen production by electrolysis, the optimal operating parameters must be identified; therefore, an optimization of these parameters is sought.

Methodology

The design of experiments method consists in establishing an experimental plan comprising the minimum number of experiments taking into account the desired results. The main advantages of this method compared to traditional methods of experimentation are as follows: reduction in the number of tests, possibility of studying the effects of a very large number of factors, detection of possible interactions between factors, determination of the results with good precision, make a reduction of the answer with a modeling and the possibility of making an optimization [12]-[18]. The response matrix of the design of experiments is the rate of hydrogen production.

The main purpose of the Plan Composite Centered (PCC) is to mathematically model the studied responses in the form of a 2nd order polynomial equation and to optimize them. This method is also called the Box-Wilson type design which uses the response surface methodology and is used for continuous variables. The Design-Expert software 18 is used.

Plan Composite Centered

The main purpose of the Plan Composite Centered (PCC) is to mathematically model the studied responses in the form of a 2nd order polynomial equation and to optimize them. This method is also called the Box-Wilson type design which uses the response surface methodology and is used for continuous variables. The Design-Expert software 18 is used.

Total number of trials

A composite plan consists of three parts:

Trials of the two-level full factorial design (coded ∓ 1). The number of experiments

$N_1 = 2^n$ (with n: number of factors)

Star trials (coded $\mp\alpha$). α is the distance from the axial points to the center. The number of experiments $N_2 = 2n$; the distance α is calculated by the statistical method:

$$\alpha = \sqrt[4]{2^n}$$

Trials at the center of the domain (coded 0). The number of experiments N_3 this number is used to assess the reproducibility of experiments.

Choice of factors

In our study, the answer is the flow of hydrogen produced. The number of factors chosen is five: the concentration of the electrolyte in the electrolyser, the distance between the electrodes, the height of the electrode immersed in the solution, the total supply voltage, the temperature of the solution and the type of electrolytes. Therefore, the total number of experiments is Ntotal=50 experiences (N1=32, N_2=10 et N3=8). We repeated this test matrix twice, once with potassium hydroxide (KOH) electrolyte and once with sodium hydroxide (NaOH) electrolyte (50+50=100 experiences).

Factor variation interval

The variation interval of each factor is deduced on the one hand from the bibliographical study and on the other hand from the experimental constraints. The five factors and their ranges of variation are listed in Table 1:

Experimental protocol

After we fixed the factors and the necessary experimental plan, we carried out the experimental tests to fill the two matrices (with KOH and with NaOH). Figure 1 gives a schematic representation of the experimental device. The response studied is the rate of hydrogen production. This flow rate is calculated indirectly by calculating the time (t) necessary for the production of a fixed volume of hydrogen for all the tests. Figure 2 and Figure 3 respectively represent the electrodes used and the distances between them. The hydrogen recovery tube is placed above the cathode.

Table 1. Factor values at different levels

Factor level	Factor level			
	- α	-1	0	1
A: Concentration (mol/l)	0.31	1	1.5	2
B: Distance between electrodes (cm)	3.24	6	8	10
C: Electrode height (cm)	1.62	3	4	5
D: Tension (V)	3.62	5	6	7
E: Temperature (°C)	39.66	50	57.5	65

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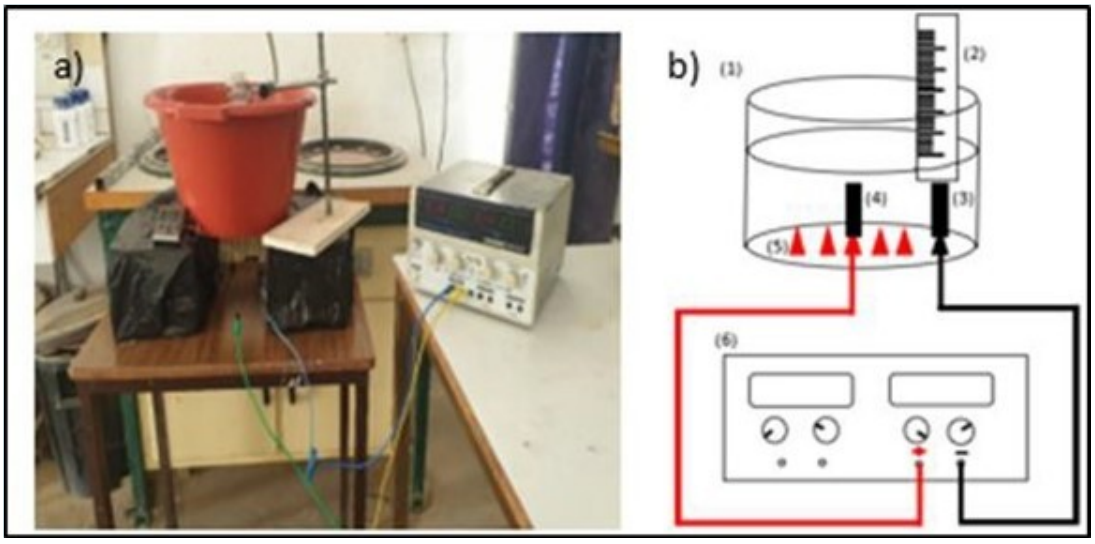

Figure 1. Experimental device: (1) reservoir, (2) test tube, (3) cathode, (4) anode, (5) alligator clip, (6) voltage generator.

