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### RESEARCH ARTICLE

#### OPTIMIZATION OF CONGO RED DYE ADSORPTION WITH ACTIVATED CARBON FROM THE COB OF CORN (ZEA MAYS L.) BY EXPERIMENTAL PLANS

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#### Abstract

Several methods and techniques are used for the treatment of polluted water. Among these techniques, adsorption on activated carbon. In this work, it is about the optimization of the elimination of the Congo Red dye by adsorption of the activated carbon prepared from corn cobs using two experimental designs. The carbon prepared has a specific surface of 673.27 m<sup>2</sup>/g. The effect of varying five factors (solution pH, carbon mass, temperature, stirring time and stirring speed) was studied. The first plan (screening plan or Hadamard plan) showed that there are three parameters (the pH of the solution, the mass of the carbon, the temperature) whose variations have a non-negligible influence on the response. The second design used, the two-level full factorial design, showed that the removal rate of Congo Red increases with the mass of charcoal. Indeed, its maximum value reached is 95.50% under optimal conditions where the pH of the colored solution is 2 at 60°C and with a mass of charcoal of 0.80 g.

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#### Introduction:-

Water is the most important element for human needs. It represents an essential and irreplaceable element to ensure the continuity of the life of living beings. The pollution of water and soil, by certain chemical products of industrial origin (hydrocarbons, phenols, dyes, etc.) or agricultural (pesticides, fertilizers, etc.) constitutes a source of environmental degradation and arouses particular interest today. at the International scale. Many studies have shown the responsibility of certain dyes in several diseases (Ahmed and Hameed, 2010; Girard, 1981; Krifa et al., 1990; Uddin et al., 2009; Vimonses et al., 2009a; Zohre et al., 2010). Some of these pollutants are dyes, more specifically Congo Red, which, once dissolved in water, is difficult to treat due to its synthetic origin and its complex molecular

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structure which makes it more stable and difficult to biodegrade. Consequently, it can constitute risk factors for human health and cause harm to the environment (Sadki et al., 2014). It is therefore necessary to considerably limit this pollutant by developing a suitable means of treatment such as a discoloration unit. There is currently a wide range of water pollution control technologies (physical, chemical and biological) including the adsorption of organic molecules such as dyes on activated carbon which has proven to be a highly effective treatment (Mameri, 2017). In fact, corn on the cob, agricultural waste, is recycled into activated carbon for depollution. This depollution that is the elimination of the Congo red dye proves to be even more effective because of its less expensive cost of realization and the obtaining of a better result with the method of the plans of experiments (N'Goran et al., 2009).

The main objective of this work is to optimize the elimination of Congo Red dye (diazo dye used in textiles) in water from activated carbon made from corn cob (*Zea mais*) using experimental plans.

## Material And Methods:-

### Procedure for making Congo Red synthetic solution

Five grams of Congo Red dye were dissolved in 1 liter of distilled water. The product has a purity of 99.98%. To obtain 20 mg/L, a distillation was carried out for each experiment. The preparation and activation of activated carbon were carried out thermally and chemically from raw materials consisting essentially of corn cobs. They were taken from the locality of Korhogo. In this locality, corn cobs are stored for various uses including heating. Corn cobs broken into small pieces were washed with tap water to remove dust, then washed with distilled water and dried in an oven at 105°C for 24 h before impregnation in a solution of orthophosphoric acid at 2 M. After 24 hours of impregnation, the corn cobs are calcined at 500° C. for 2 hours in an OBERSAL brand oven. The carbons obtained are washed thoroughly, after cooling with distilled water until the pH of the rinsing water is between 6 and 7. They are dried in an oven at 105°C for 24 hours; then crushed, sieved (size  $\leq 125 \mu\text{m}$ ), and finally stored in an airtight bottle. The activated carbon obtained in this study was characterized using different techniques in order to study its physico-chemical properties. Thus, properties such as mass loss, ash content, specific surface area, surface functions, Zero Charge Potential or pH at zero charge point (PZC) and iodine value were determined according to methods well known in the literature.

### Planification des expériences

Two experimental designs were used in this study. Thus, the Hadamard matrix was used as a first approach in order to separate the different factors acting on the phenomenon of elimination of the Congo Red dye in synthetic aqueous solution. According to Ano et al., 2019, this first plan should make it possible to know very quickly which of the various factors considered have the most influence on the response. The factors retained are: the pH of the solution (X\_1), the mass of carbon (X\_2), the temperature (X\_3), the stirring time (X\_4) and the stirring speed (X\_5). These five (5) factors lead to the experimental domain presented in Table 1 (First set of experiments). The levels of different factors were selected based on the preliminary assays. The full factorial design was used as a second approach to optimize dye removal. The study of the average effect, the main effects and the interactions made it possible to find the optimal conditions for this elimination. Three factors were retained in this plan taking into account the result of the Hadamard plan. They are: the mass of the carbon (X1), the temperature (X2) and the speed of agitation (X3). Table 1 (Second set of experiments) presents the established experimental domain.

