World Journal of Advanced Research and Reviews
eISSN: 2581-9615 CODEN (USA): WJARAI
Cross Ref DOI: 10.30574/wjarr
Journal homepage: https://wjarr.com/

(RESEARCH ARTICLE)

REMOVAL OF COPPER PHTHALOCYANINE (CUPC) BY ACTIVATED CARBON FROM *BALANITES AEGYPTIACA* TRUNK IN AQUEOUS SOLUTION: OPTIMIZATION OF ADSORPTION CAPACITY BY THE TWO-LEVEL FULL FACTORIAL DESIGN

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World Journal of Advanced Research and Reviews, 2026, 31(03), 703–712

Publication history: Received on 31 July 2026; revised on 07 September 2026; accepted on 09 September 2026

Article DOI: https://doi.org/10.30574/wjarr.2026.31.3.2178

ABSTRACT

Activated carbon produced from lignocellulosic biomass is an effective alternative for various applications, particularly in the environmental field. Water, essential to life, has always been contaminated by the discharge of effluents containing toxic chemicals. These substances not only impact the growth of aquatic plants but also, and above all, human life. Among water pollution control techniques, adsorption by activated carbon appears to be the easiest method to implement. Therefore, the aim of our study is the removal of copper phthalocyanine using activated carbon produced from the trunk of *Balanites aegyptiaca* and the optimization of its adsorption capacity using a two-level full factorial design. The optimization allowed us to achieve a maximum removal rate of 90.34%.

Keywords: Adsorption, Copper Phthalocyanine, Activated Carbon, Full Factorial Design

1 INTRODUCTION

Industrial activities and population growth have led to an exponential increase in global water demand. While essential for life on Earth, water has become increasingly polluted due to the discharge of effluents containing toxic compounds. These compounds not only alter the color of the water but also hinder light penetration, severely impacting aquatic ecosystems. However, various pollution control techniques, such as sedimentation **[1, 2]**, filtration **[3, 4]**, reverse osmosis **[5]**, oxidation **[6]**, photocatalytic degradation **[7]**, ion exchange **[8]**, etc., have been developed to minimize this

impact. But these conventional treatment methods have limited effectiveness when dealing with effluents containing dyes, as these dyes are highly soluble in water and often non-biodegradable. Thus, the adsorption of pollutants by activated carbon remains the most widely used, efficient, and cost-effective method for removing aromatic pollutants due to the large specific surface area, surface chemistry, and pore size distribution of these materials, which allow them to trap a very broad spectrum of molecules of varying sizes **[9]**. Numerous studies have focused on the use of adsorbents based on lignocellulosic biomass. Our approach aims to use the trunk of *Balanites aegyptiaca* as a precursor for activated carbon to remove copper phthalocyanine (CuPc) from aqueous solution by optimizing the adsorption capacity using a two-level, full-factor design.

2 MATERIAL AND METHODS

After characterizing our prepared activated carbons, the activated carbon produced from BA impregnated at 25% with orthophosphoric acid, pyrolyzed at 450°C with an impregnation time of 16 hours (CAE-BA-25%-450°C-16h) was chosen for the removal of copper phthalocyanine.

2.1 Preparation of copper phthalocyanine (CuPc) solution

A stock solution was prepared by dissolving 0.1 g of copper phthalocyanine (CuPc) in a 100 mL volumetric flask containing concentrated sulfuric acid (H_2SO_4). Standard solutions were prepared from this stock solution by dilution. The calibration curve of absorbances as a function of concentrations (10, 20, 30, 40, and 50 mg/L) was plotted.

2.2 Study of the adsorption of copper phthalocyanine (CuPc)

In a 200 mL beaker are introduced 50 mg/L copper phthalocyanine solution and 100 mg of activated carbon were used. This mixture was magnetically stirred at room temperature. At regular intervals, 10 mL samples were taken and then filtered using filter paper. A ***Uvi****Line 9400C* UV-Visible spectrophotometer was used. was used to measure the residual concentration of CuPc. The reading was taken at 670 nm. The amounts adsorbed at equilibrium (q_e) and the elimination rate (TE) were calculated according to equations 1 and 2:

$$q_e = \frac{(C_i - C_r)}{m} \times V \qquad (\mathbf{1})$$

$$\mathrm{TE} = \frac{(C_i - C_r)}{C_i} \times 100 \qquad (\mathbf{2})$$

With C_i and C_r, the initial and residual concentrations of Copper phthalocyanine (mg/L); V, the volume (L) of the solution used for the adsorption tests and m, the mass (g) of activated carbon.

