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Optimization of conditions for the preparation of new adsorbent material from residues of marine sponges using experimental design method

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ABSTRACT

An experimental design has been drawn up to optimize the experimental conditions of the preparation of an adsorbent material from residues of marine sponges. A series of adsorbent materials have been prepared by chemical activation with sulfuric acid. Central composite design (CCD) was applied to study the influence of activation temperature, activation time and percentage of sulfuric acid on the chemical activation process of adsorbent material. Two quadratic models were developed for yield of adsorbent material and methylene blue adsorption capacity using Design-Expert software NEMRODW. The models were used to calculate the optimum operating conditions for preparation of adsorbent material providing a compromise between yield and adsorption capacity of the process. The yield (58.14 wt.%) and adsorption capacity (77.41%) of the adsorbent material produced at these operating conditions showed an excellent agreement with the values predicted by the models. The optimal conditions with 77.41% of methylene blue adsorption capacity and 58.14% of adsorbent material yield are obtained when using 150.3 min as activation time 237°C as activation temperature and 48% as percentage of sulfuric acid.

KEYWORDS

Marine sponges;
Adsorbent material;
Chemical activation;
Optimization;
Experimental design.

INTRODUCTION

Among marine invertebrates, the sponges (Phylum Porfera) continue to be a rich source of novel secondary metabolites, with a diversity of biological activities that continue to inspire the efforts of synthetic organic chemists[1-3]. They are even considered to be the more prolific producers of new marine natural products[4]. Until now, more than 5000 different compounds have been isolated from about 500 species of sponges[5].

However the residues of extraction obtained from these marine sponges, which represent more than 80% of the weight of the raw material, are not exploited enough. This requires a valorization of theses residues by the elaboration of new adsorbent materials.

The preparation of adsorbent materials is influenced by many factors. For this reason a preliminary study on the effect of these factors on the preparation was carried out in order to determine the most important ones and their regions of interest. The most influential factors were found to be activation time (X_1), activation temperature (X_2) and the percentage of chemical activating agent (X_3). Thus, in the present work, we prepared a series of adsorbent materials from residues of marine sponges with values of (X_1), (X_2) and (X_3) included in the suitable range. Desirable preparation outputs based on mass yield (Y_1) and capacity of adsorption (Y_2) were considered as responses. Thus, an experimental design methodology is applied to relate the experimental conditions of the activated process with properties of adsorbent materials[6,7].

MATERIALS AND METHOD

Materials

The sponge used in this study was collected from atlantic litoral of Morroco. All the reagents used were of analytical grade. Distilled water was used to prepare all solutions.

Preparation of adsorbent material

Prior to extraction, the marine sponges were identified, lyophilized and ground using an electric grinder to a particle size between 1 and 2 mm. The residues of extraction obtained from these marine sponges are impregnated with the same mass of sulfuric acid and mixed for half an hour in the ambient temperature. The obtained pastas are carried in the temperature of 120°C for 24 hours. The obtained materials underwent the reaction of activation in a leading electric thermolyne oven to self-regulation provided with a programmer of temperature, connected with the oven by a thermocouple and a special steel reactor. The domains of variation of temperature, time activation and percentage of chemical activated agent were defined on the univariate analysis. After activation, the adsorbent materials are washed with distilled water until all acid was eliminated, dried, ground and sifted to obtain a powder with a particle size capable of passing through a 100µm sieve.

Univariate analysis

Univariate analysis is the first step of analysis of process variables (temperature, time and chemical activating agent). The analysis is carried out with the description of a single variable and its attributes of the applicable unit of analysis. This step was used in the first stages of research, in analyzing the data at hand, before being supplemented by multivariate analysis using experimental design.

Temperature of activation

The weight loss behavior of the residues of marine sponges was measured by using a thermobalance (TGA- SETARAM). The sample was heated up to final temperature of 600°C at a heating of 10°C/min under helium gas.

Time of activation

The analysis of activation time effect was defined using adsorption test of cationic dye, the methylene blue (MB), on the adsorbent materials elaborated at an activation temperature of 225°C for activation time than 15min, 45min, 75min, 105min, 135 min, 165 min, 195 min, 225 min, 300 min and 360min and using concentrated sulfuric acid. This latter is well known as precursors activating agent, allows the development of a large porosity in the activated material[8-10].

The change of the adsorption performance based on the activation time is determined at room temperature onto methylene blue solution of known initial concentration (10 mg / L). The adsorbent materials in particle size of less than 100ìm are dispersed in 100 ml of MB solution at the rate of 200mg of adsorbent per liter of MB solution. The suspensions were continuously agitated throughout the manipulation (overnight) to ensure a better contact between the pollutant in contact and the active sites of the product. The measurement of the concentration of methylene blue was determined using UV-visible spectrometer (Perkin Elmer) at a wavelength $\lambda = 664$.

