International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2013): 6.14 | Impact Factor (2013): 4.438

Optimization of Adsorption Performance of an Industrial Dye on Waste Mint by the Design of Experiments Method - Composite Centered Plan

Fatima KHAMMOUR, Laila LAASRI, Abdelkebir KENZ, Tarik AINANE, M'hamed ELKOUALI, Samia YOUSFI, Mohammed TALBI

Laboratory of Analytical Chemistry and Physical Chemistry of Materials,
Faculty of Sciences Ben Msik, Hassan II University, BP 7955 Casablanca - 20700, Morocco

khammourfatima@gmail.com

Abstract: ***This work aims to optimize the adsorption performance of an industrial dye on waste mint realizing an optimization approach based on composite experimental design centered. Two influencing factors of the adsorption process were selected: The initial concentration of the adsorbate and the adsorbent dose. Then these factors were optimized using statistical and mathematical tools of a second degree polynomial model: regression analysis, canonical analysis, graphical representation of curves isoresponse and response surfaces, to lead to the selection of optimal conditions: pH = 7.1, agitation time = 120 min, low initial concentration of the adsorbate and high adsorbent dose.***

Keywords: Optimization, composite centered plan, methodology surfaces response, waste mint, methylene blue.

1. Introduction

The experimental design used to optimize the organization of experimental tests to obtain maximum information with the minimum experience and the best possible accuracy on the responses calculated with the model. This is achieved if we follow the rules established mathematically and if we adopt a rigorous approach **[1-2]**.

There are many experimental designs suitable for all cases encountered by an experimenter. Among all these plans, some are more frequently used than others **[3-5].**

Design of experiments applied to the study of response surfaces solves optimization problems. This method makes from a polynomial model, to determine what values the input factors should be adjusted to obtain the desired response **[6]**.

As the adsorption performance is considered among the criteria to explain the adsorption process, the objective of this study is continuing the work carried out by previous Ainane et al. **[7-8],** by performance optimization in an experimental area of methylene blue adsorption mint waste, hence the process is influenced by two factors (initial concentration of the adsorbate and adsorbent dose) using a methodology response surfaces **[9-12]**. Then the study has been completed the steps to construct the composite central plan, then we presented the analysis necessary to validate the second degree polynomial model with Nemrodw software.

2. Materials

2.1 Adsorbent

Mint wast devoted in this work comes from the Meknes region (MOROCCO) in the form of more embossed foliage and flavor more pronounced green color. Waste mint is dried in an oven for two days at a temperature of 60 ° C, finally dry mint is crushed and sieved and particles having an average of 0.1 mm are used for this work.

2.2 Adsorbate

The methylene blue solution was prepared from a specified quantity of 100 mg/L. The mother solution is dissolved in a corresponding volume of distilled water to obtain the desired concentration.

3. Methods

A known mass of the adsorbent is introduced into a solution containing a concentration of methylene blue at pH 7.1.

The mixture is then stirred at room temperature for any manipulation. After a stirring time of 2 hours the samples are left to stand for one hour.

Finally the residual concentration of methylene blue was determined using UV spectroscopy Visible to the wavelength λ_{max} = 664 nm. The performance defined by **[13]**:

$$R(\%) = \frac{C_0 - C_r}{C_0} \times 100 \quad (1)$$

Where:

C_0: initial concentration (mg/L).
C_r: residual concentration (mg/L).

4. Results and discussion

Construction of Experimental Design

The number of experiences of a centered composite design is determined using the following equation $N = k^2 + 2k + 1$, in our study the number of factors is equal 2:

- The initial concentration of the adsorbate (20 mg/L and 40 mg/L)
- The adsorbent dose (between 0.2 g/L and 1 g/L)

Thus, the polynomial model is written as follows:

$$Y = a_0 + a_1.C + a_2.M + a_3.CM + a_4.C^2 + a_5.M^2 \quad (2)$$

a_0: constant Model.
a_1: effect of initial concentration of adsorbate.
a_2: effect of the adsorbent dose.
a_3: interaction effect between the concentration of adsorbate and adsorbent dose.
a_4: effect of the quadratic component of the concentration of adsorbate.
a_5: effect of the quadratic component of the adsorbent dose.
The following figure shows the experimental points of a vector plane on two factors 2^2 (A, B, C and D), point E is the center point and points F, G, H and I are the axial points: they are located on two axes at a distance from the center equal to α = 1.414.