2.3 Modeling the adsorption kinetics of CuPc

Pseudo-first-order, pseudo-second-order and intra-particle diffusion models were studied to model adsorption kinetics.

2.4 Pseudo-first-order kinetic model

$$\ln(q_e - q_t) = lnq_e - k_1 t \qquad (\mathbf{3})$$

With:

q_e: Quantity of solute adsorbed at equilibrium (mg/g);
q_t Quantity of solute adsorbed at time t (mg/g);

k_1: Rate constant (min^{-1}) of the pseudo-first-order equation determined by plotting$\ln(q_e - q_t)$ as a function of time (t) **[10]**.

2.5 Pseudo second-order kinetic model

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \qquad (\mathbf{4})$$

Integrating this equation for t from 0 to t and q_t from 0 to t q_t leads to:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (\mathbf{5})$$

2.6 Intraparticle diffusion model

$$q_t = k_d * t^{1/2} + C \qquad (6)$$

Or :

k_d Intra-particle diffusion rate constant (mg/g.min$^{1/2}$),

3 RESEARCH INTO THE OPTIMAL CONDITIONS FOR CUPC ELIMINATION AND STUDY OF THE FACTORS USING A TWO-LEVEL FULL FACTORIAL DESIGN

The effects of factors on the effectiveness of CuPc elimination were investigated using a two-level full factorial design. This design allows for the analysis of interaction effects between factors with fewer trials. For this purpose, three factors were considered, namely the The concentration of CuPc, the mass of activated carbon, and the pH were used to determine the optimal conditions. The number of experiments to be performed was defined by the full factorial design. Thus, eight (8) experiments were carried out according to the experimental model.

The model is presented as follows:

$$Y = b_0 + \sum_i b_i X_i + \sum_{ij} b_{ij} X_i X_j \qquad (7)$$

With, b_0, the value of the mean coefficient; b_i, the effect of factors X_i and b_{ij} the effect of the interactions of factors i and j.

The independent and coded factors and the factor matrix are presented in Tables I and II.

Table 1 Experimental domain of variable factors

Factors	Level -1	Level +1
pH (X_1)	7	12
Concentration (mg/L) (X_2)	25	50
Mass of activated charcoal (mg) (X_3)	25	50

Table 2 Factorial matrix

Trials	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

Table III gives the experimental plan by replacing the coded values (-1 and +1) with the actual values (pH, concentration and mass).

Table 3 Experimental plan

Trials	pH	Concentration	Mass
1	12	50	50
2	7	50	50
3	12	25	50
4	7	25	50
5	12	50	25
6	7	50	25
7	12	25	25
8	7	25	25

The results were processed using Nemrod software version 2000. Significant coefficients were determined by comparing the calculated coefficients to twice the standard deviation (2σ) **[11,12].**

4 RESULTS AND DISCUSSION

4.1 Adsorption kinetics

The kinetic results shown in Figure 1 indicate that the adsorption kinetics of CuPc on the prepared activated carbon are rapid during the first five minutes. This can be explained by the availability of active sites on the adsorbent surface. Subsequently, the kinetics slow down between five and sixty-five minutes, due to the saturation of active sites on the adsorbent surface. During this period, the amount adsorbed is only 4.565 mg/g. Adsorption equilibrium is reached after 15 minutes.