Percentage of chemical agent activating

To minimize the production cost of adsorbent ma-

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terials via the activation by the sulfuric acid. We studied the impact of this factor on the activation of residues of the sponges by testing the methylene blue adsorption capacity and the mass yield of adsorbent materials prepared at 225 ° C, 150min and at different concentrations of sulfuric acid. The evolution of the adsorption capacity and mass yield versus percentage of sulfuric acid was determined at room temperature on a solution of methylene blue (10 mg / L). The adsorbent materials in particle size of less than 100ìm are dispersed in 100 ml of BM solution at the rate of 100 mg of adsorbent per liter of MB solution. The suspensions were agitated continuously throughout the manipulation (5 hours) to ensure a better contact between the pollutant in contact and the active sites of the product. The measurement of the concentration of methylene blue was carried out using UV-visible spectrometer at a wavelength $\lambda = 664$.

The adsorbent material yield was calculated based on Eq. (1).

$$\text{Yield}(\%) = \frac{W_C}{W_0} \times 100 \quad (1)$$

where W_C is the dry weight (g) of final activated material and W_0 is the dry weight (g) of precursor.

The capacity of adsorption (%) was calculated based on Eq. (2).

$$\text{Capacity of adsorption (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where C_0 and C_e are the initial and final dye concentrations (mg/L) respectively.

Multivariate experimental design

Response surface methodology (RSM) is a statistical method that uses quantitative data from appropriate experiments to determine regression model equations and operating conditions[11]. RSM is a collection of mathematical and statistical techniques for modeling and analysis of problems in which a response of interest is influenced by several variables[12]. A standard RSM design called central composite design (CCD) was applied in this work to study the variables for preparation of adsorbent material from residues of marine sponges.

The central composite design was widely used for fitting a second-order model. By using this method, modeling is possible and it requires only a minimum number of experiments. It is not necessary in the modeling procedure to know the detailed reaction mechanism since the mathematical model is empirical. Generally, the CCD consists of a 2^n factorial runs with 2n axial runs and n_c center runs. These designs consist of a 2^n factorial or fractional (coded to the usual ±1 notation) augmented by 2n axial points (±α,0,0,..., 0), (0, ±α,0,...,0),..., (0, 0, ...,±α), and n_c center points (0, 0, 0, ...,0)[13-14]. The center points were used to evaluate the experimental error and the reproducibility of the data. The axial points are chosen such that they allow rotatability[15], which ensures that the variance of the model prediction is constant at all points equidistant from the design center. Replicates of the test at the center are very important as they provide an independent estimate of the experimental error. Each variable is investigated at two levels. Meanwhile, as the number of factors, n, increases, the number of runs for a complete replicate of the design increases rapidly. In this case, main effects and interactions may be estimated by fractional factorial designs running only a minimum number of experiments. Individual second-order effects can not be estimated separately by 2^n factorial designs. The responses and the corresponding parameters are modeled and optimized using ANOVA to estimate the statistical parameters by means of response surface methods.

Basically this optimization process involves three major steps, which are, performing the statistically designed experiments, estimating the coefficients in a mathematical model and predicting the response and checking the adequacy of the model.

$$Y = f(X_1, X_2, X_3, X_4..., X_n) \quad (3)$$

where Y is the response of the system, and X_i is the variables of action called factors. The goal is to optimize the response variable (Y). It is assumed that the independent variables are continuous and controllable by experiments with negligible errors. It is required to find a suitable approximation for the true functional relationship between independent variables and the response surface[16].

The experimental sequence was randomized in order to minimize the effects of the uncontrolled factors. The response was used to develop an empirical model that correlated the responses to the adsorption of methylene blue and production of adsorbent material pro-

