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Procedure for the Elimination of Remazol Black by Coagulation-Flocculation Based on the Methodology of Experimental Designs

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ABSTRACT

Dyes are omnipresent in everyday life, in clothing, cosmetics and food. Synthetic dyes, recalcitrant in nature, pose an environmental problem. The objective of this work is to find, from experimental designs, the optimal conditions for the elimination of Remazol black by coagulation-flocculation. The method used is based exclusively on experimental designs. After the Hadamard design for the identification of the factors influencing the phenomenon, we used several factorial designs to optimize the treatment. The results allowed us to retain three factors: the concentration of the dye (Remazol), the concentration of the coagulant (Al^{3+}) and the volume of coagulant (VAl^{3+}) which influence the elimination of Remazol in solution. A modeling of the same phenomenon using a composite design (surface and response design) was then carried out to optimize these factors. The conditions are as follows: 38.77 mg/L for the dye concentration, 41.3 g/L for the coagulant concentration and finally 1.7 mL for the coagulant volume. These optimal conditions made it possible to obtain a Remazol reduction rate of 98.15 %.

Keywords: Dye, Experimental design, Remazol Black, synthetic solutions, reduction rate, optimization, modeling.

INTRODUCTION

The use of dyes in industry has increased in recent decades. This growing demand is forcing humanity to synthesize its own dyes that were once natural [1]. Among these dyes is Remazol Black. Unfortunately, after use, some of these dyes end up in the environment, causing negative consequences for terrestrial and aquatic biodiversity. Indeed, dyes contain chemical molecules that are difficult to biodegrade [2]. Several researchers here and elsewhere have conducted studies to eliminate these recalcitrant molecules from dyes. This is the case of researchers such as [3,4]; and [5] who used electrocoagulation to eliminate methylene blue and nitrate respectively at 95.15%. In addition to electrocoagulation, some have used passive methods such as membrane, activated carbon and clays. Among these researchers there are [6] who used a member based on two physical copolymers polysulfone (PSU) and polyetherimide (PEI) to eliminate cibacrone red at pH = 6.75 and indigo at pH = 11.30, respectively at 71.20 % and 85.36 % [6] in water. Also, Gore Bi et al [7] used activated carbon to eliminate gentian violet at 96.5 %. As for Martínez Stagnaro et al [8], they used clay nanoparticles to eliminate dyes from textiles. Coagulation flocculation which is not a new method is often used for the elimination of dyes in wastewater [9]. However, in view of the elimination percentages, no method leads to neutralization of dyes in the discharges. Hence the motivation of researchers to continue research with a view to achieving total clarification of colored water discharged into nature. It is with this in mind that this study was initiated. Its objective is to determine the factors influencing elimination by coagulation-flocculation and to find the optimal conditions for total elimination of the dye.

MATERIALS & METHODS

II-1. Experimental Design

The experimental design corresponds to the set of experimental conditions imposed on the factors. The formal mathematical structure of which the experimental design is a real-world application is called an experimental matrix [10]. Furthermore, there are several experimental design methods: the full factorial design (FFD), the fractional factorial design (FFD), the central composite design (CCD), the so-called PLACKETT-BURMAN design or HADAMARD design, etc. Similarly, there are several specialized experimental design software programs: SAS, NEMRODW, the Microsoft Office Excel Solver utility, etc. In our study, we will use NEMRODW software.

II-3. Experimental Planning

The phenomenon studied is the elimination of Remazol black from water. The response is the elimination rate of Remazol black. This elimination will be achieved by coagulation-flocculation using a VELP Scientific JLT6 JAR TEST. Several factors are involved in this phenomenon. The factors considered are: Remazol black concentration (X_1), Remazol black volume (X_2), coagulant type (X_3), coagulant concentration (X_4), and coagulant volume (X_5). These five (5) factors initially lead us to adopt the Hadamard design.

II-3-1. Hadamard Design

Hadamard matrices are optimal matrices for non-interacting experimental designs. This type of design allows for an initial assessment of the influence of factors on the experimental response with very few tests to be performed, even for a large number of significant factors. It was chosen for screening, i.e., to detect the relative "weights" of factors on the measured response. Thus, the choice of a screening design as an initial approach is justified by the objective of separating the factors into several classes according to their influence on the elimination of Remazol black. This initial design should allow for a rapid determination of which of the various factors considered have the greatest influence on the response. The choice of this design leads us to the experimental domain presented in Table 1.