The experimental values of  $U_i$  can be calculated from the coded variables  $X_i$  using the following equation (eq. 1):

$$X_i = \frac{U_i - U_{i,0}}{\Delta U_i} \text{ (eq.1)}$$

Where  $U_{i,0} = (U_{i,\text{max}} + U_{i,\text{min}})/2$  represents the value of  $U_i$  at the center of the experimental field and  $\Delta U_i = (U_{i,\text{max}} - U_{i,\text{min}})/2$  represents the step of the variation, with  $U_{i,\text{max}}$  and  $U_{i,\text{min}}$  which are the maximum and minimum values of the effective variable  $U_i$ , respectively.

The main interactions, correlation coefficients, variance analysis, residual, standard deviation were performed by using the NEMROD-W program software (design NEMROD-W, version 9901 French LPRAI-Marseille Inc, France) (Goupy J, 2006). All the experiments were duplicated in order to estimate the variability of measurements.

**Table 1:-** Experimental domain of the two planes used.

Coded variables (X <sub>i</sub> )	Factors (U <sub>i</sub> )	Experimental field		U <sub>i,0</sub>	ΔU <sub>i</sub>
		Min value (-1)	Max value (+1)		

First set of	Experiments				
X <sub>1</sub>	U <sub>1</sub> : pH of the solution	2.000	13.000	7.000	5.500
X <sub>2</sub>	U <sub>2</sub> : Mass of carbon (g)	0.050	0.300	0.175	0.125
X <sub>3</sub>	U <sub>3</sub> : Temperature (°C)	30.000	60.000	45.000	15.000
X <sub>4</sub>	U <sub>4</sub> : Agitation time (min)	20.000	100.000	60.000	40.000
X <sub>5</sub>	U <sub>5</sub> : stirring speed (rpm)	15.000	60.000	37.500	22.500
Second set of	Experiments				
X <sub>1</sub>	U <sub>1</sub> : pH	2.000	4.000	3.000	1.000
X <sub>2</sub>	U <sub>2</sub> : Mass of carbon (g)	0.500	0.800	0.650	0.150
X <sub>3</sub>	U <sub>3</sub> : Température (°C)	50.000	70.000	60.000	10.000

## Results And Discussion:-

### Characteristics of activated carbon prepares

The characteristics of the charcoal activated by H<sub>3</sub>PO<sub>4</sub> obtained in this study are given in Table 2. The value of the specific surface area obtained is equal to 673.27 m<sup>2</sup>/g (Table 2). This value is twice that found by Aboua in 2013 for the activated carbon based on *Tieghmelia heckelii* known under the name of Makoré. It has in fact obtained a specific surface area of 229.51 m<sup>2</sup>/g (Aboua, 2013). Concerning the surface functions, the presence of both acidic and basic groups on the surface of the activated carbon gives it a double effectiveness with respect to the chemical nature of the pollutant to be treated. These results are similar to those obtained by Atheba et al. (2015). These deniers obtained a total acidity of 3.99 meq/g and a total basicity of 1.075 meq/g from an activated carbon prepared on the basis of coconut shell of Ivorian origin (Afrane G., and Achaw O., 2008). The value of the surface functions 2.05 obtained (Table 2) shows that the activated charcoal we used has a marked acidic character. The value of the zero charge potential of the prepared activated carbon value is comparable to that obtained within the framework of the determination of the isoelectric point of an activated carbon prepared from peanut shell and activated with orthophosphoric acid by Kafack in 2012. The latter obtained a value of 2.18 Kafack F., (2012). The result of the iodine number obtained is 168.31 mg.g<sup>-1</sup> (Table 2). This value is low compared to that obtained by Afrane and Achaw in 2008. Indeed, the latter obtained values of iodine number ranging from 250 mg/g to 640 mg/g from activated carbons prepared with the shell of coconut (Afrane G., Achaw O., 2008).

**Table 2:-** Characteristics of prepared activated carbon.

Physical quantities			Values
Mass loss (%)			61.440
Ash content (%)			5.870
Specific surface m <sup>2</sup> /g			480.560
Zero charge potential (pH <sub>PZC</sub> )			2.050
Iodine number (mg/g)			168.210
		NaOH	2.050
	Neutralization	NaHCO <sub>3</sub>	0.650
Surface functions	(méq/g)	Na <sub>2</sub> CO <sub>3</sub>	1.270
		HCl	1.075
	Total Acidity		3.970
	Total Basicity		1.070

### Analysis of the results from the Hadamard matrix

The results of the experiments related to the Hadamard plan made it possible to have the elimination rates of Congo Red in synthetic aqueous solution summarized in Table 3. These results vary from 18.72 to 90.70%. (Table 3). The high Congo Red removal rate is obtained with Experiment 2.