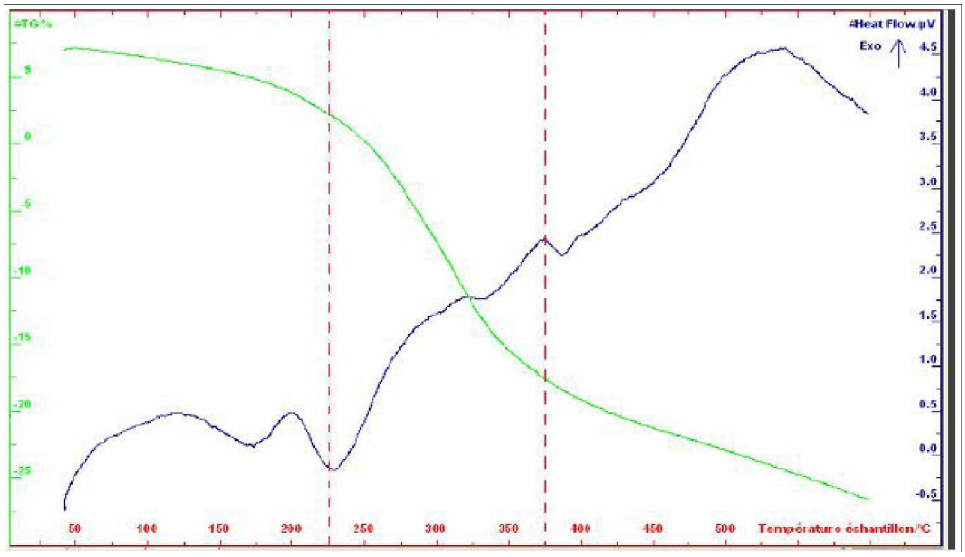

Figure 1 : Thermogram of marine sponges residues

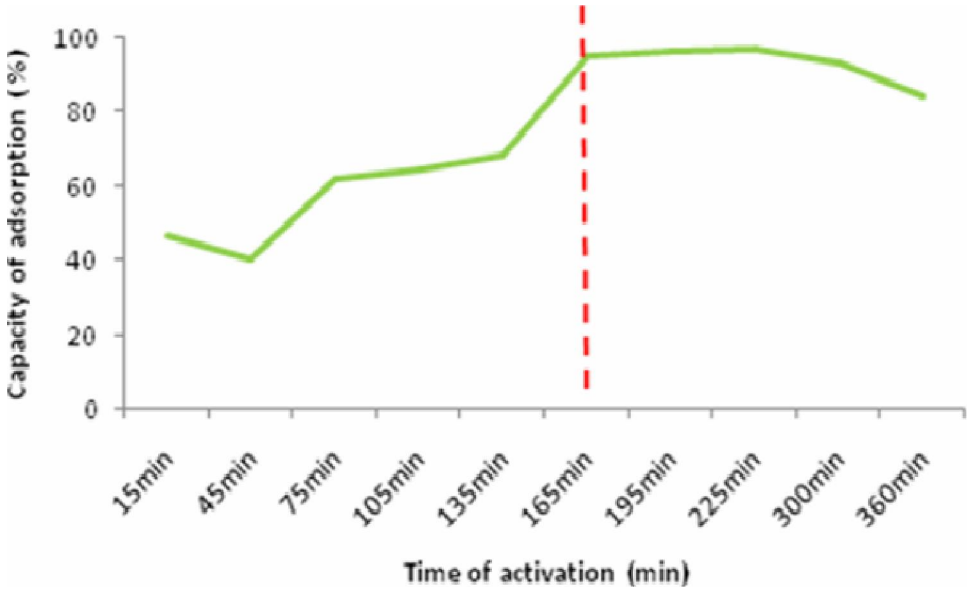

Figure 2 : The effect of the activation time on the adsorption capacity of methylene blue on the elaborated materials

cess variables using a second-degree polynomial equation as given by the following equation:

$$Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} b_{ii} X_i^2 + \sum_{i=1}^{n} \sum_{j>1}^{n} b_{ij} X_i X_j \quad \textbf{(4)}$$

where Y is the predicted response, b_0 the constant coefficient, b_i the linear coefficients, b_{ij} the interaction coefficients, b_{ii} the quadratic coefficients and X_i, X_j are the coded values of the production and adsorption of methylene blue variables. The number of tests required for the CCD includes the standard 2^n factorial with its origin at the center, 2n points fixed axially at a distance, say α from the center to generate the quadratic terms, and replicate tests at the center; where n is the number of variables. Hence, the total number of tests (N) required for the three independent variables is:

$$N = 2^n + 2n + n_c = 2^3 + (2 \times 3) + 2 = 16 \quad \textbf{(5)}$$

Once the desired ranges of values of the variables are defined, they are coded to lie at ±1 for the factorial points, 0 for the center points and ±á for the axial points.

Model fitting and statistical analysis

The statistical software package Design-Expert, NEMRODW, was used for regression analysis of experimental data to fit the equations developed and also to plot response surface. ANOVA was used to esti-