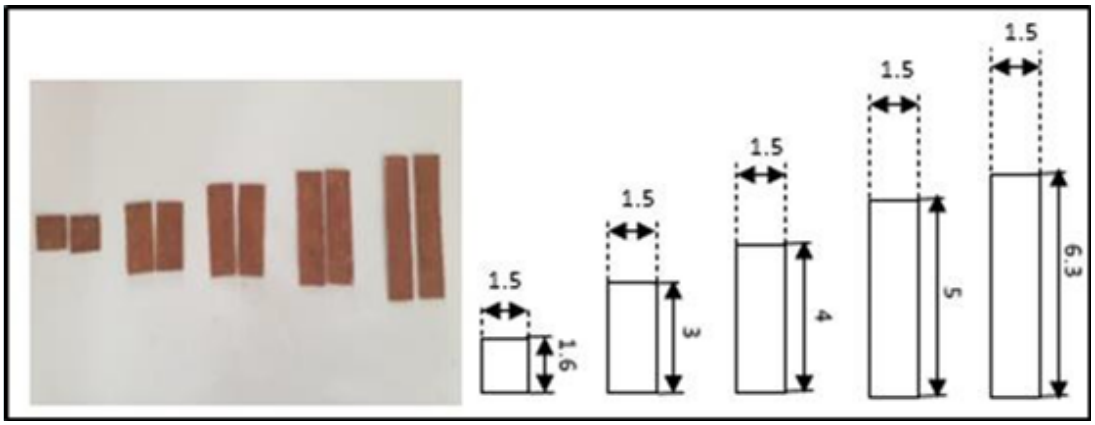

Figure 2. Heights of electrodes used.

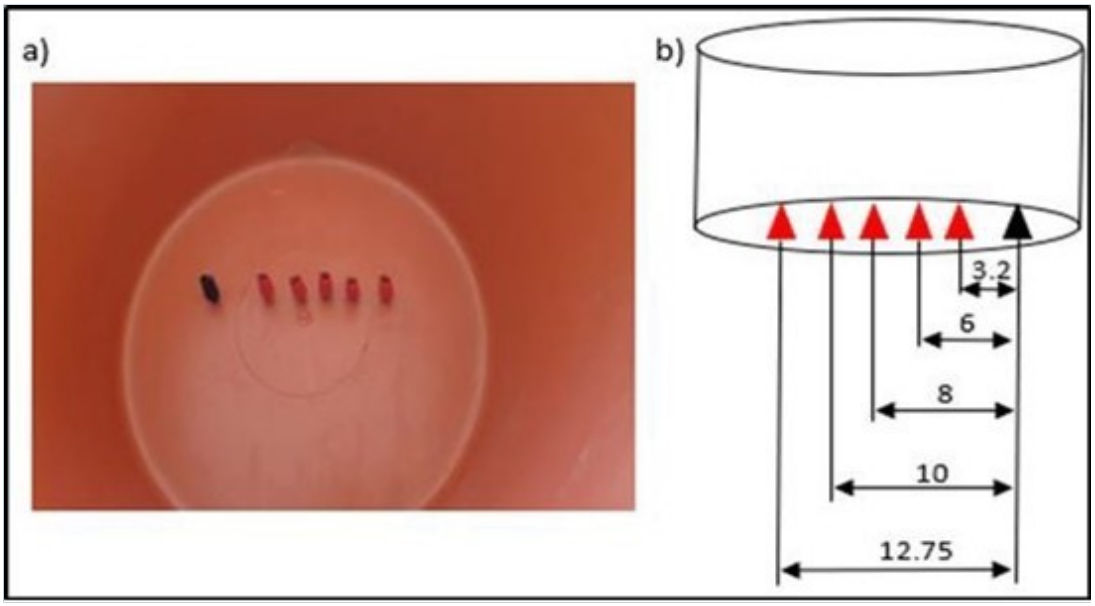

Figure 3. Preparing the electrode sites.

To carry out the necessary experiments, we first prepared a tank with the five distances between the electrodes (- α, -1, 0, +1 et + α) where we placed crocodile clips. Then we prepared the solutions with the requested concentrations. We heated the solution to the required temperatures. We carried out the experiments following an order where there is the minimum of the chemicals used.

Results and discussion

Filling the experience matrix

The experimental results found for the volume flow of hydrogen produced are grouped in Table 2.