**Table 3:-** Experimental field of coded and real variables of the Hadamard design factors.

No exp.	Factors										Red Congo
	Coded		Variables			Reals					Removal (%)
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	X <sub>5</sub>	pH of solution	Mass of	T (°C)	Ag. Time	Stir. Sp.	

							carbon		(min)	(rpm)	
1	+1	+1	+1	-1	+1	12.00	0.30	60.00	60.00	150.00	<b>58.80</b>
2	-1	+1	+1	+1	-1	2.00	0.30	60.00	15.00	300.00	<b>90.70</b>
3	-1	-1	+1	+1	+1	2.00	0.05	60.00	60.00	150.00	<b>60.50</b>
4	+1	-1	-1	+1	+1	12.00	0.05	30.00	60.00	300.00	<b>18.72</b>
5	-1	+1	-1	-1	+1	2.00	0.30	30.00	60.00	300.00	<b>56.80</b>
6	+1	-1	+1	-1	-1	12.00	0.30	60.00	15.00	300.00	<b>30.50</b>
7	+1	+1	-1	+1	-1	12.00	0.05	30.00	15.00	150.00	<b>42.68</b>
8	-1	-1	-1	-1	-1	2.00	0.05	30.00	15.00	150.00	<b>68.50</b>

Ag. time: Agitation time (min) Stir. Sp.: Stirring speed (rpm)

The estimates and statistics of the coefficients are presented in table 4. Thus, the average elimination rate is expressed by  $b_0$  and is equal to 52.40%. This indicates an average elimination rate of 52.40% in this chosen experimental area. Furthermore, the analysis of the different coefficients shows that the abatement rate decreases by 29.45% ( $b_1 \times 2$  or  $14.725 \times 2$ ) and by 7.39% ( $b_5 \times 2$  or  $3.695 \times 2$ ) respectively when the pH increases by 2 to 12 and when the stirring speed increases from 150.00 to 300.00 rpm. However, this elimination rate increases by 19.69% ( $b_2 \times 2$  or  $9.845 \times 2$ ), 15.45% ( $b_3 \times 2$  or  $7.725 \times 2$ ) and 1.50% ( $b_4 \times 2$  or  $0.75 \times 2$ ) respectively, when the absorbent mass increases from 0.05 to 0.3 g; when the temperature increases from 30 to 60°C and the stirring time increases from 15 to 60 minutes.

A coefficient is statistically significant if its absolute value is greater than twice the standard deviation (Zahangir A. et al., 2007). The experimental error (standard deviation) obtained is 4.075. Thus, the significant factors are the main effects  $b_1$  (the pH) and  $b_2$  (the mass of the coal). However, the temperature-related factor  $X_3$  has an average effect on the elimination of Congo Red; the factor  $X_5$  related to the stirring speed has a relatively moderate effect on this phenomenon. As for the factor  $X_4$  linked to the stirring time, it has a negligible effect. The importance of the factors is also highlighted on the elimination of Congo Red in synthetic aqueous solution, using eq. 2. Indeed, it is possible to provide more significant information by calculating the contribution of each factor to the response (elimination of Congo Red).

$$P_i = \left( \frac{b_i^2}{\sum b_i^2} \right) * 100 \quad (i \neq 0) \quad (\text{eq. 2})$$

Where  $b_i$  represents the different estimates the main effect of the factors.

Le diagramme de Pareto de la figure 1 montre les contributions du pH de la solution ( $X_1$ ), de la masse du charbon ( $X_2$ ), la température ( $X_3$ ), la vitesse d'agitation ( $X_5$ ) et le temps d'agitation ( $X_4$ ) sur l'élimination du Rouge Congo que sont respectivement, 55.93 %, 25.00 %, 15.39 %, 3.52 % et 0.15 % (figure 1). Le diagramme de Pareto confirme l'analyse faite précédemment sur les différents facteurs. Ces résultats ne sont valables que dans le domaine expérimental considéré.

Thus, in order to optimize the elimination of Congo Red, another experimental domain was defined the most influential factors on the phenomenon studied. In view of the previous analyses, the factors retained are;  $X_1$ ,  $X_2$  and  $X_3$  which are respectively the pH, the mass of carbon and the temperature which will be varied in a new experimental domain. However, the two other factors (stirring time and stirring speed) will be kept constant at their values which allow the best rate of elimination of Congo Red in synthetic aqueous solution. The stirring time will be maintained at 90 min and the speed will be maintained at 150 rpm.

The objective of using the Hadamard plan is to find the most important factors on the removal of Congo Red dye. Thus, the use of the complete factorial plan is not only to study the interactions between the factors but also and above all to optimize the elimination of Congo Red in synthetic aqueous solution.