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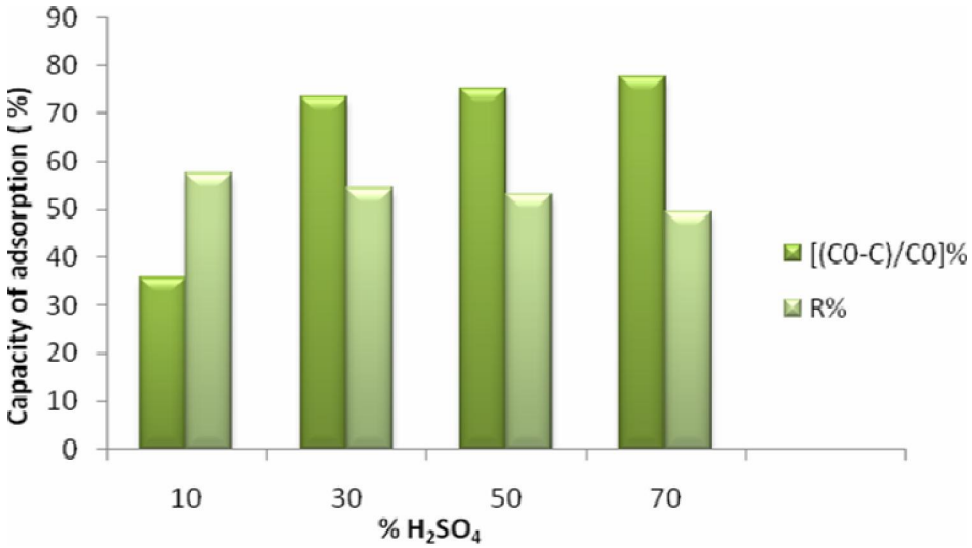

Figure 3 : The effect of the percentage of H_2SO_4 on the yield and adsorption of methylene blue on the materials elaborated

TABLE 1: Experimental domains of the factors intervener in the elaboration of adsorbent materials

Factors	Lower Level (-1)	Central points(0)	Higher level (+1)
Time of activation (X_1)	15 min	82,5 min	150 min
Temperature of activation (X_2)	225°C	300°C	375°C
% activating Agent (X3)	10%	40%	70%

mate the statistical parameters.

RESULTS AND DISCUSSION

Univariate analysis

The curve shown in Figure 1 represents the evolution of the normalized mass of marine sponges residues during pyrolysis in accordance with the temperature. The thermogram of marine sponges residues shows two distinct losses:

The first took place between 25 and 225 °C, and corresponds to a slight loss, mainly due to the departure of water. The second loss from 225 to 600 °C, which corresponds to a larger and complex loss. This loss begins at 225 °C and reaches its maximum at about 375 °C before ending at 600 °C. It corresponds to a loss in weight, due to the departure of molecules derived from the decomposition of organic matter.

Time of activation

From the Figure 2 we find that the maximum adsorption capacity is reached from 150 min. These results allowed us define the experimental field of study activation time which is between 15 and 150min.

Percentage of activating chemical agent

From the Figure 3 we find that the adsorbent material prepared at 225 ° C and activated with sulfuric acid at 70% is the one that gives the best adsorption capacity. These results allowed us to define the experimental field of study in the percentage of sulfuric acid which is between 10 and 70%.

Synthesis of univariate analysis

The univariate study allowed to determine the experimental domains for each of the three selected factors. The results are shown in TABLE 1.

Development of regression model equations

Central composite design was used to develop correlation between the adsorbent material preparation variables to the methylene blue adsorption capacity and adsorbent material yield. Runs15 and 16 at the center point were used to determine the experimental error. The design of this experiment is given in TABLE 2, together with the experimental results. Regression analysis was performed to fit the response function of methylene blue adsorption capacity (%) and adsorbent material yield (%). The model expressed by Eq.(4), where the variables take their coded values, represents adsorbent material yield (Y_1) and methylene blue adsorption capacity (Y_2) as a function of activation times (X_1), activation temperature (X_2), and percentage of chemical activating agent (X_3). The final empirical models in

TABLE 2 : Experimental design matrix and results.

Run	Coded level			Actual level of variables			Adsorbent material yield, Y_1 (%)	Capacity of adsorption, Y_2 (%)
	X_1	X_2	X_3	X_1 (min)	X_2 (°C)	X_3 (%)		
1	-1	-1	-1	15	225	10	42.20	46.83
2	+1	-1	-1	150	225	10	57.30	59.17
3	-1	+1	-1	15	375	10	54.70	24.75
4	+1	+1	-1	150	375	10	45.15	10.28
5	-1	-1	+1	15	225	70	40.80	57.10
6	+1	-1	+1	150	225	70	50.76	78.17
7	-1	+1	+1	15	375	70	55.05	43.41
8	+1	+1	+1	150	375	70	44.35	33.95
9	-α	0	0	15	300	40	62.83	69.91
10	+α	0	0	150	300	40	62.23	54.77
11	0	-α	0	82.5	225	40	52.17	80.01
12	0	+α	0	82.5	375	40	52.05	32.85
13	0	0	-α	82.5	300	10	63.85	45.54
14	0	0	+α	82.5	300	70	57.80	62.03
15	0	0	0	82.5	300	40	63.35	54.26
16	0	0	0	82.5	300	40	62.90	54.43

TABLE 3: Analysis of variance (ANOVA) for response surface quadratic model for adsorbent material yield (Y_1).