Table 2. Experimental results

Number of experience	Flow rate (ml/min)	
	with KOH	with NaOH
1	0.184706	0.09696
2	0,207318	0,105009
3	0,143133	0,090897
4	0,185099	0,095343
5	0,230176	0,066979
6	0,249283	0,105197
7	0,167715	0,088113
8	0,170097	0,101885
9	0,378215	0,199641
10	0,424809	0,219925
11	0,318674	0,168563
12	0,378	0,237727
13	0,433557	0,236798
14	0,449035	0,290276
15	0,338983	0,174474
16	0,350324	0,206058
17	0,188324	0,129803
18	0,239378	0,168919
19	0,179953	0,090897
20	0,200844	0,167827
21	0,254162	0,1344
22	0,319693	0,195599
23	0,167715	0,096656
24	0,343761	0,130736
25	0,402982	0,282765

Table 3a. Choice of the suitable model (the electrolyte KOH)

Source	Sequential P-value	lack of adjustment P-value	R2	Adjusted R2	predicted R2	
Linear	< 0.0001	0.0029	0.7772	0.7519	0.7183	
2FI	0.9856	0.0014	0.7931	0.7018	0.6042	
Quadratic	0.0018	0.0061	0.8899	0.8139	0.5395	Suggested
Cubic	0.9661	0.0008	0.9213	0.7245	-9.0284	

Table 3b. Choice of the suitable model (the electrolyte NaOH)

Source	Sequential P-value	lack of adjustment P-value	R2	Adjusted R2	predicted R2	
Linear	< 0.0001	0.0004	0.8689	0.854	0.8273	
2FI	0.728	0.0003	0.8909	0.8428	0.8167	
Quadratic	<0.0001	0.0028	0.9547	0.9235	0.8161	Suggested
Cubic	0.2259	0.003	0.9827	0.9393	-0.7385	

Choice of model type

The mathematical model is a polynomial model according to the coded factors and applicable to the defined experimental domain. To choose the suggested model, the R^2 value must be on the one hand close to 1 and on the other hand it is necessary that R^2 and R^2 adjusted do not differ considerably; if not then there is a strong chance that there are insignificant terms in the model. Table 3a and Table 3b respectively give the analysis results for the choice of the suitable model for the tests with KOH and for the tests with NaOH. We note that the quadratic model is the suggested model for both types of electrolytes.

ANOVA analysis

Table 4a and Table 4b present the ANOVA analysis of variance to judge the performance of the model obtained successively for KOH and for NaOH. Based on the ANOVA analysis, we can conclude that:

The two models (for KOH and for NaOH) are validated by the Fischer test.

The significant parameters if the electrolyte is KOH are A, B, C, D, E, B^2 and D^2

The significant parameters if the electrolyte is NaOH are A, B, D, E, DE, D^2 and E^2

Effects and Interactions

The effects and interactions on the response are grouped in Table 5a and Table 5b.

The histograms of figure 4a and figure 4b give a comparison between the different effects and interactions respectively with the electrolyte KOH and NaOH.

From the ANOVA analysis, we can note that:

Concentration has a positive and significant effect regardless of the electrolyte used. This shows that if the concentration of the electrolyte increases, the flow rate increases. This is explained by the fact that if the concentration increases then the charge transfer increases. These results are in agreement with the results of Fatima ezzahra Chakik et al [19], who studied the effect of electrolyte concentration on hydrogen production. They used a solution of NaOH with different concentrations, and they found that as long as the concentration matters so much the production.

The distance between the electrodes has a negative and significant effect for both types of electrolytes. That is, if the distance between the electrodes increases then the hydrogen production rate decreases. Indeed, increasing the distance between the electrodes increases the path traveled by the charges, so it

Table 4a. ANOVA analysis (the electrolyte KOH)

Source	Sum of squares	Freedom degree	Mean of Square	Ratio-F	Valeur-p Prob > F	Statistical significance
Model	0.4711	20	0.0236	11.71	< 0.0001	Significant
A concentration	0.0185	1	0.0185	9.21	0.005	Significant
B Distance	0.0164	1	0.0164	8.14	0.0079	Significant
C Height	0.01	1	0.01	4.97	0.0337	Significant
D Voltage	0.3569	1	0.3569	177.48	< 0.0001	Significant
E Temperature	0.0097	1	0.0097	4.83	0.0362	Not significant
AB	0.0001	1	0.0001	0.0261	0.8727	Not significant
AC	0	1	0	0.019	0.8912	Not significant
AD	0.0006	1	0.0006	0.2832	0.5986	Not significant
AE	0.0016	1	0.0016	0.7729	0.3866	Not significant
BC	0.001	1	0.001	0.476	0.4957	Not significant
BD	0.0013	1	0.0013	0.6405	0.4301	Not significant
BE	0.001	1	0.001	0.476	0.4957	Not significant
CD	0.0019	1	0.0019	0.9482	0.3382	Not significant
CE	0.0008	1	0.0008	0.4129	0.5255	Significant
DE	0.0003	1	0.0003	0.1344	0.7166	Not significant
A^2	0.002	1	0.002	1	0.3252	Significant
B^2	428	1	428	21.3	<0.0001	Not significant
C^2	0.0114	1	0.0114	0.9152	0.3467	Significant
D^2	0.0018	1	0.0018	5.67	0.024	Significant
E^2	0.0064	1	0.0064	3.17	0.0856	Not significant

limits the production speed. N. Nagai et al [20] have shown that if the space between the electrodes increases then the electrical resistance increases and the efficiency of electrolysis decreases.