Table 1: Experimental Domain of the Hadamard Design

Factors	Designation	Low level (-)	High level (+)
U1	Remazol Black concentration	20	50
U2	Remazol black volume	300	500
U3	Coagulation type	Fe^{3+}	Al^{3+}
U4	Coagulant concentration	50	100
U5	Coagulant volume	3	5

The five (5) factors, each with two levels, allow for a matrix of eight (8) trials summarized in Table 2. For each factor, the high level is rated +1 and the low level is rated -1

Table 2: Hadamard Design Experiment Matrix

N° Exp	X_1	X_2	X_3	X_4	X_5
1	1	1	1	-1	1
2	-1	1	1	1	-1
3	-1	-1	1	1	1
4	1	-1	-1	1	1
5	-1	1	-1	-1	1
6	1	-1	1	-1	-1
7	1	1	-1	1	-1
8	-1	-1	-1	-1	-1

The response studied is a linear function of all factors (eq. 1):

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_4X_4 + a_5X_5$$
eq. 1

With a_i representing the effect of factor Xi. The various coefficients were calculated using NEMROD-W version 9901 software, along with the standard deviations and calculated responses (Ycalc.). Significance tests for each coefficient were performed by considering that a coefficient is statistically significant if its absolute value is greater than 2σ (σ being the standard deviation). By replacing each coded value in the experimental matrix with its actual value, we obtain the experimental design presented in Table 3.

Table 3: Experimental plan of the Hadamard design

N° Exp	RGB Concentration (mg/L)	RGB Volume (mL)	Coagulant	Concentration of coagulant (g/L)	Volume of coagulant (mL)
1	50	500	Al^{3+}	50	5
2	20	500	Al^{3+}	100	3
3	20	300	Al^{3+}	100	5
4	50	300	Fe^{3+}	100	5
5	20	500	Fe^{3+}	50	5
6	50	300	Al^{3+}	50	3
7	50	500	Fe^{3+}	100	3
8	20	300	Fe^{3+}	50	3

The Hadamard design only allows the factors to be separated into several classes. This will distinguish between important, medium and negligible factors. The determination of the influential factors in the area studied led us to the establishment of a second experimental design. This is the full factorial design. It allows us to calculate the average effect, the main effects of the factors and their interactions 2 to 2, 3 to 3, etc. The main objective is to search for the optimal conditions for the elimination of Remazol black in an aqueous medium.

II-3-2. Three-factor full factorial design

Since the first experimental design (namely the Hadamard design) has highlighted the factors influencing the phenomenon, we will use the full factorial design to attempt to optimize dye removal. This will involve studying the mean effect, main effects, and interactions. Three factors were retained in this design, taking into account the results of the Hadamard design. These are: the concentration of Remazol Black (X_1), the concentration of Al^{3+} (X_2), and the volume of Al_{3+} (X_3). Table 4 presents the established experimental domain.

Table 4: Experimental domain.

Factors	Designation	Low level (-)	High level (+)
U1	Concentration of Remazol Black	30	75
U2	Concentration of coagulant	50	75
U3	Volume of coagulant	2	4

These three (3) factors, each at two levels, make it possible to obtain the factorial matrix of 23 (8) tests summarized in table 5.

Table 5: Full factorial matrix

N° Exp	X_1	X_2	X_3
1	-1	-1	-1
2	1	-1	-1
3	-1	1	-1
4	1	1	-1
5	-1	-1	1
6	1	-1	1
7	-1	1	1
8	1	1	1

By replacing each coded value with its real value, we obtain the experimental plan in table 6 below:

Table 6: Experimental plan of the complete factorial design 1

N° Exp	Concentration of RGB	Concentration of Al^{3+}	Volume of Al^{3+}
	(mg/L)	(g/L)	(mL)
1	30	50	2
2	75	50	2
3	30	75	2
4	75	75	2
5	30	50	4
6	75	50	4
7	30	75	4
8	75	75	4

The response studied is a linear function of all factors (eq. 2)

$$Y = a_0 + a_1X_1 + a_2X_2 + \cdots + a_kX_k + a_{12}X_1X_2 + \cdots + a_{k-1k}X_{k-1}X_k + \cdots + a_{1\cdots k}X_1X_2\cdots X_k$$

With ai representing the effect of factor X_i and a_{ij} representing the interaction between factors i and j.

The results obtained led to the implementation of another full factorial design.

II-3-3. Full Factorial Design with Addition of H_2O_2

Studies conducted by [11] have shown an improvement in the removal rate of Remazol by adding hydrogen peroxide (H_2O_2) during its degradation. We will also verify whether the addition of H_2O_2 can also improve the yield in the case of coagulation-flocculation. This consisted of adding 30 % hydrogen peroxide (H_2O_2) to the aqueous dye solution. This gave us four factors, the experimental range of which is presented in Table 7.

Table 7: Experimental Domain of the Factorial Design with H_2O_2

Factors	Designation	Low level (-)	High level (+)
U1	Concentration of Remazol Black	30	75
U2	Concentration of coagulant	50	75
U3	Volume of coagulant	2	4
U4	Volume of H_2O_2	3	5

These four (4) factors, each at two levels, make it possible to obtain the factorial matrix of 16 tests summarized in table 8.