Source of variation	Sum Of squares	Degree of freedom	Mean square	F_{exp}	Signif
Regression	926.3612	9	102.9290	53.3368	0.0176***
Residual	11.5788	6	1.9298		
Lack of fit	11.4775	5	2.2955	22.6716	16.5
Pure error	0.1013	1	0.1013		
Correlation total	937 9400	15			

***** Significant at a 99.9 % level of confidence**

terms of coded factors after excluding the insignificant terms for adsorbent material yield (Y_1) and methylene blue adsorption capacity (Y_2) are as follows: Eqs 6 and 7

$$Y_1 = 63.242 + 0.420X_1 + 0.807X_2 - 1.445X_3 - 0.776X_1X_1 - 11.188X_2X_2 - 2.476X_3X_3 - 5.663X_1X_2 - 0.787\ X_1X_3 + 0.937\ X_2X_3 \quad (6)$$

$$Y_2 = 59.768 - 0.567X_1 - 17.604X_2 + 8.809X_3 - 0.142X_1X_1 - 6.050X_2X_2 - 8.692X_3X_3 - 7.167X_1X_2 + 1.718X_1X_3 + 1.634X_2X_3 \quad (7)$$

Positive sign in front of the terms indicates synergistic effect, whereas negative sign indicates antagonistic effect. The coefficient of the model for the response was estimated using multiple regression analysis technique included in the RSM. Fit quality of the models was judged from their coefficients of correlation and determination.

Statistical analysis

Eq.(4) has been used to visualize the effects of experimental factors on methylene blue adsorption capacity and adsorbent material yield percentage response in Figures. 6, 7, 8 and 9. The quality of the model developed was evaluated based on the correlation coefficient value. The R^2 values for Eqs.(6) and (7) were 0.988 and 0.939, respectively. Both the R^2 values obtained were relatively high (close to unity), indicating that there was a good agreement between the experimental and the calculated values of the determination coefficients of the multilinear regression from the models.

The adequacy of the models was further justified through analysis of variance (ANOVA). The ANOVA for the quadratic model for adsorbent material yield is listed in TABLE 3.

Reading the data in TABLE 3 reveals the validity of the model since the value of F_{exp} (22.67), which is the ratio between the Lack of fit and the pure error, is much lower than the critical value of Fisher ($F_{0.001(5.1)} = 5764$) at a 99.9 % level of confidence with 5 and 1 degrees of freedom.

The results of the ANOVA (TABLE 3) also shows that the experimental value of Scnedecor (Fexp= 53,33), which is the ratio between the square of the

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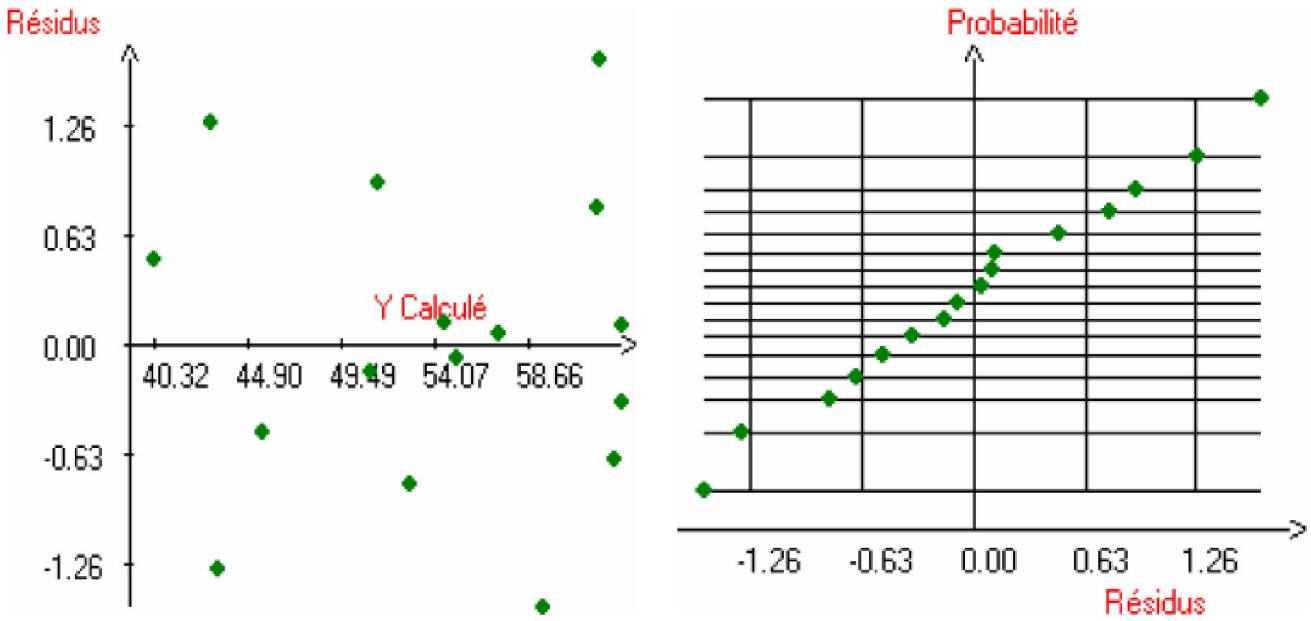
TABLE 4: Analysis of variance (ANOVA) for response surface quadratic model for methylene blue adsorption capacity (Y2).