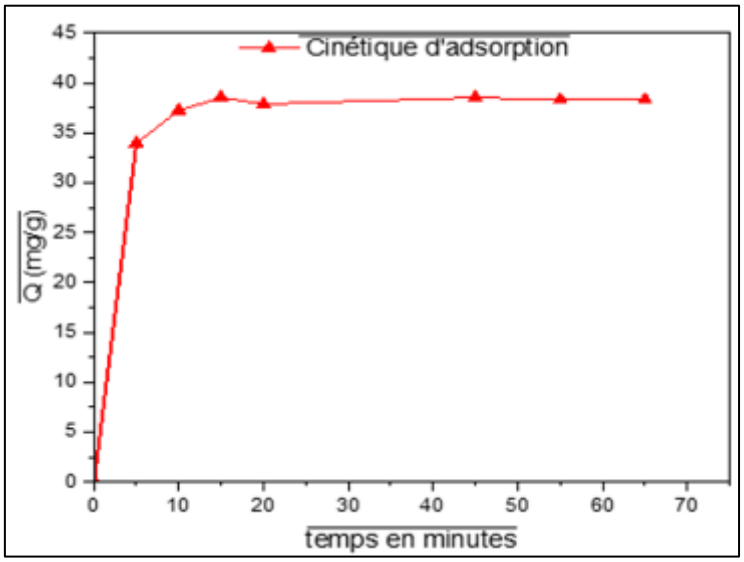

Figure 1 Results of the adsorption kinetics of CuPc on activated carbon

4.2 Modeling of adsorption kinetics

The modeling of the kinetics (figure 2) by three models mentioned above, shows that the correlation coefficient relating to the pseudo second order kinetic model is close to 1 ($R^2 = 0.9999$), which confirms the validity of this model.

Indeed, we can understand that the adsorption kinetics of CuPc on activated carbon are second-order. Thus, the second-order model is generally used to describe chemisorption. According to several authors, the adsorption kinetics of dyes on activated carbon are consistent with the hypothesis of the second-order model, according to which the molecules interact with the different surface functional groups of activated carbon **[13]**.

However, we observe that the correlation coefficients obtained (with first order and intraparticle diffusion) are not significant, which indicates that the adsorption of copper phthalocyanine is not favorable with these models.

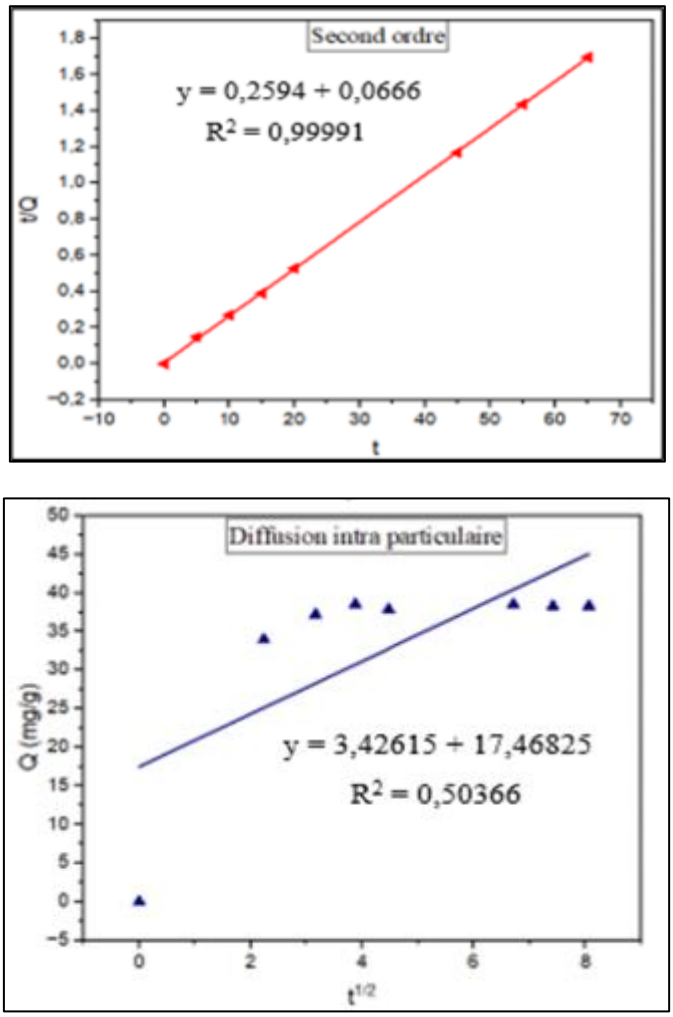

Figure 2 The modeling of adsorption kinetics of CuPc on activated carbon

4.3 CuPc adsorption study by experimental design

The result from the full 2-level factorial design of two responses Y1 and Y2 is shown in Table IV.