Table 8: Full factorial matrix 2

N° Exp	X_1	X_2	X_3	X_4
1	-1	-1	-1	-1
2	1	-1	-1	-1

3	-1	1	-1	-1
4	1	1	-1	-1
5	-1	-1	1	-1
6	1	-1	1	-1
7	-1	1	1	-1
8	1	1	1	-1
9	-1	-1	-1	1
10	1	-1	-1	1
11	-1	1	-1	1
12	1	1	-1	1
13	-1	-1	1	1
14	1	-1	1	1
15	-1	1	1	1
16	1	1	1	1

By replacing each coded value with its actual value, we obtain the experimental plan in Table 9 below:

Table 9: Experimental plan for the complete factorial design 2

N° Exp	Concentration of RGB (mg/L)	Concentration of Al^{3+} (g/L)	Volume of Al^{3+} (mL)	Volume of H_2O_2 (mL)
1	30	50	2	3
2	75	50	2	3
3	30	75	2	3
4	75	75	2	3
5	30	50	4	3
6	75	50	4	3
7	30	75	4	3
8	75	75	4	3
9	30	50	2	5
10	75	50	2	5
11	30	75	2	5
12	75	75	2	5
13	30	50	4	5
14	75	50	4	5
15	30	75	4	5
16	75	75	4	5

II-3. Implementation of Coagulation-Flocculation

Coagulation and flocculation are performed using a VELP Scientific JLT6 JAR TEST. A quantity of coagulant is added to a volume of dye of known concentration. The assembly is installed in the jar test and set at 175 rpm for 2 minutes. Thus, the agglomeration of micro-flocs into larger flocs (flocculation) is achieved by setting the jar test at a speed of 50 rpm for 20 minutes. After 2 hours of rest, the solution is decanted and subsequently filtered using filter paper before reading the concentration using the HACH DR 3900 spectrophotometer at a wavelength of 560 nm.

III. RESULTS AND DISCUSSION

III-1. Analysis of the results from factor screening using the Hadamard matrix

Table 10 presents the results of the various experiments conducted for the Hadamard design. The reduction rates range from 46.20 % to 76.50 %. The highest rate was obtained during Experiment 2. This involved the treatment of 500 mL of Remazol at 20 mg/L with 5 mL of Al^{3+} at a concentration of 50 g/L as a coagulant.

Table 10: Reduction rates of Remazol black using the Hadamard design

N° Experience	Reduction Rate (%)
1	67.20
2	76.50
3	68.00
4	46.20
5	70.00
6	68.00
7	56.20
	74.00

III-1-1. Statistical Analyses

The statistical analysis of this model initially leads to the analysis of variance table (Table 11).

Table 11: Analysis of variance

Source of variation	Sum of squares	Degrees of liberty	Mean square	Ratio	Signif.
Regression	684.19	5	136,83	87.64	1.13 *
Residuals	3.12	2	1.56		
Total	687.31	7			

It mainly indicates that the model used is well adjusted. Indeed, the error due to the residuals (the adjustment error) is very low. The sum of squares due to the error (3.12) is very insignificant compared to the total sum of squares (687.31) due to the regression. This good adjustment is confirmed by the more detailed analysis given by the value of the multiple linear correlation coefficient (R^2 = 0.995 or 99.5 %, given by the software). Thus, the adjustment achieved is satisfactory [10].

III-1-2. Coefficient Estimates and Statistics

The coefficient estimates and statistics are presented in Table 12.

Table 12: Coefficient Estimates and Statistics of the Response Y (Hadamard Design)

	a_0	a_1	a_2	a_3	a_4	a_5
Coefficients	65.762	-6.362	1.712	4.162	-4.037	-2.912
Standard deviation	0.442	0.442	0.442	0.442	0.442	0.442

The average reduction rate is expressed by a_0 and is equal to 65.762 %. The «+» or «– » signs in front of the coefficients indicate the increase or decrease, respectively, in the reduction rate when the factor studied varies from the low level to the high level. The experimental error (standard deviation) obtained is 0.442. Analyzing Table X, we see that all factors have an influence on the phenomenon in our field of study because the absolute value of their coefficient is greater than $2*\sigma = 0.882$ [12].

When the concentration of Remazol black increases from 20 to 50 mg/L, the reduction rate decreases by 6.362×2 or 12.724 %. However, when the volume of the dye increases from 200 to 500 mL, the reduction rate increases by 1.712×2 or 3.424 %. Moreover, the reduction rate increases by 4.162×2 or 8.324 % when switching from iron III (Fe3+) to aluminum III (Al^{3+}) as coagulant. This result is consistent with the use of aluminum sulfate by [6] in the treatment of Congo red by coagulation-flocculation.

It is important to note that, when the coagulant concentration also increases from 50 to 100 g/L, the reduction rate decreases by 4.037×2 or 8.074 %, while the reduction rate decreases by 2.912×2 or 5.824 % when the amount of coagulant added increases from 3 to 5 mL.

The dye volume has little influence on the reduction rate, which is why we continued our work using the maximum volume of 500 mL. By fixing the dye volume and the coagulant concentration, we are at three factors; hence the use of the full factorial design. For this purpose, we defined another domain of study.