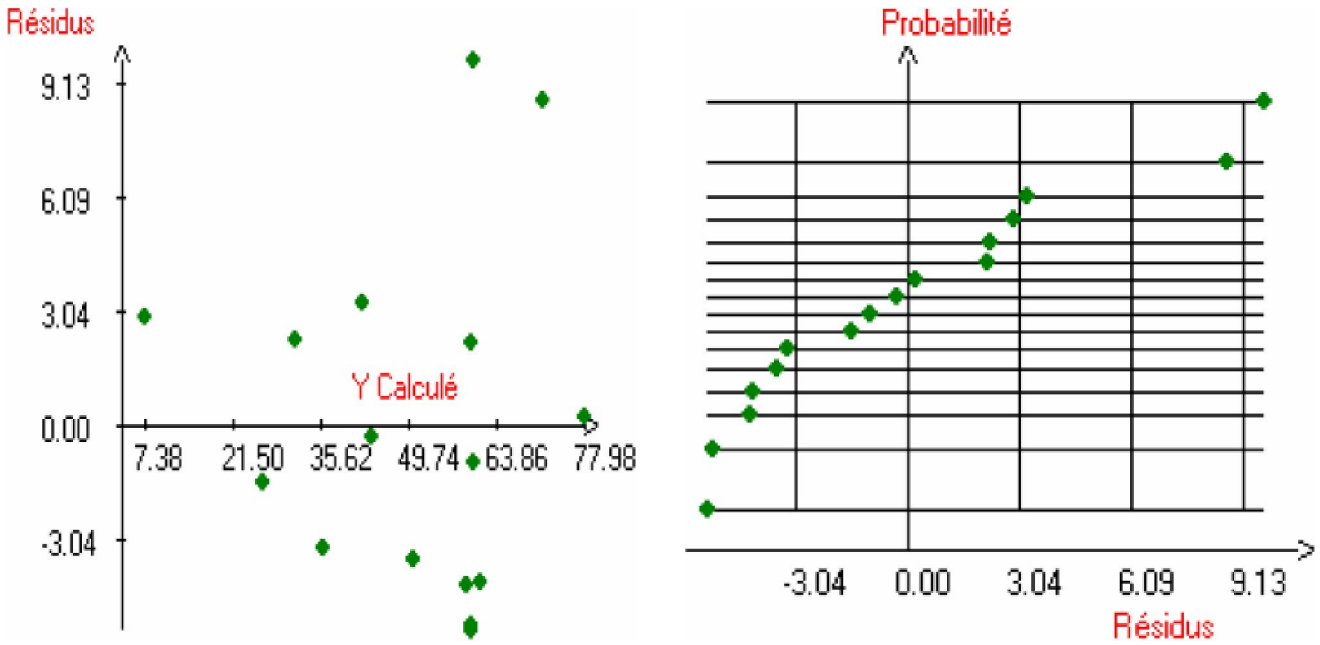
Source of variation	Sum of squares	Degree of freedom	Mean square	F_{exp}	Signif
Regression	4.94626E+0003	9	5.49585E+0002	38033.5531	0.827 **
Residual	3.21093E+0002	6	5.35154E+0001		
Lack of fit	3.21078E+0002	5	6.42156E+0001	4443.9870	1.43 *
Pure error	1.44500E-0002	1	1.44500E-0002		
Correlation total	5.26736E+0003	15			