**Table 4:-** Estimates and statistics of the coefficients of response Y (Hadamard plan).

Name	$b_0$	$b_1$	$b_2$	$b_3$	$b_4$	$b_5$
Coefficients	52.400	-14.725	9.845	7.725	0.750	-3.695
Standard deviation	4.075	4.075	4.075	4.075	4.075	4.075

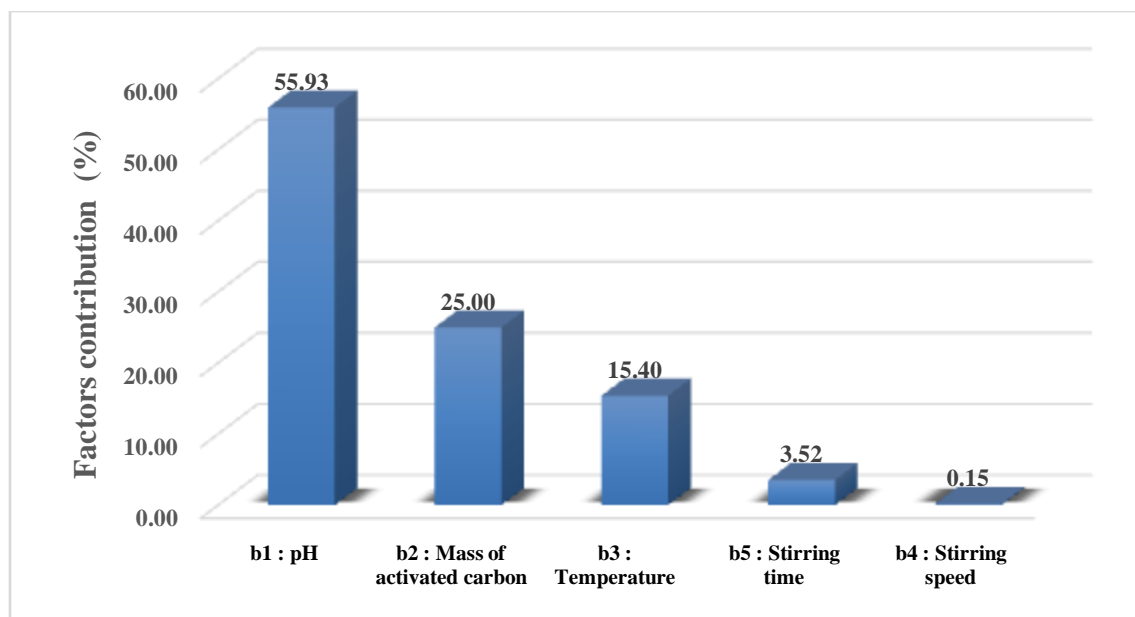


Figure 1:- Individual Pareto effect of Hadamard matrix factors.

#### Analysis of the results from the full factorial plan Estimates, statistics and contribution of coefficients

The elimination rates vary from 75.00 to 95.50% (Table 5). The experiment took place at a stirring time of 90 min at a speed of 150 rpm. The concentration being fixed at 20 mg/L. The results show a very satisfactory Congo Red elimination rate (Table 5). This rate is even better in experiment 3 at a pH of 2, a carbon mass of 0.8 g and a temperature of 60°C (Table 5). This result is justified by the value of the average coefficient which is 88.429% (Table.). This coefficient represents the average of the responses from the 8 tests, thus indicating an average elimination rate of Congo Red in a synthetic aqueous solution of the order of 88.42%. This confirms the choice of a better experimental domain for this second experimental design of this study.