III-2. Analysis of the results from the full factorial design

III-2-1. In the absence of H_2O_2 as a factor in the experimental domain

The average reduction rate is expressed by a_0 and is equal to 65.762 %. The «+» or «–» signs in front of the coefficients indicate the increase or decrease, respectively, in the reduction rate when the factor studied varies from the low level to the high level. The experimental error (standard deviation) obtained is 0.442. Analyzing Table X, we see that all factors have an influence on the phenomenon in our field of study because the absolute value of their coefficient is greater than $2*\sigma = 0.882$ [12]. Indeed:

When the concentration of Remazol Black increases from 20 to 50 mg/L, the reduction rate decreases by 6.362×2, or 12.724 %, and when the dye volume increases from 200 to 500 mL, the reduction rate increases by 1.712×2, or 3.424 %. The reduction rate also increases by 4.162×2, or 8.324 %, when switching from iron III (Fe3+) to aluminium III (Al^{3+}) as the coagulant. This result is consistent with the use of aluminum sulfate by [6] in the treatment of Congo red by coagulation-flocculation.

However, when the coagulant concentration increases from 50 to 100 g/L, the reduction rate decreases by 4.037 x 2, or 8.074 %, and the reduction rate also decreases by 2.912 x 2, or 5.824 %, when the amount of coagulant added increases from 3 to 5 mL.

The dye volume has little influence on the reduction rate, which is why we continued our work using the maximum volume of 500 mL. By fixing the dye volume and the coagulant concentration, we are at 3 factors; hence the use of the full factorial design. For this purpose, we defined another area of study.

III-2. Analysis of the results from the full factorial design

III-2-1. In the absence of H2O2 as a factor in the experimental domain

The results of the different experiments are presented in Table 13. The reduction rate varies from 51.20 % to 82.70 %. The highest rate is obtained during experiment 1 for 500 mL of dye at 30 mg/L in the presence of 2 mL of Al^{3+} at 50 g/L.

Table 13: Remazol black reduction rate with the full factorial design

N° Experience	Reduction Rate (%)
1	82.70
2	66.00
3	74.70
4	62.10
5	71.00
6	62.50
7	65.30
8	51.20

In the chosen area, we notice a clear improvement in treatment compared to the Hadamard design.

III-2-1-1. Statistical Analysis

The statistical analysis of this model initially leads to the analysis of variance table (Table 14).

Table 14: Analysis of Variance

Source of variation	Sum of squares	Degrees of liberty	Mean square	Ratio	Signif
Regression	607.7775	6	101.2963	8.6127	25.5
Residuals	11.7612	1	11.7612		
Total	619.5388	7			

It mainly indicates that the model used is well adjusted. Indeed, the error due to the residuals (the adjustment error) is very low. The sum of squares due to the error (11.76) is very insignificant compared to the total sum of squares (619.53) due to the regression. This good adjustment is confirmed by the more detailed analysis given by the value of the multiple linear correlation coefficient (R^2 = 0.981 or 98.1% given by the software). Thus, the adjustment achieved is satisfactory.

III-2-1-2. Coefficient Estimates and Statistics

The 8 experiments yielded 7 coefficients. Table 15 presents the estimates and statistics for these coefficients. The average coefficient (a_0) gives the average reduction rate obtained over the 8 experiments. Its value is 66.94 %. This rate is significant because the value is above the average of 50 %. The three (3) main effects related to the factors influencing this study are marked with a "-" sign, indicating that the reduction rate decreases as we move from the lowest level to the highest level.
Coefficients a_{12}, a_{13}, and a_{23} are the interaction effects between the different factors. Considering that a coefficient is significant if its absolute value is greater than 2×σ (σ being the standard deviation), the interaction effects are not significant.

Table 15: Estimates and statistics of the coefficients of the response Y.

Name	Coefficient	F. Inflation	Standard deviation	t. exp.	Signi f. %
a_0	66,937		1,212	55,21	1,15 *
a_1	-6,487	1,00	1,212	-5,35	11,8
a_2	-3,612	1,00	1,212	-2,98	20,6
a_3	-4,437	1,00	1,212	-3,66	17,0
a_{12}	-0,187	1,00	1,212	-0,15	90,2
a_{13}	0,838	1,00	1,212	0,69	61,5
a_{23}	-0,638	1,00	1,212	-0,53	69,2

Figure 1 shows the contribution of different factors to the elimination of Remazol black from the solution. This figure represents the Pareto chart of the different factors.
A Pareto chart is a graph representing the importance of different causes of a phenomenon. It allows us to highlight the most important causes out of the total number of effects and thus take targeted measures to improve a situation. Thus, the factor with the greatest contribution is identified by coefficient a1, i.e., the concentration of Remazol black in the solution, which has a contribution of 42.08%, followed by the volume of the coagulant, with a contribution of 19.68 %, and finally the concentration of the coagulant, which is 13.04 %.