In total the plan requires nine tests with six coefficients to estimate.

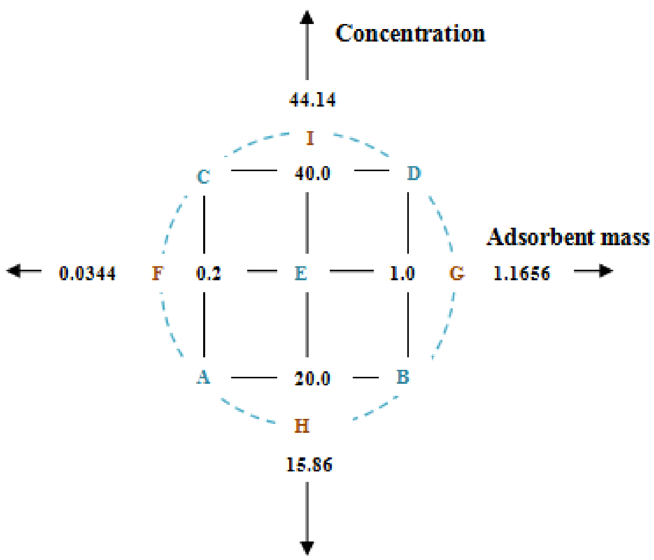

Figure 1: Geometric representation of the experimental design

The test results are summarized in the following table:

Table 1: Design of Experiments and results

Test	Coded variable		Real variable		Performance (%)
	C (mg/L)	M (g/L)	C (mg/L)	M (g/L)	
1	-1	-1	20	0.2	86
2	1	-1	40	0.2	71
3	-1	1	20	1	91
4	1	1	40	1	83
5	1.414	0	44.14	0.6	77
6	-1.414	0	15.86	0.6	95
7	0	1.414	30	1.1656	87
8	0	-1.414	30	0.0344	75
9	0	0	30	0.6	86

4.1 Statistical Analysis of Results

4.1.1 Factor Model

The calculation of estimated values of the six model coefficients is done using the method of least squares.

Table 2 shows the values of the coefficients for the estimated response adsorption efficiency, and the significance of each coefficient.

Table 2: Coefficients analysis table

	Coefficients	Significance
a_0	86.000	**< 0.01*** **
a_1	-6.057	**0.0138*** **
a_2	4.246	**0.0392*** **
a_3	1.750	**1.98***
a_4	-0.188	71.0
a_5	-2.688	**0.877** **

According to the table 2, we notice that the coefficient a_4 (effect of the quadratic component of the concentration of adsorbate) does not affect the adsorption performance since the value of the significance of the coefficient is greater than 5% while coefficients a_1, a_2, a_3 and a_5 have an influence on the adsorption efficiency.

The evaluation of the overall quality of the adjusted mathematical model allows knowing if the model correctly summarizes the test results of the experimental design and that using four statistical tools: Regression analysis, Coefficients of the multilinear regression R^2, R^2 adjusted and Residues.

4.1.2 Regression analysis

The Table-3 shows the regression analysis results.

Table 3: Regression analysis table

Source of variation	Sum of squares	Degree of freedom	Mean square	F_c	Probability F_c
Regression	482.343 2	5	96.468 6	154.022 9	0.0791***
Residues	1.8790	3	0.6263		
Tot	484.222 2	8			

From the results of this table we can see that the F_c probability value is less than 5%, we can say that the model correctly describes the variation of the test results.

4.1.3 Determination of coefficients of the multilinear regression R^2 and R^2 adjusted

We present in Table 4 the different values of R^2 determinations coefficients and adjusted R^2 adjusted

Table 4: R^2 and adjusted R^2 coefficients

R^2	0.996
$R^2_{adjusted}$	0.990

Based on these values, we see that both coefficients are nearer to 1; in this case we can say that the descriptive quality of the model is satisfactory.