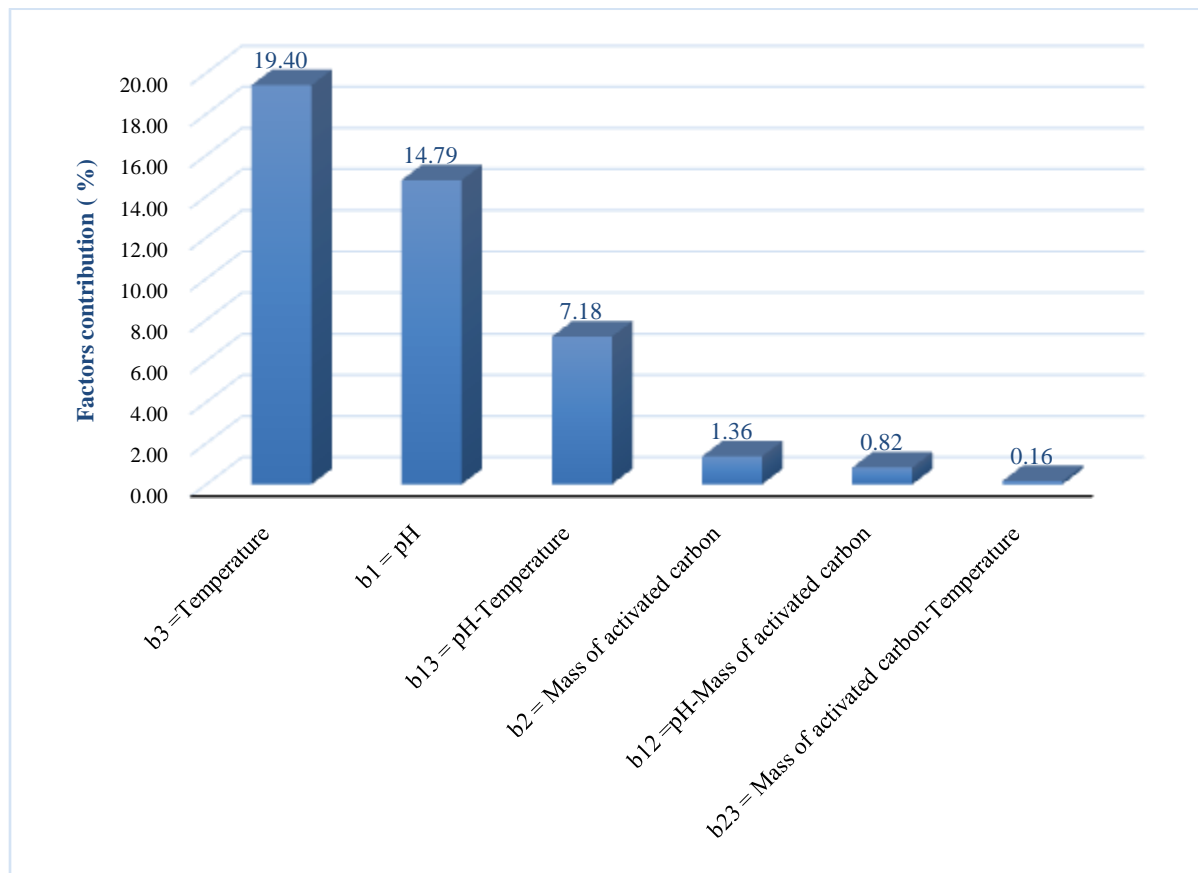
All the coefficients (Table 6) related to the different factors being significant (Zahangir A. et al., 2007), this indicates a significant impact of all the factors (pH (X1); mass of carbon (X2) and temperature (X3)) in this new area on the elimination of Congo Red. As for the effects of interactions, only the interaction between the mass of the carbon (X2) and the temperature (X3) is insignificant on this phenomenon. All of these findings were confirmed by analysis of the Pareto chart (Figure 2). Through this figure, the analysis of the Pareto diagram shows us the weight of each factor (figure 2), in descending order. Temperature (19.40%) and pH (14.79%) appear to be the most influential factors in this field of study. The main effect (b2) which is linked to the mass of the activated carbon (1.36%) has a relatively low weight even though the coefficient linked to this factor is high. As for the interaction effects, that between pH and temperature (b13) (7.80%) is the most important. In addition, the mass of activated carbon (b2 positive) has a positive effect on the response because its increase leads to an increase in the elimination rate of 3.358%. As for the pH of the solution (b1 is negative) and the temperature (b3 is negative), they have negative effects on the response. Indeed, their increases cause a significant decrease in the elimination rate of Red Congo of 7.692% and 8.808% respectively. Their interaction also leads to a high decrease in this rate. So for high pH and temperature, the elimination rate will be low. It is therefore necessary to work with low pH and temperatures in order to minimize their effects and the effect of their interaction.

**Table I:-** Experimental domain of coded and real variables of the factors of the Complete Factorial design.

No exp.	Factors			Real	Variables		Remazol Black Removal (%)
	Coded	Variables	Real		Variables	Temperature (°C)	
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	pH	Masse du charbon actif (g)	Temperature (°C)	
1	-1	-1	-1	2.00	0.50	60.00	92.50
2	+1	-1	-1	4.00	0.50	60.00	89.00
3	-1	+1	-1	2.00	0.80	60.00	95.50
4	+1	+1	-1	4.00	0.80	60.00	94.33
5	-1	-1	+1	2.00	0.50	75.00	90.50
6	+1	-1	+1	4.00	0.50	75.00	75.00
7	-1	+1	+1	2.00	0.80	75.00	90.60
8	+1	+1	+1	4.00	0.80	75.00	80.00