Table 4 Experimental design and responses

Exp. No.	X_1 (pH)	X_2 (CuPc concentration) (mg/L)	X_3 (mass of activated charcoal) (mg)	Y_1 (CuPc adsorption capacity) (mg/g)	Y_2 (Rate elimination) (%)
1	12	50	50	44.840	89.69
2	7	50	50	45.500	90.34
3	12	25	50	9.840	39.39
4	7	25	50	4.410	17.65
5	12	50	25	56.650	56.65
6	7	50	25	73.600	73.60
7	12	25	25	18.820	37.65
8	7	25	25	18.820	37.65

This result shows that the highest adsorption capacity (73.600 mg/g) is obtained in trial 6, while the lowest adsorption capacity (4.410 mg/g) is obtained in trial 4. The values obtained in trials 7 and 8 are identical regardless of the response. Furthermore, the best removal rate (90.34%) is obtained in trial 2. The equations for the complete model for both responses are as follows:

$$Y_1 = 34.060 - 1.522X_1 + 21.087X_2 - 7.912X_3 - 2.880X_1X_2 + 2.715X_1X_3 - 2.065X_2X_3$$

$$Y_2 = 55.410 + 0.435X_1 + 22.325X_2 + 4.022X_3 - 5.000X_1X_2 + 4.672X_1X_3 + 8.587X_2X_3$$

The estimates and statistics of the different coefficients from the experimental plan are presented in Table V.

Table 5 Estimates and statistics of the different coefficients

	Coefficients		**Standard deviation**		**2* Standard deviation**	
Name	Y1 Y2		Y1 Y2		Y1 Y2	
b0	34.060	55.410	1.357	0.763	2.714	1.526
b1	-1.522	0.435	1.357	0.763	2.714	1.526
b2	21.087	22.325	1.357	0.763	2.714	1.526
b3	-7.912	4.022	1.357	0.763	2.714	1.526
b12	-2.880	-5.000	1.357	0.763	2.714	1.526
b13	2.715	4.672	1.357	0.763	2.714	1.526
b23	-2.065	8.587	1.357	0.763	2.714	1.526

For Y_1 (Figure 3), the factors that have a significant influence are

X_2 (concentration en CuPc) et X_3 (masse du charbon actif)..

Furthermore, the interaction effects are also significant for Y_1. The factor X_1 (pH) is not statistically significant regardless of the response. The model equation for adsorption capacity as a function of different factors deviates:

$$Y_1 = 34.060 + 21.087X_2 - 7.912X_3 - 2.880X_1X_2 + 2.715X_1X_3$$

For response Y2 (Figure 4), the factors X_2 , X_3 are those that significantly influence the elimination rate. Thus, all interaction effects (X_{12} , $X_{13} et\ X_{23}$) have a significant influence on the elimination rate.

$$Y_2 = 55.410 + 22.325X_2 + 4.022X_3 - 5.000X_1X_2 + 4.672X_1X_3 + 8.587X_2X_3$$

5 INFLUENCE OF DIFFERENT FACTORS AND GRAPHICAL STUDY OF THE EFFECTS OF TWO RESPONSES

5.1 Influence of the pH of the CuPc solution

b_1 coefficients of two responses are less than twice the standard deviation. This shows that pH does not have a significant influence on Y_1 and Y_2.

5.2 Influence of CuPc concentration

When the concentration of CuPc increases from 25 to 50 mg/g, the adsorption capacity and the elimination rate increase by 42.174 mg/g and 44.65% respectively (2 x b_2).

5.3 Influence of activated carbon mass

When the mass of activated charcoal increases from 25 to 50 mg, the adsorption capacity decreases by 15.824 mg/g while the elimination rate increases by 8.044% (2 x b_3).