The supply voltage has a positive effect and is the most important parameter regardless of the electrolyte used (which is noticeable in Figures 4-a and 4-b). Dayana D'arc of Fatima Palhares et al [21] found that if the tension increases the production also increases. The same result is found by Kenji Kikuchi et al [22]

The operating temperature has a positive and significant effect whatever the electrolyte used. Yangyang Li et al [23], Boissonneau et al [24] and Damien le Bideau [25] studied the effect of temperature on the efficiency of electrolysis, and they found that if the temperature increases then the production increase. Yangyang Li et al [26] experimentally studied the effect of temperature and pressure under different current densities; they found that if the operating temperature increases, the voltage required, for the same amount of hydrogen production, decreases. We can explain this by the fact that increasing the operating temperature increases the activity of the catalyst.

The height of the electrodes has a positive and significant effect if the electrolyte is KOH. If the electrolyte is NaOH, the effect of the height of the electrodes is also positive but not significant.

From our results, we can notice very clearly that electrolysis with KOH is more efficient than electrolysis with NaOH. As long as the diameter of the electrolyte is large as long as the transfer is faster. These results are in good agreement with the results of M. Hassen sellami et al [27]. They experimentally studied the effect of the nature of the electrolyte on the volume of hydrogen produced. We can increase the efficiency of electrolysis by using KOH (since it is more efficient) not in pure water but in another solution (for example water from the air conditioning system or wastewater) [28]. This shows that the

Table 4b. ANOVA analysis (the electrolyte NaOH)

Source	Sum of Squares	Freedom degree	Mean of squares	Ratio-F	Valeur-p Prob > F	Statastical Significance
Model	0.3493	20	0.0175	30.56	< 0.0001	Significant
A concentration	0.0245	1	0.0245	42.91	< 0.0001	Significant
B Distance	0.0114	1	0.0114	20	0.0001	Significant
C Height	0.0003	1	0.0003	0.4439	0.5105	Not significant
D Voltage	0.2479	1	0.2479	433.84	< 0.0001	Significant
E Temperature	0.0338	1	0.0338	59.07	< 0.0001	Significant
AB	0.0002	1	0.0002	0.3327	0.5685	Not significant
AC	0.0001	1	0.0001	0.2102	0.65	Not significant
AD	0.0007	1	0.0007	1.17	0.2892	Not significant
AE	0.0015	1	0.0015	2.6	0.1178	Not significant
BC	0.001	1	0.001	1.77	0.1935	Not significant
BD	0.0008	1	0.0008	1.4	0.2463	Not significant
BE	0.0005	1	0.0005	0.896	0.3517	Not significant
CD	0.0004	1	0.0004	0.686	0.4143	Not significant
CE	0.0001	1	0.0001	0.1479	0.7034	Not significant
DE	0.0028	1	0.0028	4.92	0.0345	Significant
A^2	0.0001	1	0.0001	0.1182	0.7335	Not significant
B^2	0.0012	1	0.0012	2.01	0.1666	Not significant
C^2	0.0001	1	0.0001	0.1379	0.7131	Not significant
D^2	0.0154	1	0.0154	26.99	< 0.0001	Significant
E^2	0.0101	1	0.0101	17.66	0.0002	Significant

number of valence electrons in the electrolyte has an important effect, and the application of a magnetic field can promote the transfer [29]. The concern that: in a stoichiometric approach, it takes 9 kg of water

Table 5a. Effects and Interactions of factors (KOH)

Effects		Interactions		Quadratic effects	
x_0	0.2249	x_{12}	0.0013	x_{11}	0.006
x_1	0.0207	x_{13}	-0.0011	x_{22}	0.0278
x_2	-0.0194	x_{14}	-0.0042	x_{33}	0.0058
x_3	0.0152	x_{15}	0.007	x_{44}	0.0143
x_4	0.0908	x_{23}	-0.0055	x_{55}	0.0107
x_5	0.015	x_{24}	-0.0063		
		x_{25}	0.0055		
		x_{34}	-0.0077		
		x_{35}	0.0051		
		x_{45}	-0.0029		

Table 5b. Effects and Interactions of factors (NaOH)

Effects		Interactions		Quadratic effects	
x_0	0.1556	x_{12}	0.0024	x_{11}	0.0011
x_1	0.0238	x_{13}	0.0019	x_{22}	0.0045
x_2	-0.0162	x_{14}	0.0046	x_{33}	0.0012
x_3	0.0024	x_{15}	0.0068	x_{44}	0.0167
x_4	0.0757	x_{23}	-0.0056	x_{55}	0.0135
x_5	0.0279	x_{24}	-0.005		
		x_{25}	-0.004		
		x_{34}	0.0035		
		x_{35}	-0.0016		
		x_{45}	0.0094		

to produce 1 kg of hydrogen; however, after taking into consideration the inefficiencies of the process the water consumption amounts to 18-24 kg per kg of hydrogen [30]-[31]; will no longer be the case, especially if wastewater is used in the electrolyser [32].