**Table 6:-** Estimates and statistics of coefficients (PFC).

Name	b <sub>0</sub>	b <sub>1</sub>	b <sub>2</sub>	b <sub>3</sub>	b <sub>12</sub>	b <sub>13</sub>	b <sub>23</sub>
Coefficients	88.429	-3.846	1.167	-4.404	0.904	-2.679	-0.404
Standart dev.	3.321	0.321	0.321	0.321	0.321	0.950	0.321

**Figure 2:-** Effet Pareto individuel des facteurs et des interactions du Plan Factoriel Complet.

### Study of interaction effects

Figure 3 shows the effect of the interaction between solution pH and carbon mass on GR removal. This shows removal rates of more than 80.00% of Congo Red in an acid medium. However, according to this figure, the best rate obtained is 93.05%, and this, at a pH of 2 and with a mass of activated carbon of 0.8 g. As for the interaction between the pH of the solution (X<sub>1</sub>) and the temperature (X<sub>3</sub>), the best rate is obtained at pH 2 and at a temperature of 60 (figure 4). This confirms the fact that in synthetic aqueous solution, Congo Red is abundantly eliminated in a

very acid medium. From the analysis of the effects of interactions, it appears that the best elimination rates obtained are in a very acid medium (pH = 2). These results are agree with the results of Khaoula Sehaïlia and Fatma Talbi in 2019.

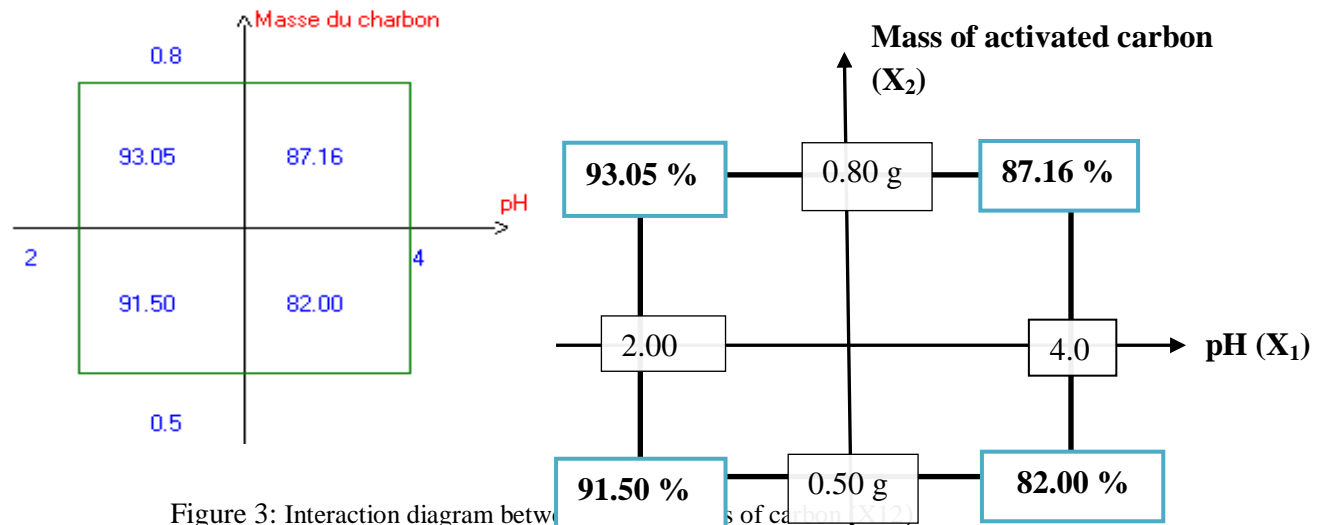


Figure 3: Interaction diagram between Mass of activated carbon (X<sub>2</sub>) and pH (X<sub>1</sub>)