4.1.4 Residues

The difference between the measured responses Y_{Exp} and the predicted Y_{calc} responses is very small which allows us to say that the model has good descriptive quality.

Table 5: Residues chart.

N° exp	Yexp	Ycalc	Différence	Normed	dU
1	86	86.686	-0.686	-0.866	0.625
2	71	71.072	-0.072	-0.090	0.625
3	91	91.678	-0.678	-0.857	0.625
4	83	83.064	-0.064	-0.081	0.625
5	95	94.191	0.809	1.022	0.625
6	77	77.059	-0.059	-0.075	0.625
7	75	74.620	0.380	0.480	0.625
8	87	86.630	0.370	0.467	0.625
9	86	86.000	-0.000	-0.000	1.000

4.2 Mathematical Analysis of the Results

In order to determine the optimum conditions for a better adsorption of methylene blue on the residue of mint, three mathematical tools were used which include: Canonical analysis, 3D graphing and 2D graphing.

4.2.1 Canonical analysis

The equation of the canonical form of the mathematical module can locate the optimum and determine its nature:

$$Y1 = 88.908 - 4.500\, Z_1 + 0.000\, Z_2 + 0.088\, Z_1^2 - 2.963\, Z_2^2 \quad (3)$$

The Z_1^2 and Z_2^2 are coefficients: 0.088 (positive) and -2.963 (negative), in this case we can say that the stationary point is a minimax.

4.2.2 3D Graphing

The 3D graphical analysis allows studying the effects of initial concentration of the adsorbate and adsorbent dose on the adsorption performance.

In analyzing figure 2, we see that the adsorption performance increases from 62.2% to 94.5% when the adsorbent dose increases and the initial concentration of the adsorbate decreases.

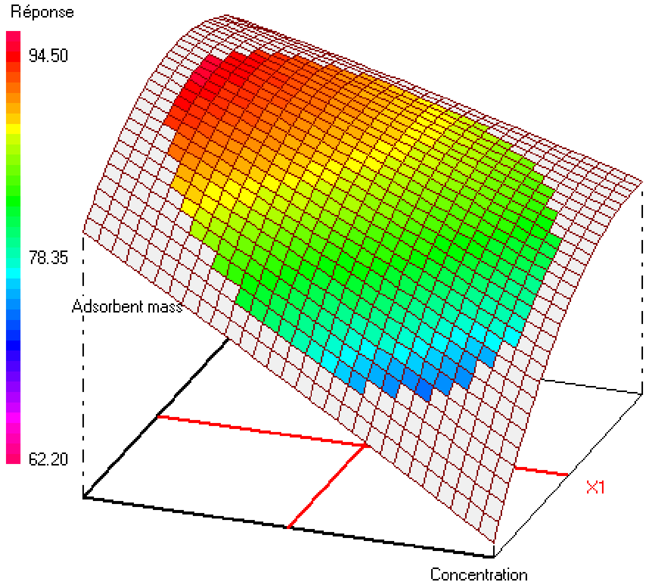

Figure 2: Response surface

4.2.3 2D Graphical Representation

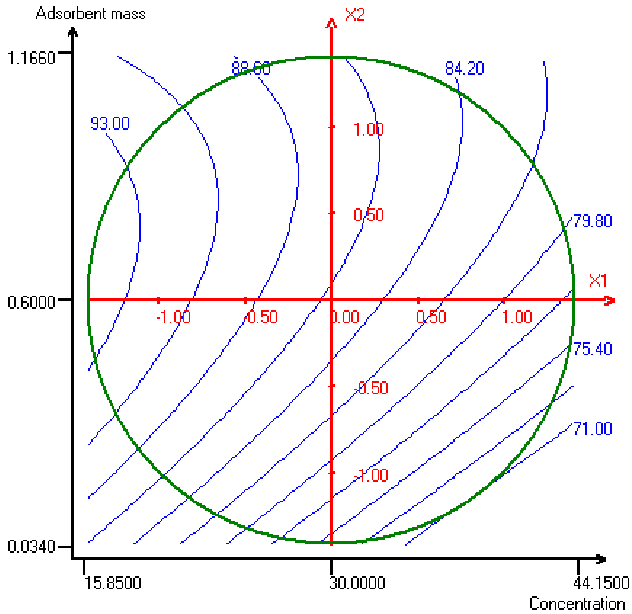

Figure 3: Isoresponse curves

The figure 3 represents the curves of the response isoresponse according to input factors initial concentration of the adsorbate and the adsorbent mass.