According to the ANOVA analysis, we notice that no interaction of order 2 is significant with the KOH electrolyte. The only significant interaction found is DE (voltage-temperature) with the electrolyte NaOH. Figure 5 gives a representation of the response surfaces of these interactions with the NaOH electrolyte. For five factors we have ten interactions of order 2 (AB, AC, AD, AE, BC, BD, BE, CD, CE, DE).

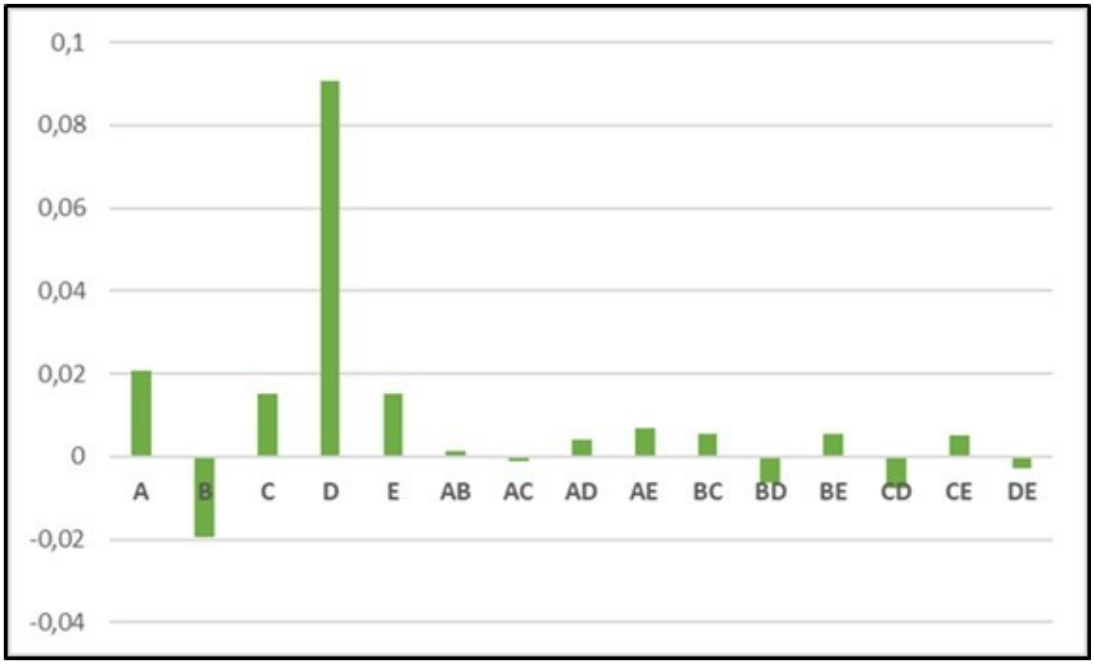

Figure 4a. Histogram of comparison between the different effects and interactions (with KOH)