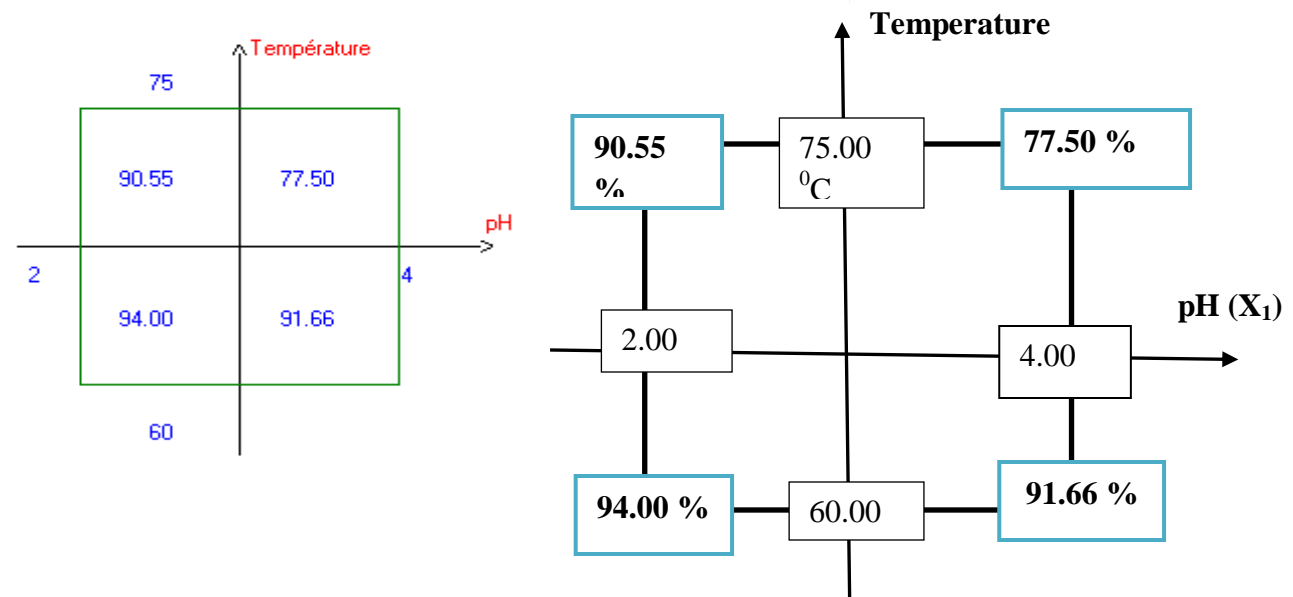


Figure 4:- Interaction diagram between pH and temperature (X13).

**Influences of factors on Congo Red elimination**

For the pH, the Hadamard plan allowed us to notice that the increase in pH has a negative influence on the elimination of RC. To have a good elimination, it is necessary to be in acid medium. But with the full factorial plan this elimination is better with a solution of pH = 2. This pH value is in agreement with that found by Khaoula Sehaïlia and Fatma Talbi in 2019 where they found a pH equal to 2.03. As for the mass of charcoal also has a significant influence on the elimination of Congo Red. This elimination increases with the mass of the coal. Indeed, work on the elimination of organic pollutants on activated carbons enabled their authors to reach similar results (Elbariji et al., 2006). This could be explained by the increase in the exchange surface of the adsorbent and the (increasing) availability of more adsorption sites. As for the temperature, the influence of the latter with the Hadamard plane on the response is positive when the temperature increases. But when we continue to increase this temperature in the PFC, this influence becomes negative. This would mean that we have exceeded the threshold

temperature which, in our experimental domain, is 60°C. Agitation time and agitation speed have no significant influence on the removal of Congo Red Dye.

**Analysis of the linear model obtained**

The statistical analysis of this model leads to the analysis of variance table (Table 7). This variance shows that the model used is well adjusted because the sum of squares due to the error (0.83) is very small compared to the total sum of squares (362.11). This good adjustment is confirmed by the finer analysis given by the value of the multiple linear correlation coefficient ( $R^2 = 0.998$  or 99.8%), given by the software. This coefficient indeed tends towards 1. We then have a very satisfactory adjustment because the value of this coefficient indicates that the model explains 99.8% of the phenomenon of elimination of Congo Red in synthetic solution. The analysis of the residuals (Table 7) also shows a quality adjustment of the model studied. Indeed, the correlation coefficient obtained previously by the software has the same value as that obtained by the plot (figure 5)  $Y_{exp}$  (measured responses) as a function of  $Y_{calc}$  (responses predicted by the model) presented in table 7 (given by The software). Moreover, the comparison between the  $Y_{exp}$  and  $Y_{calc}$  columns (Table 7) confirms the conclusion obtained above. Indeed, all residues do not exceed 5% (Feinberg 1996). All these findings validate the linear model of the phenomenon studied. The mathematical model that emerges from this study is in the following definitive form (eq. 3):