It is important to note that when the dye concentration increases from 30 to 75 mg/L, the reduction rate decreases by 12.96 %. It is also noted that the concentration of Al^{3+} increases from 50 to 75 g/L, the reduction rate decreases by 7.22 % but also, when the volume of Al^{3+} solution poured increases from 2 to 4 mL, the reduction rate decreases by 8.86 %. In general, in this field of experimentation, a decrease in the reduction rate is noted when there is an increase in the influencing factors. This field is therefore not appropriate for a good elimination rate of this dye.
Based on the analysis made on the significance of the coefficients, we therefore have the mathematical equation

$$Y = 66.937 - 6.487X_1 - 3.612X_2 - 4.437X_3.$$

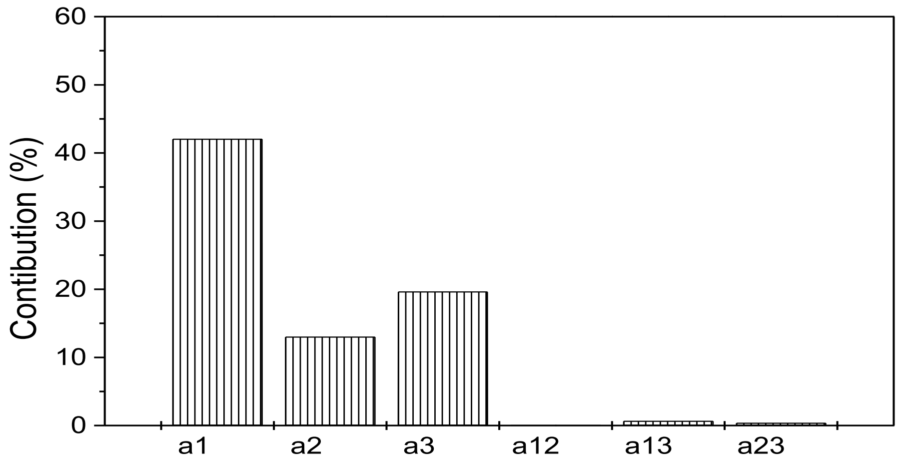

Figure 1: Pareto analysis graph of the effects of different factors

The higher the dye concentration, the lower the reduction rate. It would therefore be appropriate to work at low concentrations. However, studies in [10] have shown that pre-oxidation of remazol allows an improvement in the reduction rate. We will therefore use the same factors in the same definition areas by adding hydrogen peroxide.

III-2-2. In the presence of H_2O_2 as a factor in the experimental domain

The second full factorial design led to 16 experiments, the results of which are presented in Table 16. The reduction rates ranged from 51.10 % to 82.30 %. The highest rate was obtained during experiment 1 for 500 mL of dye at 30 mg/L in the presence of 2 mL of Al^{3+} at 50 g/L and 3 mL of hydrogen peroxide. The presence of hydrogen peroxide did not improve the coagulation-flocculation treatment.

Table 16: Remazol black reduction rate obtained

N° Expérience	Reduction Rate (%)
1	82.30
2	60.90
3	75.30
4	60.90
5	69.00
6	59.10
7	58.60
8	54.00
9	76.00
10	64.80
11	74.30
12	58.10
13	66.00
14	60.90
15	57.30
16	51.10

III-2-2-1. Statistical Analysis of the Results

The analysis of Table 17 shows that the model used fits well. Comparing the error due to the residuals (the adjustment error) and the total sum of squares due to the regression, we see that the sum of squares due to the error (2.52 × 101) is very insignificant compared to the total sum of squares (1.16 × 103) due to the regression.

Table 17: Analyse of variance

Source of variation	Sum of squares	Degrees of liberty	Mean square	Ratio	Signi f.
Regression	$1.14.10^3$	10	$1.14.10^2$	22.6298	0.151 **
Residuals	$2.52.10^1$	5	5.05		
Total	$1.16.10^3$	15			

This good adjustment is confirmed by the more detailed analysis given by the value of the multiple linear correlation coefficient (R^2 = 0.978 or 97.8 %, given by the software). Thus, the adjustment achieved is satisfactory.

III-2-2-2. Coefficient Estimations and Statistics

III-2-2-2-1. Coefficient Study

The second experimental design led to 16 experiments. After data processing using the NEMRODW software, 10 coefficients were generated (Table 18). The average reduction rate represented by a_0 is 64.29 %. This rate is above the average of 50 %. The four (4) main effects related to the factors influencing this study are marked with a "-" sign, indicating that the reduction rate decreases when moving from the lowest level to the highest level. The interaction effects between the different factors are represented by the coefficients a_{ij} (i and j ∈N^*). A coefficient is significant if its absolute value is greater than 2×σ (σ being the standard deviation) [13].

Table 18: Estimates and statistics of the coefficients of the response *Y*

Name	Coefficient	F. Inflation	Standard Deviation	t. exp.	Signi f. %
a_0	64.287		0.562	114.38	< 0.01 ***
a_1	-5.562	1.00	0.562	-9.90	0.0180 ***
a_2	-3.088	1.00	0.562	-5.49	0.273 **
a_3	-4.788	1.00	0.562	-8.52	0.0367 ***
a_4	-0.725	1.00	0.562	-1.29	25.4
a_{12}	0.388	1.00	0.562	0.69	52.1
a_{13}	2.337	1.00	0.562	4.16	0.883 **
a_{23}	-1.163	1.00	0.562	-2.07	9.3
a_{14}	0/725	1.00	0.562	1.29	25.4
a_{24}	-0.275	1.00	0.562	-0.49	64.5
a_{34}	0.050	1.00	0.562	0.09	93.3

The contribution of each factor on the elimination of remazol black is given by the Pareto diagram in Figure 2.