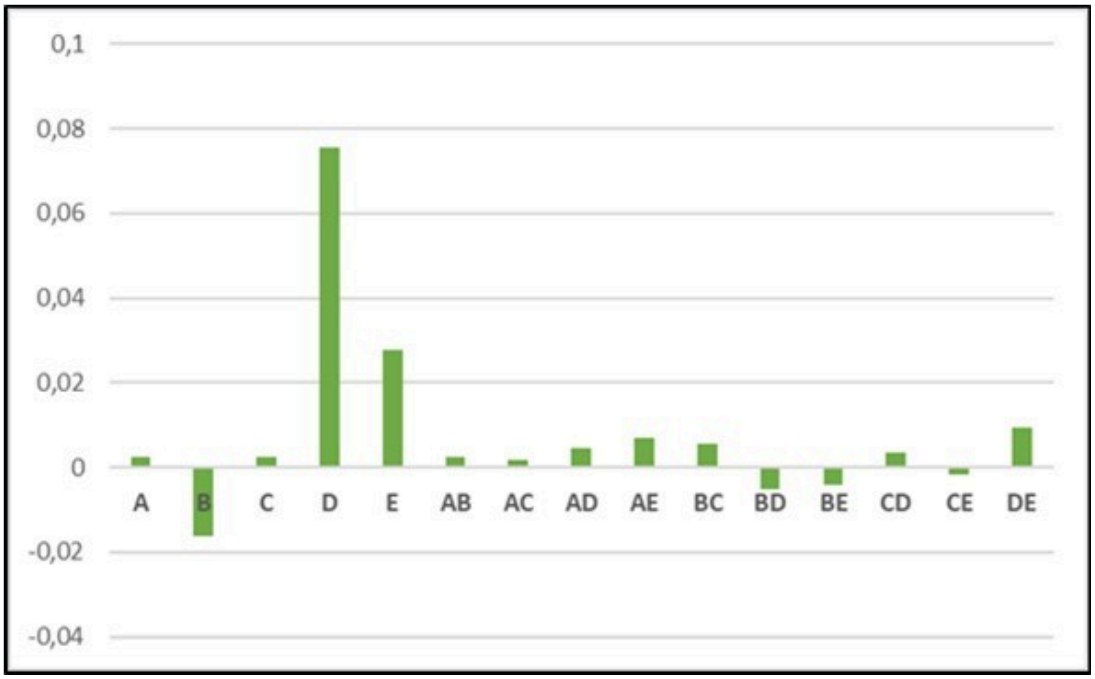

Figure 4b. Histogram of comparison between the different effects and interactions (with NaOH)

Modelization

The equation in terms of coded factors can be used to make predictions about rethink for given levels of each factor. By default, higher levels of factors are coded as +1 and lower levels are coded as -1. The coded equation is useful for determining the relative impact of factors by comparing factor coefficients.

Complete model

The quadratic complete model used is given by the following equation:

$$q = x_0 + x_1 A + x_2 B + x_3 C + x_4 D + x_5 E + x_{12} AB + x_{13} AC + x_{14} AD + x_{15} AE + x_{23} BC + x_{24} BD + x_{25} BE + x_{34} CD + x_{35} CE + x_{45} DE + x_{11} A^2 + x_{22} B^2 + x_{33} C^2 + x_{44} D^2 + x_{55} E^2$$

with:

x_0, x_1, x_2, x_3, x_4, x_5: average effects of variables respectively A, B, C, D, E.

x_{12}, x_{13}, x_{14}, x_{15}, x_{23}, x_{24}, x_{25}, x_{34}, x_{35}, x_{45}: effect of variable interactions.

x_{11}, x_{22}, x_{33}, x_{44}, x_{55}: quadratic effects of variables.

Reduced model

We can improve the complete model by eliminating insignificant factors from the complete model and we obtain a so-called reduced model.

If the electrolyte is KOH the reduced model is:

$$q = 0.2249 + 0.0207\,A - 0.0194\,B + 0.0152\,C + 0.0908\,D + 0.0150\,E + 0.0278\,B^2 + 0.0143\,D^2$$

If the electrolyte is NaOH the reduced model is:

$$q = 0.1556 + 0.0238\,A - 0.0162\,B + 0.0757\,D + 0.0279\,E + 0.0094\,DE + 0.0167\,D^2 + 0.0107\,E^2$$

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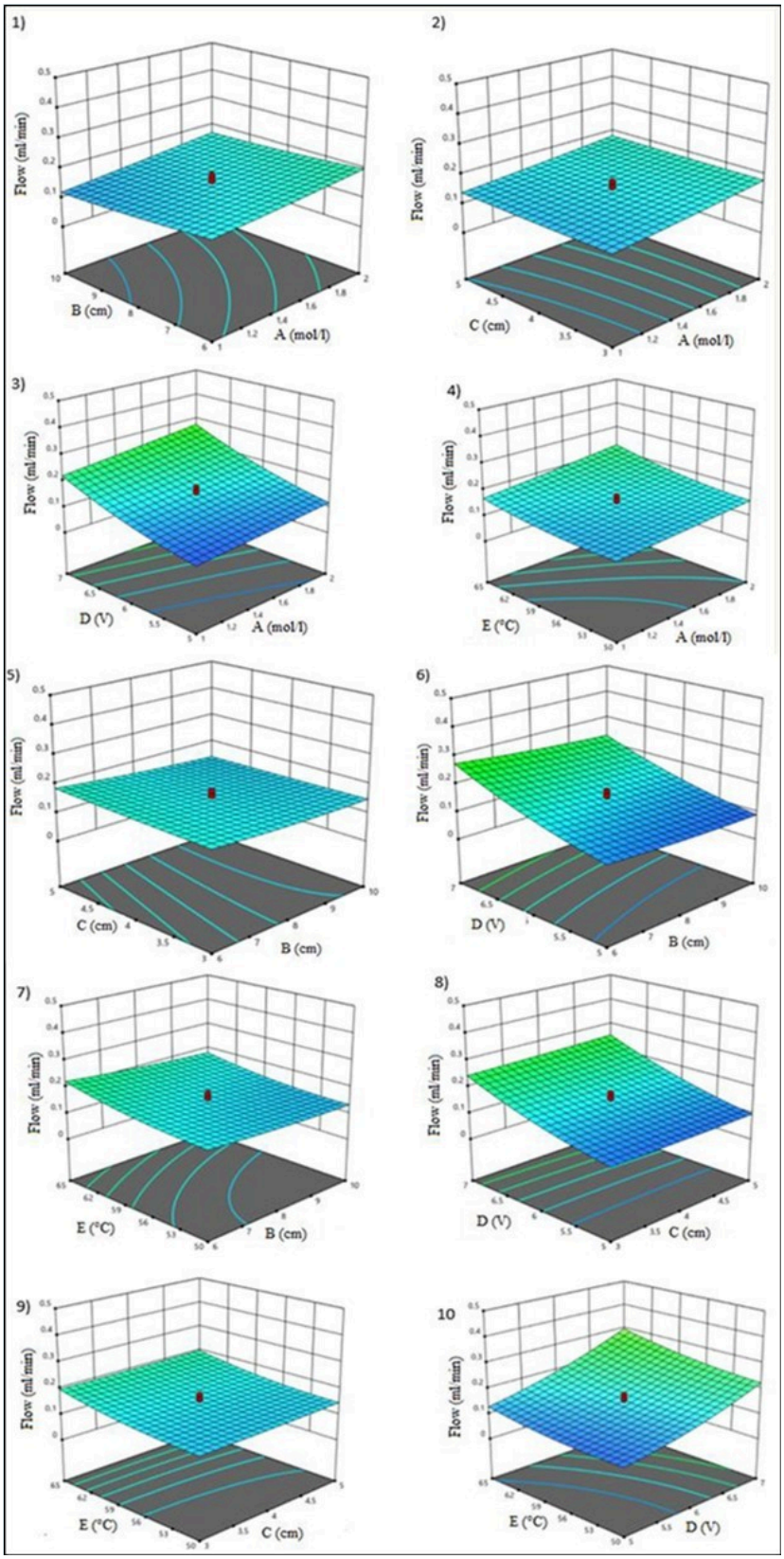

Figure 5. Response surfaces of combined effects (ten Interactions)

Model adequacy

To study the adequacy of the found model, we draw the curve which represents the values given by the model according to the experimental values. Figure 5-a and Figure 5-b respectively show the adequacy of the model if the electrolyte is KOH and if the electrolyte is NaOH.

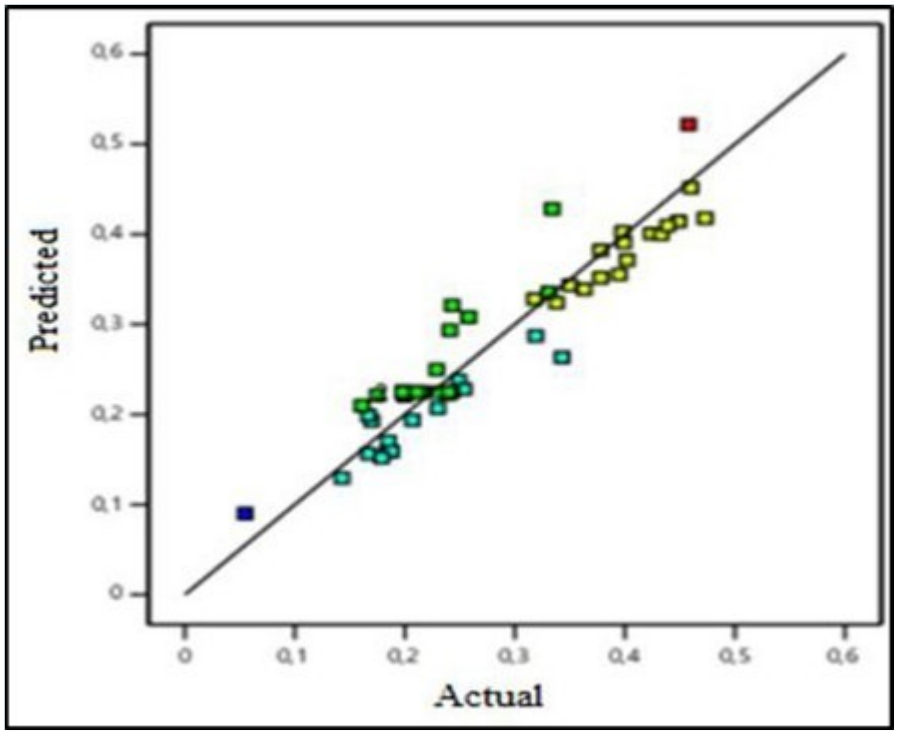

Figure 5a. Volume flow predicted as a function of actual volume flow (KOH)

Optimization

Optimization seeks a combination of factor levels that simultaneously meet the established criteria for a best desired response. Table 6 represents the objectives, the importance of each objective and the results.