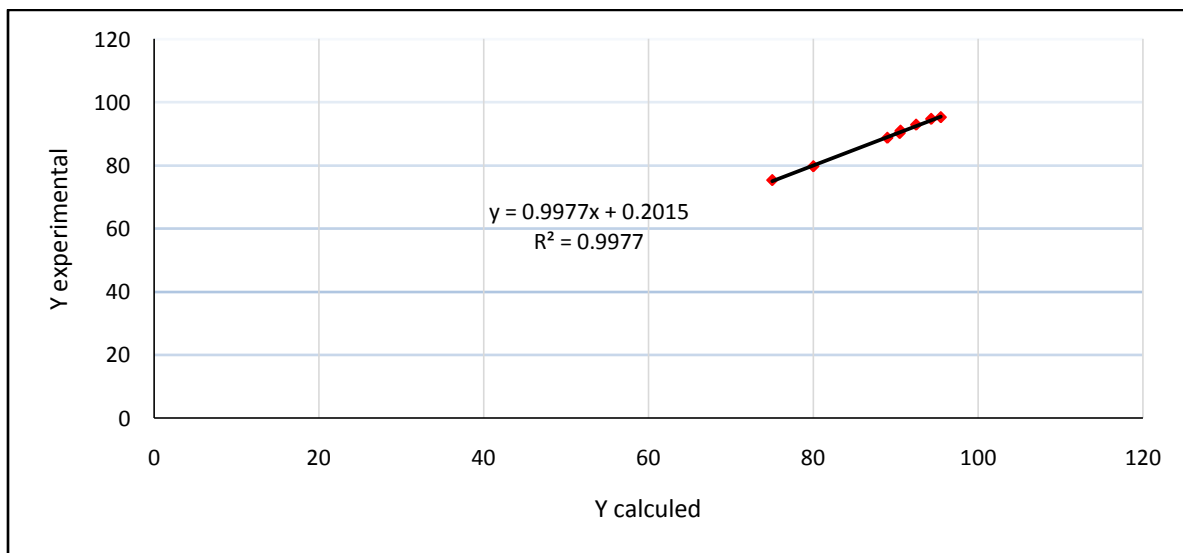
$$Y = 88.429 - 3.845X_1 + 1.679X_2 - 4.404X_3 + 0.904X_{12} - 2.0679X_{13}$$

**Table 7:-** PFC variance analysis.

source of variation	sum of squares
Regression	361.80
Residue	0.83
Total	362.11

**Table 8:-** Analysis of RC adsorption residues by PFC.

N°Exp	Yexp.	Ycalc	Différence
1	92.500	92.821	-0.321
2	89.000	88.679	0.321
3	95.500	95.179	0.321
4	94.330	94.651	-0.321
5	90.500	90.179	0.321
6	75.000	75.321	-0.321
7	90.600	90.921	-0.321
8	80.000	79.679	0.321



**Figure 5:-** Variation of the residuals of the experimental response as a function of those of the calculated response.



### Determination of the optimal conditions for the elimination of Congo Red dye

In this part, we will call optimal output response:  $Y = 100$ , which amounts to eliminating all the dye (100 %, ideal condition) in the effluent after its treatment with activated carbon. Thus, finding the conditions to satisfy this output amounts to solving the equation (eq. 3):

$$Y = 88.429 - 3.845X_1 + 1.679X_2 - 4.404X_3 + 0.904X_{12} - 2.0679X_{13} = 100$$

$$88.429 - 3.845X_1 + 1.679X_2 - 4.404X_3 + 0.904X_{12} - 2.0679X_{13} = 100$$

Using the Excel Solver utility, we get the following results:

$$Y = 100.000055 \text{ for } X_1 = -1, X_2 = 0.705 \text{ and } X_3 = +1$$

The optimal retention conditions for Congo Red in synthetic solution in this field of experimentation are: the pH ( $X_1$ ) is 2,000; the mass of the activated carbon ( $X_2$ ) is 0.564 g; the temperature ( $X_3$ ) is 60.000 °C; the stirring time is 90.000 minutes and a stirring speed of about 150 rpm. Thus, under these optimal conditions, the maximum value predicted by the model for the theoretical elimination rate of Congo Red in synthetic aqueous solution is 100.00 %.

### Verification of optimal conditions for the elimination of Congo Red

The theoretical result previously predicted by the model was confirmed experimentally by carrying out three experiments under the predicted conditions (Table 9). Indeed, the average of the 3 tests of the rate of elimination of Congo Red in synthetic solution obtained is  $(95.26 \pm 0.04)$  %. The error is less than 5 %. Based on this fact, the comparison with the predicted value gives residuals between 0.02 and 0.06, thus indicating an almost perfect agreement between the experimental values and the predicted values. The results of this study are in agreement with those of Toor, (2012), Sami Guiza et al. (2013) and Bekakra et al. (2017).

**Table 9:-** Removal rate under optimal condition.

No. of experiments	No. of experiments
1	95.30
2	95.24
3	95.26

### Conclusion:-

The main objective of this study was to optimize the elimination of Congo Red dye in synthetic aqueous solution from activated carbon based on corn cob using experimental designs. Activated carbon, made from the cob of corn prepared and activated by the chemical process with ortho-phosphoric acid as an activating agent has proven effective in the elimination of Congo Red. This activated carbon has a strong acid character with a non-negligible basicity. To make a success of this study, the treatment of the data by two plans of experiments used (the plan of Hadamard and the factorial plan) showed that in the experimental field the rate of elimination increases with the mass of carbon while the pH has a contrary effect. As for the temperature, it has a limit value beyond which its effect becomes negative. Optimization of the process has shown that for a mass of activated carbon equal to 0.8 g, pH 2, and a temperature of 60°C, a Congo Red elimination rate of 95.26% is achieved.

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