**** Significant at a 99% level of confidence; * Significant at a 95 % level of confidence**



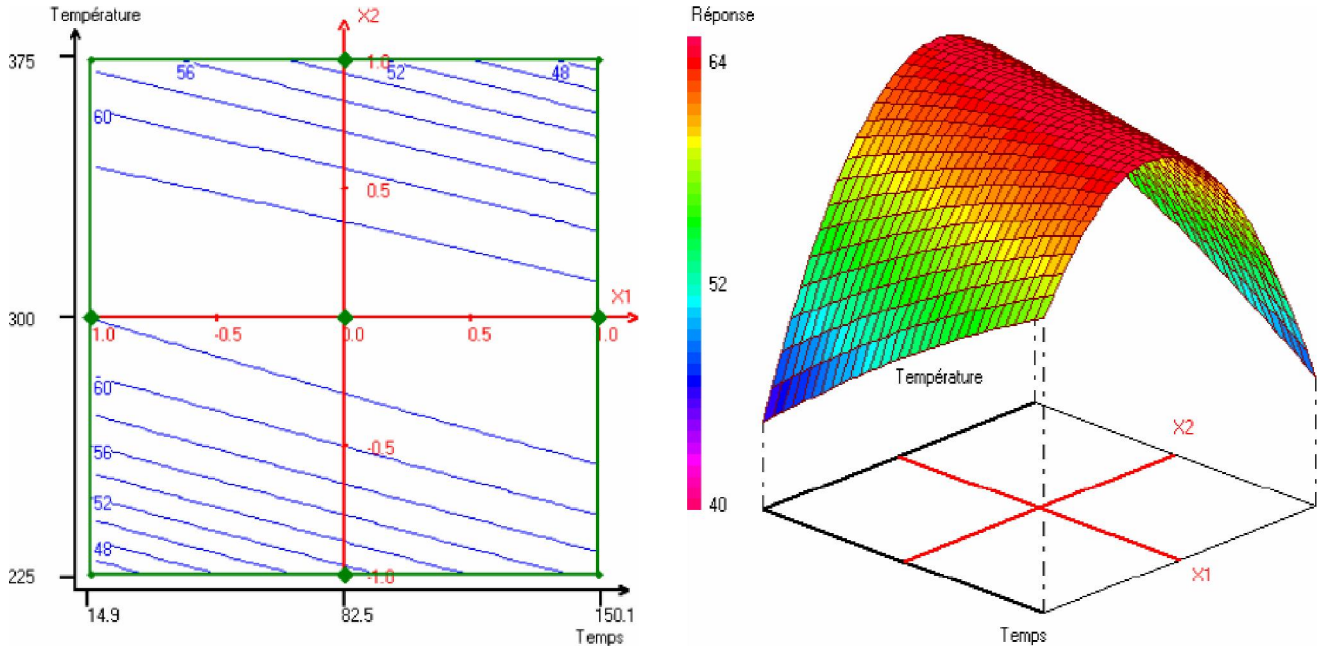
a- Graphical study of residues **b- Henry's straight line**

Figure 4 : Study of residues of response Y_1 (adsorbent material yield)



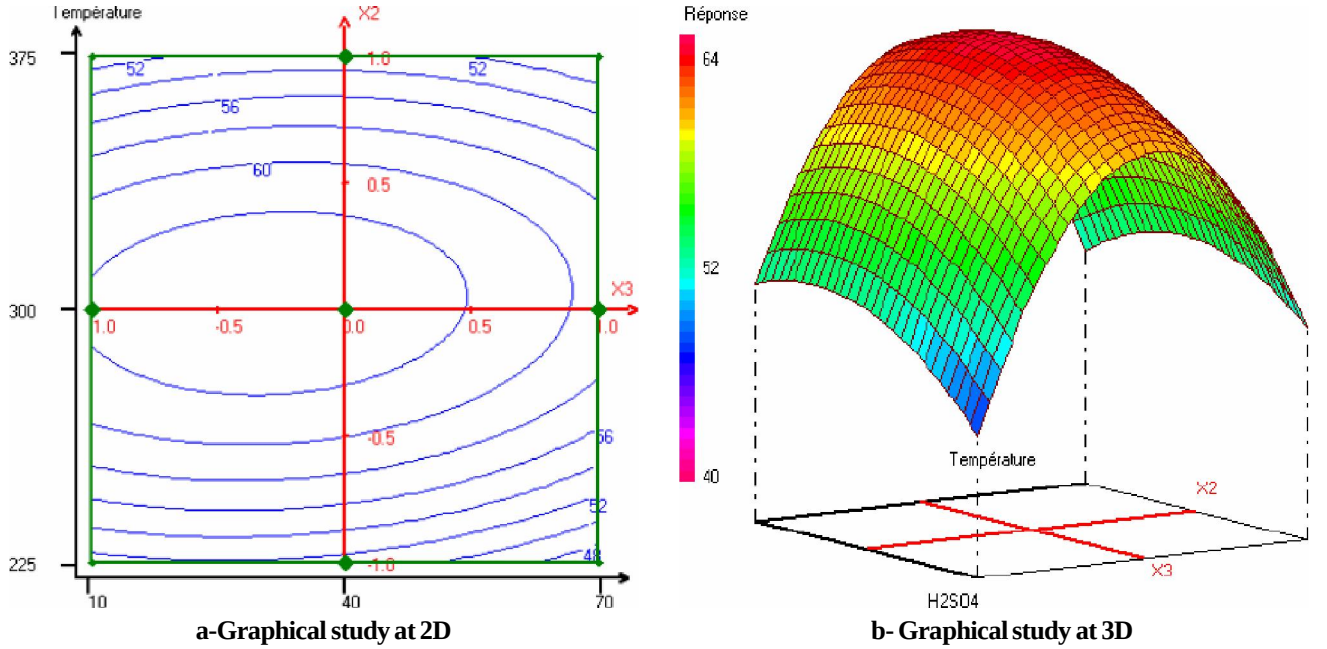
a- Graphical study of residues **b- Henry's straight line**