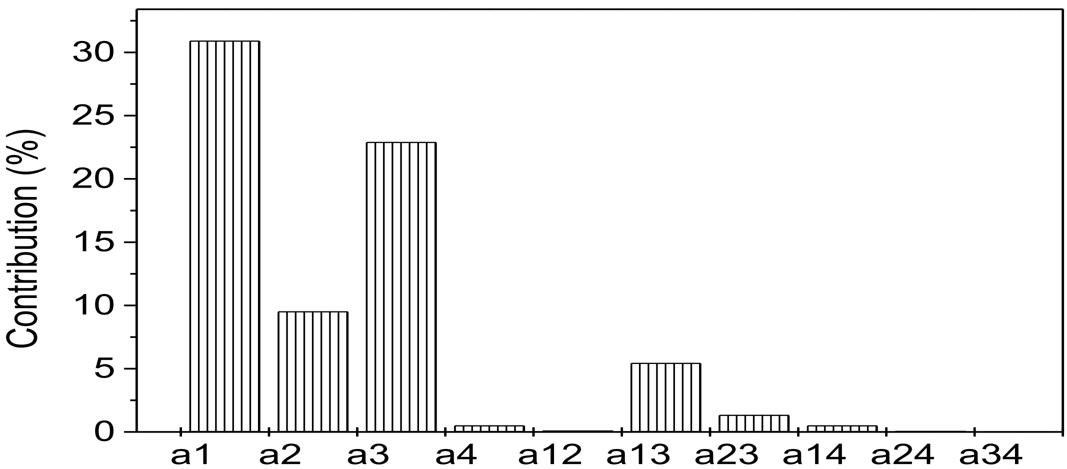

Figure 2: Pareto analysis graph of the effects of different factors

Factor a_1, which represents the remazol concentration, contributes 30.9 % to its elimination in the aqueous medium. This is followed, respectively, by the volume of Al^{3+} with a contribution of 22.9 %, the concentration of Al^{3+} with a contribution of 9.5 %, the interaction between the concentration of remazol and the volume of Al^{3+}, and the interaction between the concentration of Al^{3+} and the volume of Al^{3+}. When the dye concentration increases from 30 to 75 mg/L, the reduction rate decreases by 11.12 %. When the concentration of Al^{3+} increases also from 50 to 75 g/L, the reduction rate decreases by 6.17 %. And when the volume of Al^{3+} solution added increases from 2 to 4 mL, the reduction rate decreases by 9.58 %.

III-2-2-2-1. Study of interaction effects

- **Interaction between Remazol concentration and Al3+ volume**
  Figure 3 shows the effect of the interaction between Remazol concentration and Al^{3+} volume on the response studied.