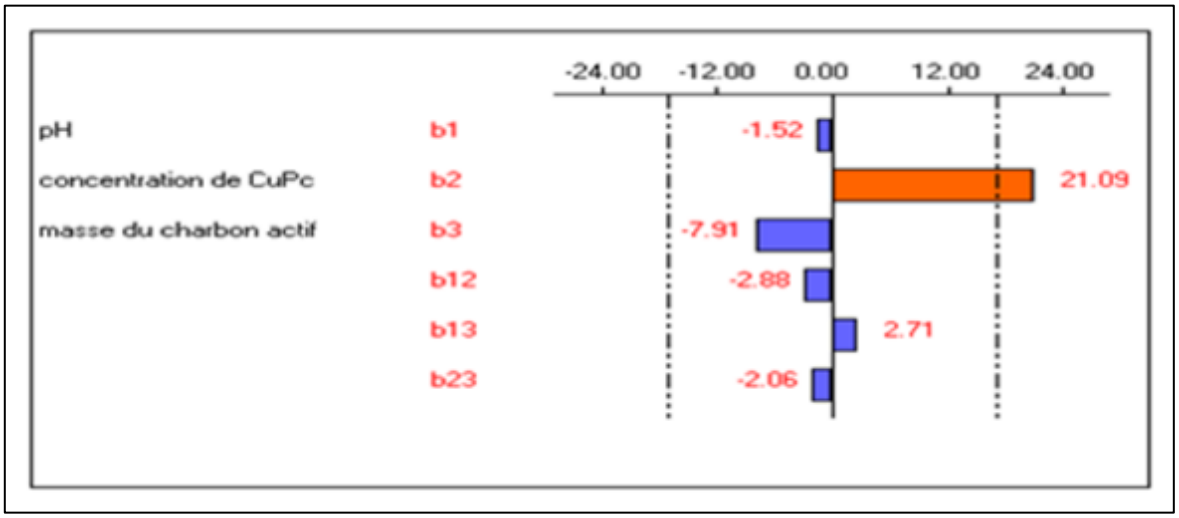

Figure 3 Graphical study of the effects of the Y1 response (adsorption capacity)

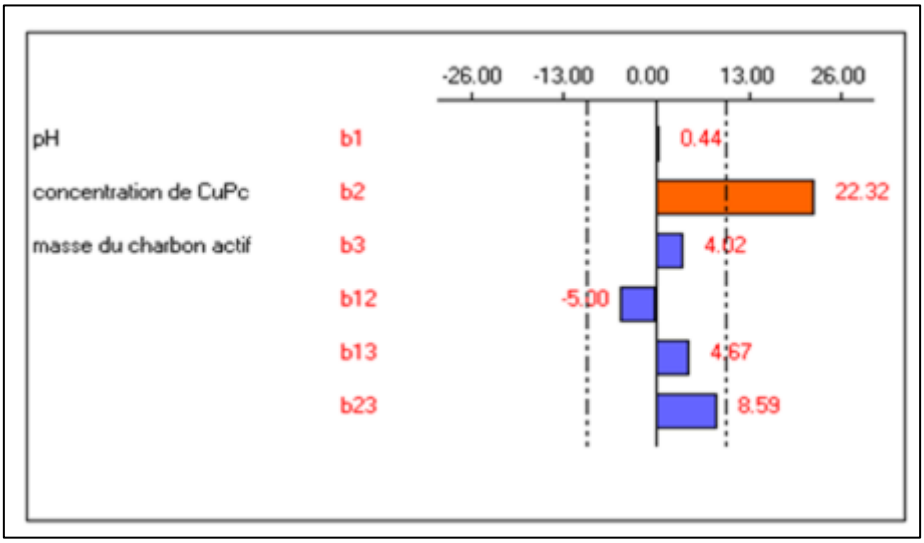

Figure 4 Graphical study of the effects of the Y2 response (adsorption rate)

Indeed, the quality of the developed model is evaluated based on the determination of the correlation coefficient and the adjusted coefficient presented in Table VI:

Table 6 Correlation coefficient and adjusted coefficient of two responses

Correlation coefficient	Y_1 (capacity adsorption)	Y_2 (Rate adsorption)
R^2	0.997	0.999
R^2 Adj	0.976	0.994

The correlation between the experimental and predicted values is very good, which confirms the validity of this model.

5.4 Interactions between different factors for the Y_1 response (adsorption capacity)

Only the interaction between CuPc mass and concentration (Figure 5) is significantly more important among the interactions studied. Analysis of the diagram shows that the optimal values (65.12 and 45.17 mg/g) of the CuPc adsorption capacity on CAEs are obtained when the CuPc concentration and CA mass increase from 25 to 50 mg/L and 25 to 50 mg, respectively. Similar results were reported by B. Lemya and B. Ghaniyya, 2016 **[14].** on the removal of methylene blue by optimizing adsorption using a two-level full factorial design.