In this study three optimizations are carried out:

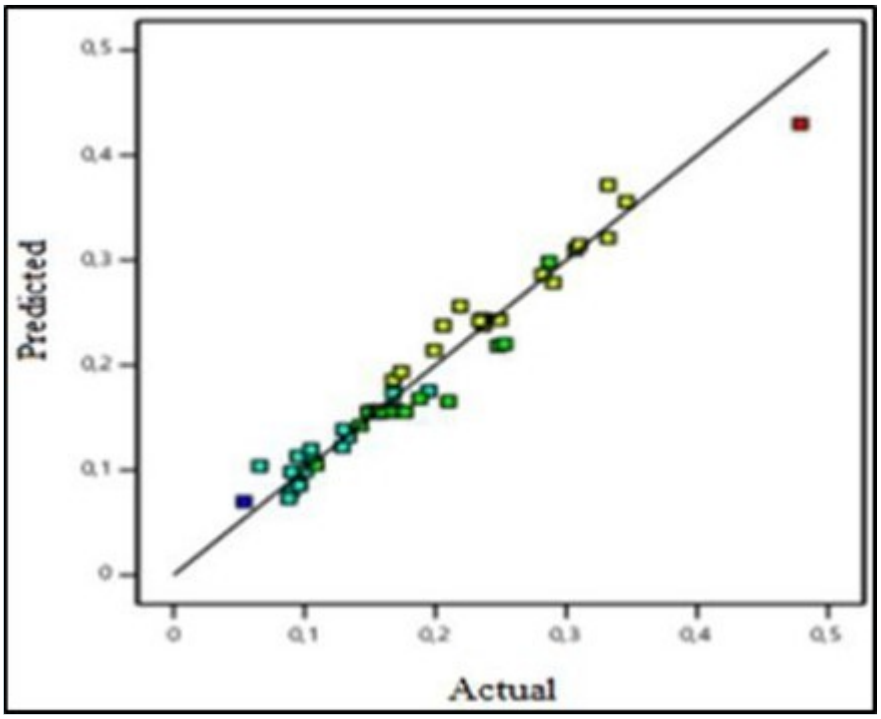

Figure 5b. Volume flow predicted as a function of actual volume flow (NaOH)

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Table 6. Result of different optimizations

		Objective	Importance	Results
Optimization 1	A: Concentration (mol/l)	-	-	2
	B: Distance between electrodes	-	-	6
	(cm)			
	C: Height (cm)	-	-	5
	D: Voltage (V)	-	-	7
	E: Temperature (°C)	-	-	65
	Hydrogen flow rate (ml/min)	Maximize	5	0.372
Optimization 2	A: Concentration (mol/l)	-	-	2
	B: Distance between electrodes	-	-	6
	(cm)			
	C: Height (cm)	-	-	4.99
	D: Voltage (V)	Minimize	2	6.167
	E: Temperature (°C)	-	-	65
	Hydrogen flow rate (ml/min)	Maximize	5	0.274
Optimization 3	A: Concentration (mol/l)	Minimize	2	1
	B: Distance between electrodes	-	-	6
	(cm)			
	C: Height (cm)	-	-	5
	D: Voltage (V)	Minimize	2	6.262
	E: Temperature (°C)	-	-	65
	Hydrogen flow rate (ml/min)	Maximize	5	0.229

Optimization 1: The objective in this optimization is to maximize hydrogen production independently of other parameters. That is, to maximize the hydrogen production rate and the other parameters are random.

Optimization 2: In this optimization we will keep the same objective as the first optimization, and we add the condition to minimize the supply voltage of the electrolyser.

Optimization 3: In this optimization we will keep the same objective as the first optimization, and we add the condition to minimize the supply voltage of the electrolyzer and the condition to also minimize the concentration of the electrolyte.

Three optimizations of ascending constraints on the operating parameters to have a maximum hydrogen production are carried out. We notice that if we increase the constraints on the optimized parameters, the value of the response (Hydrogen flow rate) decreases.

Conclusion

A planned experimental study on the production of green hydrogen by alkaline electrolysis is carried out by the Centered Composite Plan (PCC) method. The parameters studied are the concentration, the distance between electrodes, the height, the voltage, the temperature, and the type of electrolytes. The results show that the effects of the concentration, the height of the electrodes, the total voltage and the temperature are positive whatever the type of electrolyte. So, increasing these parameters increases hydrogen production. However, the effect of the distance between the electrodes is negative, so an increase in this distance leads to a decrease in the production of hydrogen. The interactions between the different parameters are weak and the only significant interaction is the voltage-temperature interaction. The experimental study conducted in this work gives a good agreement with previous studies. Two models (with KOH and with NaOH) suitable for predicting the flow of hydrogen produced are presented. Three optimizations of ascending constraints on the operating parameters to have a maximum hydrogen production are carried out.

Competing Interests

no conflict of interest

Acknowledgments

no conflict of interest

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