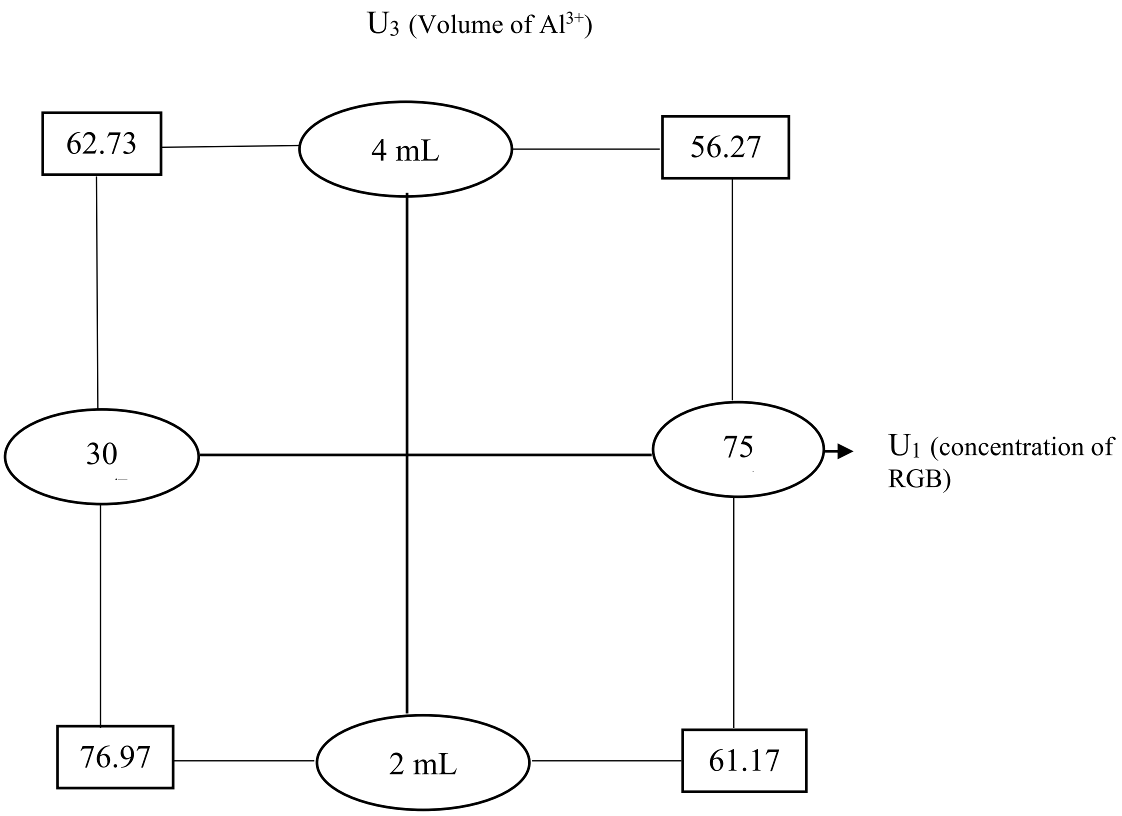

Figure 3: Graph of the effect of the Remazol concentration-Al^{3+} volume interaction

Remazol is eliminated at 76.97 % for a concentration of 30 mg/L in the presence of 2 mL of Al^{3+}. This rate represents the maximum remazol removal capacity for all combinations performed. Under the same remazol concentration conditions, the elimination rate is 62.73 % for a volume of 4 mL. Remazol elimination is better for a low concentration in aqueous media with a low volume of Al^{3+}.

- ***Al^{3+} concentration-Al^{3+} volume of interaction***
  The effect of the Al3+ concentration-Al3+ volume interaction on the studied response is presented in the following figure.

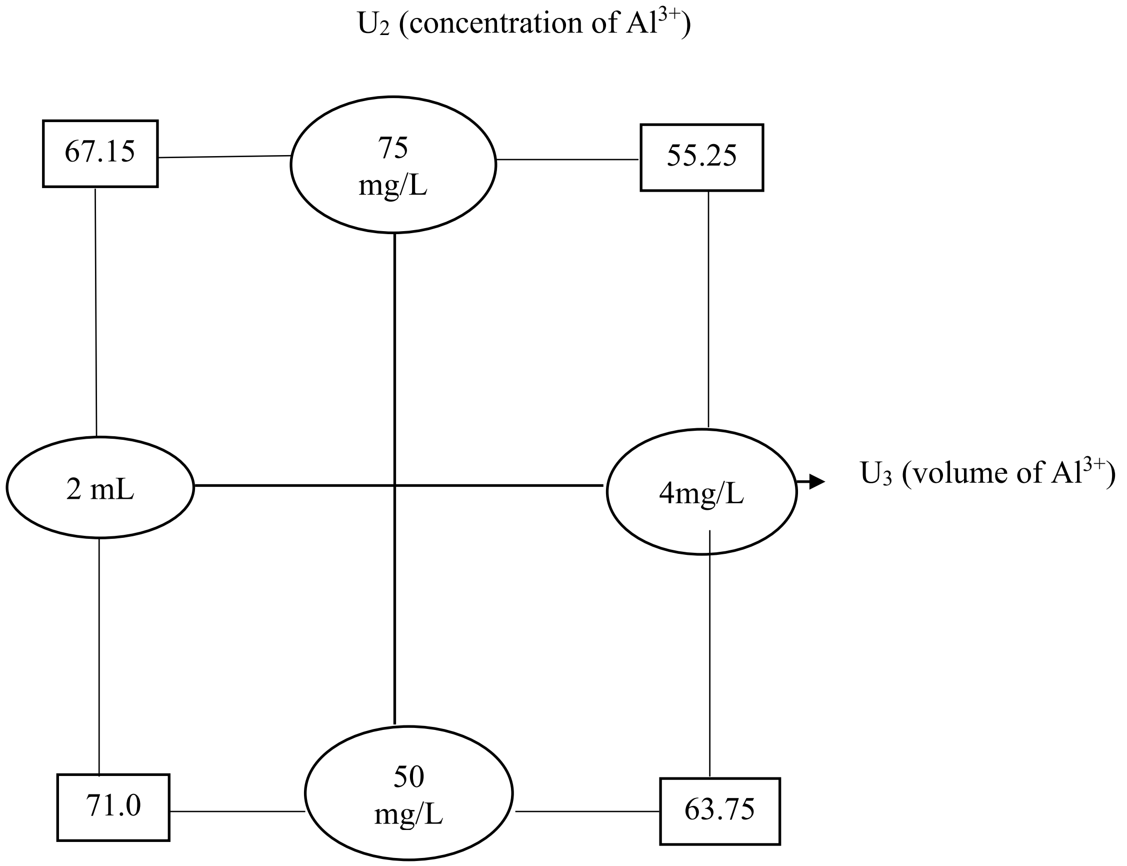

Figure 4: Graph of the effect of the interaction between Al^{3+} concentration and Al^{3+} volume