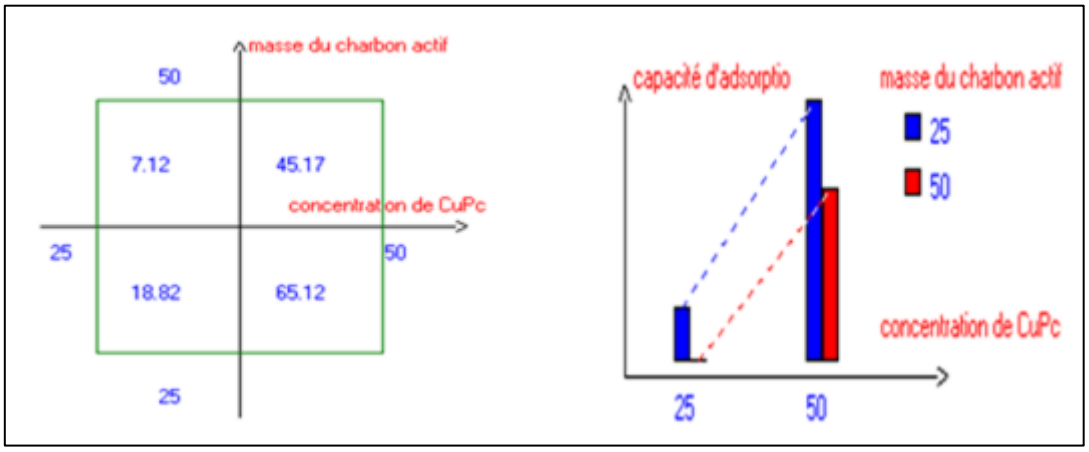

Figure 5 Interaction diagram between mass and concentration for Y1

5.5 Interactions between different factors for the Y_2 response (elimination rate)

5.5.1 Interaction between pH and CuPc concentration

The interaction between pH and CuPc concentration Figure 6 highlights the importance of concentration in the adsorption process. Increasing the concentration leads to a significant increase in the adsorption rate, regardless of the pH. The maximum adsorption rate (82.30%) is obtained at a concentration of 50 mg/L and pH 7.

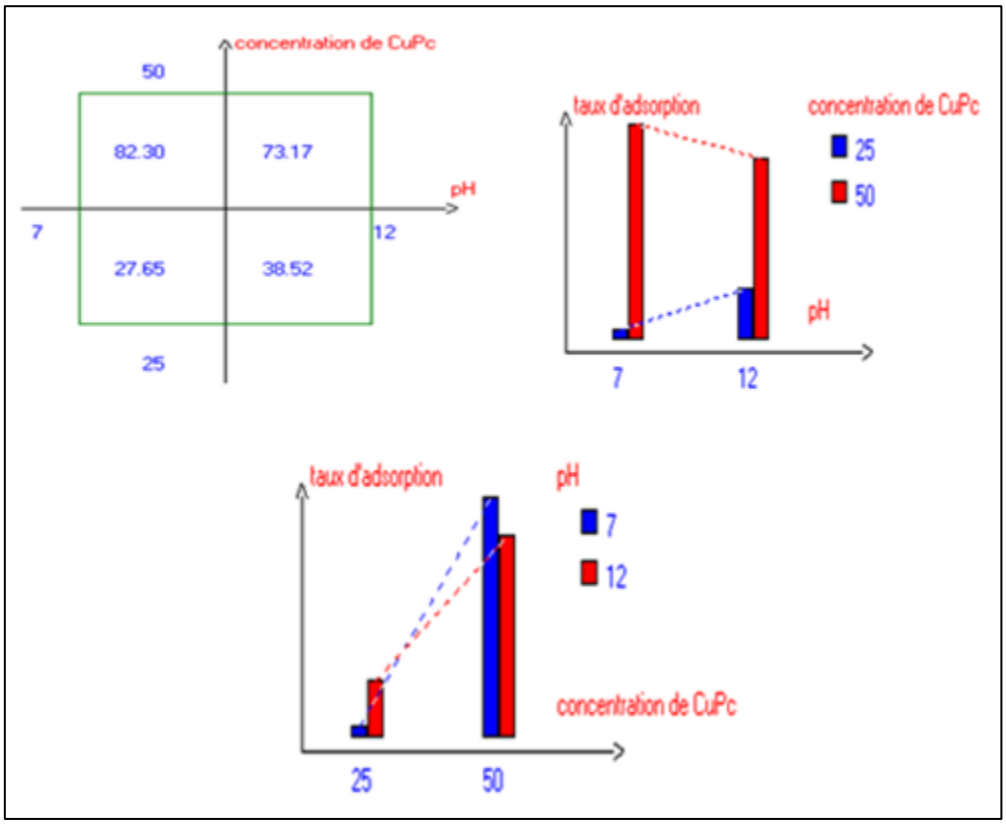

Figure 6 Concentration-pH interaction diagram for Y2 response

The result of the interaction between mass and pH shows an increase in the adsorption rate when the mass of activated carbon increases from 25 to 50 mg. The maximum rate (64.54%) is obtained at pH 12 with a mass of 50 mg. However, at pH 7, the adsorption rate is significant (55.62%) when the mass of activated carbon is low (25 mg).