We see from this figure that the response is maximum for a maximum value of the adsorbent dose and a minimum value of the initial concentration of the adsorbate.

5. Conclusion

In this study, the composite experience centered plan has determined the effects of two parameters such as initial concentration of the adsorbate and the adsorbent dose in the adsorption process of methylene blue on waste of mint.

The results showed that the polynomial regression model of the second order is able to correctly interpret the experimental data by statistical analysis of a regression equation of the second order polynomial.

On the other hand, mathematical analysis has shown that the adsorption efficiency increases as the adsorbent dose increases and the initial concentration of the adsorbate decreases.

References

[1] D.C. Montgomery, "Design and analysis of experiments", John Wiley & Sons, 2008.
[2] R.H. Myers, D.C. Montgomery, C.M. Anderson-Cook, "Response surface methodology: process and product optimization using designed experiments", Vol. 705, John Wiley & Sons, 2009.
[3] J. Goupy, "Plans d'expériences", Ed. Techniques Ingénieur, 2006.
[4] T.J. Santner, B.J. Williams, W.I. Notz, "The design and analysis of computer experiments", Springer Science & Business Media, 2013.
[5] N. Juristo, A.M. Moreno, "Basics of software engineering experimentation", Springer Science & Business Media, 2013.
[6] E.F. Camacho, C. Bordons, "Model predictive control in the process industry", Springer Science & Business Media, 2012.
[7] T. Ainane, F. Khammour, M. Talbi, M. Elkouali, "A novel bio-adsorbent of mint waste for dyes remediation in aqueous environments: study and modeling of isotherms for removal of methylene Blue", Oriental Journal of Chemistry, 30(3), pp. 1183-1189, 2014.
[8] T. Ainane, F. Khammour, O. Belghazi, M. Kabbaj, S. Yousfi, M. Talbi, M. Elkouali, "Study and modelling of kinetics biosorption of methylene blue on biomass material from waste mint", BioTechnology: An Indian Journal,11(8), pp. 281-285, 2015.
[9] K. Ravikumar, K. Pakshirajan, T. Swaminathan, K. Balu, "Optimization of batch process parameters using response surface methodology for dye removal by a novel adsorbent", Chemical Engineering Journal, 105(3), pp. 131-138, 2005.
[10] J.N. Sahu, J. Acharya, B.C. Meikap, "Response surface modeling and optimization of chromium (VI) removal from aqueous solution using Tamarind wood activated carbon in batch process", Journal of hazardous materials, 172(2), pp. 818-825, 2009.
[11] K. P. Singh, S. Gupta, A.K. Singh, S. Sinha, "Optimizing adsorption of crystal violet dye from water by magnetic nanocomposite using response surface modeling approach", Journal of Hazardous Materials, 186(2), pp.1462-1473, 2011.
[12] A. Asfaram, M. Ghaedi, S. Agarwal, I. Tyagi, V.K. Gupta, "Removal of basic dye Auramine-O by ZnS: Cu nanoparticles loaded on activated carbon: optimization of parameters using response surface methodology with central composite design", RSC Advances, 5(24), pp. 18438-18450, 2015.
[13] T. Ainane, A. Abourriche, M. Kabbaj, M. Elkouali, A. Bennamara, M. Charrouf, M. Talbi, "Removal of hexavalent chromium from aqueous solution by raw and chemically modified seaweed Bifurcaria bifurcata", Journal of Materials and Environmental Science, 5(4), pp.975-982, 2014