The maximum response (71 %) is obtained for an Al^{3+} concentration of 50 g/L and a volume of 2 mL. The lowest rate is obtained for an Al^{3+} concentration of 75 g/L and a volume of 4 mL. It is also noted that the highest rates are for the smallest volume of Al^{3+}. The best removal rate therefore depends on the volume of Al^{3+} applied.
The resulting mathematical equation is (eq. 3):

$$Y = 64.287 - 5.562X_1 - 3.088X_2 - 4.788X_3 + 2.337X_1X_3 - 1.163X_2X_3$$

III-2-2-2-2. Residual Analysis

Table 19 of the residuals allows us to assess the quality of the fit achieved. Comparing the values of the measured responses (Yexp.) with those of the responses predicted by the model (Ycalc) shows that the fit is of good quality. Indeed, any "studentized" residual greater than 2 (in absolute value) reflects a significant lack of fit. The results obtained indicate, apart from Experiment 9, that the values of the studentized residuals are less than 2 (in absolute value). Experiment 9, being the most studentized with a Cook distance of 0.539, constitutes the experiment with the greatest influence on the study of effects.

Table au 19: Table of residuals

N° Exp	Y exp.	Y calc.	Difference	Standardized	d U	Student-R	R-Student	D-Cook
1	82.300	80.512	1.788	0.795	0.688	1.422	1.649	0.405
2	60.900	62.487	-1.587	-0.706	0.688	-1.263	-1.369	0.319
3	75.300	76.437	-1.137	-0.506	0.688	-0.905	-0.885	0.164
4	60.900	59.962	0.937	0.417	0.688	0.746	0.708	0.111
5	69.000	68.487	0.513	0.228	0.688	0.408	0.371	0.033

6	59.100	59.812	-0.712	-0.317	0.688	0.567	-0.524	0.064
7	58.600	59.762	-1.162	-0.517	0.688	-0.925	-0.909	0.171
8	54.000	52.637	1.363	0.606	0.688	1.084	1,109	0.235
9	76.000	78.063	-2.062	-0.917	0.688	-1.641	-2.161	0.539
10	64.800	62.938	1.863	0.828	0.688	1.482	1.770	0.439
11	74.300	72.887	1.413	0.628	0.688	1.124	1.163	0.253
12	58.100	59.313	-1.212	-0.539	0.688	-0.965	-0.957	0.186
13	66.000	66.237	-0.237	-0.106	0.688	-0.189	-0.170	0.007
14	60.900	60.462	0.438	0.195	0.688	0.348	0.315	0.024
15	57.300	56.412	0.888	0.395	0.688	0.706	0.666	0.100
16	51.100	52.187	-1.087	-0.484	0.688	-0.865	-0.839	0.150

III-2-3. COMPARISON OF THE TWO COMPLETE FACTORIAL DESIGNS

The study of the second design showed that hydrogen preoxide had no effect on the elimination rate of remazol under the chosen conditions. Table 20 shows the changes in the various parameters with or without hydrogen preoxide during treatment.

Table 20: Changes in parameters with or without hydrogen preoxide during treatment

	R^2	Taux moyen a_0	Contribution de a_1	Contribution de a_2	Contribution de a_3	Effet de a_1	Effet de a_2	Effet de a_3
Sans H_2O_2	0,981	66,937	42,08	13,04	19,65	12,96	7,22	8,86
Avec H_2O_2	0,978	64,287	30,9	9,5	22,9	11,12	6,17	9,58
Moyenne	0,9795	65,612	36,49	11,27	21,275	12,04	6,695	9,22
Ecartype	0,002	1,87	7,91	2,50	2,30	1,30	0,74	0,51
Variance	0,22	2,86	21,66	22,21	10,80	10,80	11,08	5,52

This table presents the variance of some parameters studied in the two PFCs at the 5 % risk. There is a significant difference if the variance is greater than 15 % [12]. The analysis of this table shows that the use of hydrogen peroxide has no significant influence on the value of the linear correlation coefficient of the experiments and on the average rate of dye removal, even if a slight decrease is observed. Nevertheless, a significant difference can be observed in the two PFCs between the contributions of the concentration of remazol and the contributions of the concentration of Al^{3+}. Indeed, the variances observed are 21.66 % and 22.21 % respectively for the contribution of the concentration of remazol and that of the concentration of Al^{3+}. The contributions decrease considerably when hydrogen peroxide is added to the reaction medium. There is no significant difference at the 5 % risk for the effects of factors a1, a_2 and a_3 in the two PFCs

CONCLUSION

Identifying the factors involved in the elimination of remazol black in aqueous solution by coagulation-flocculation is important in the context of an experimental design. The Hadamard design made it possible to identify the most influential factors. The experimental domains chosen for the full factorial designs (with or without H_2O_2) resulted in average reduction rates of around 64 to 67 %. Indeed, in these domains, the factors have a negative influence on the response because they cause the reduction rate to decrease when moving from the low level to the high level in the chosen domain. Improving this rate would require careful adjustment of the experimental domain. Future work will allow us to establish an experimental domain taking into account what has been done in this experimental approach.

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