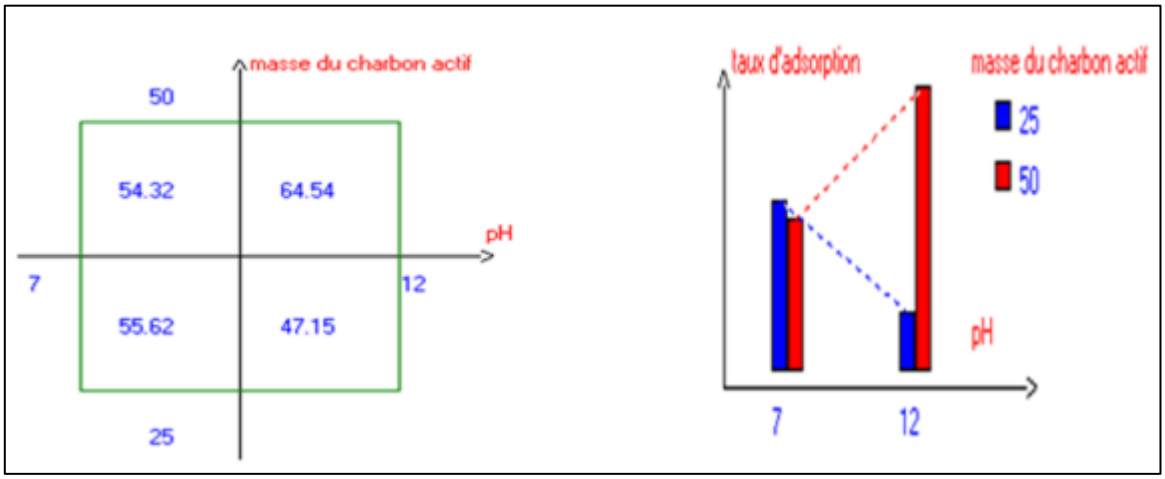

Figure 7 Mass and pH interaction diagram for Y2 response

5.5.2 Interaction between CuPc concentration and CA mass

Finally, the interaction between the CuPc concentration and the mass of activated carbon reveals a range where the CuPc removal efficiency is optimal (90.34%). This efficiency is obtained with 50 g of activated carbon and 50 mg/L of CuPc at pH 7.

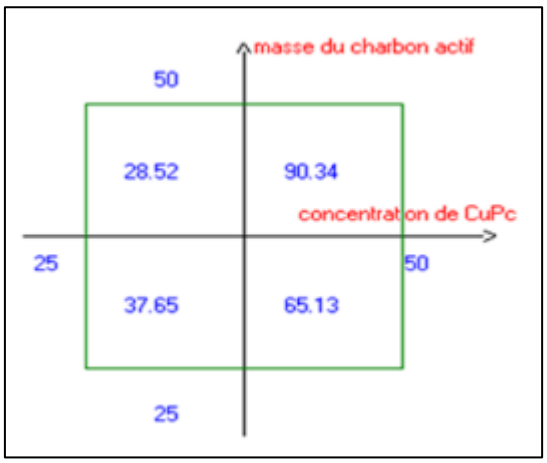

Figure 8 Interaction diagram between mass and concentration for the Y2 response

6 CONCLUSION

This study highlights the role of each selected factor in the adsorption process of copper phthalocyanine (CuPc) in aqueous media. Of the three factors considered, only pH was not statistically significant for all responses, as the pH coefficient values for two responses were less than twice the standard deviation. The analysis of interactions between factors demonstrates the important role of pH in the interactions between different factors. Optimal conditions allowed for a maximum removal rate of 90.34%, achieved with 50 mg of activated carbon and a CuPc concentration of 50 mg/L.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Acknowledgments

We would like to thank Abdou Moumouni University in Niamey for providing us with the setting in which the work was carried out.

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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