Figure 5 : Study of residues of response Y_2 (methylene blue adsorption capacity)



a- Graphical study at 2D b- Graphical study at 3D

Figure 6 : Isoresponse curves (a) and response surface (b) of adsorbent material yield: (The combined effect of activation temperature and time at constant percentage of chemical agent (40%)



a-Graphical study at 2D b- Graphical study at 3D

Figure 7 : Isoresponse curves (a) and response surface (b) of adsorbent material yield: (the combined effect of activation temperature and percentage of chemical agent at constant activation time (82,5 min)

model and the average mean square of the residue, is largely higher than the critical value of Fisher ($F_{0.001\,(9.6)}$ = 18,69) at a 99.9 % level of confidence with 9 and 6 degrees of freedom. Consequently, the regression is thus very significant and the model is considered corresponding.

The ANOVA for the quadratic model for methylene blue adsorption capacity is listed in TABLE 4. Reading the data in TABLE 4 reveals the validity of the model since the value of F_{exp} (4443,99) is lower than the critical value of Fisher ($F_{0.001\,(5.1)}$ = 5764) at a 99.9 % level of confidence with 5 and 1 degrees of freedom.

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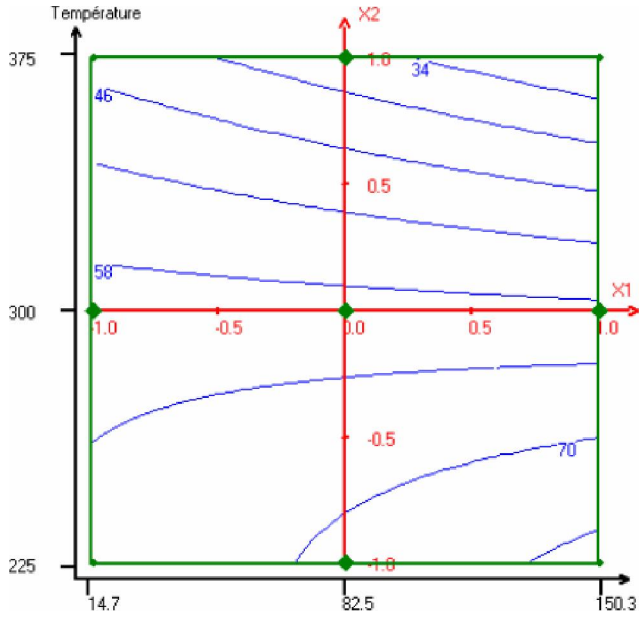
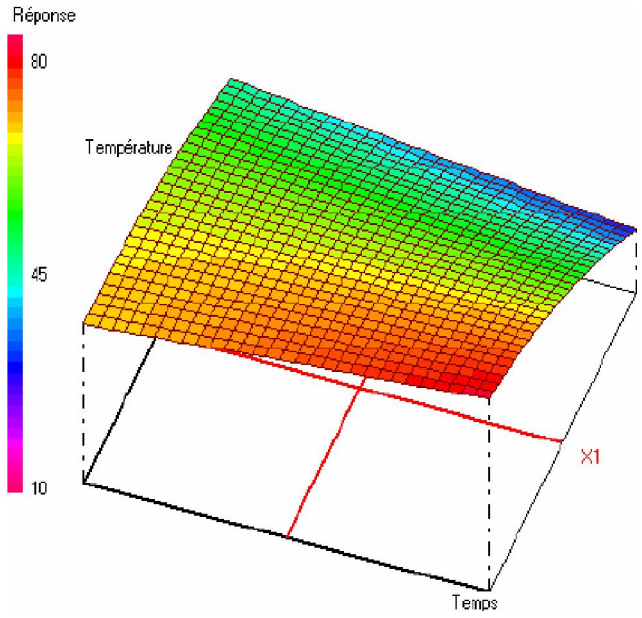
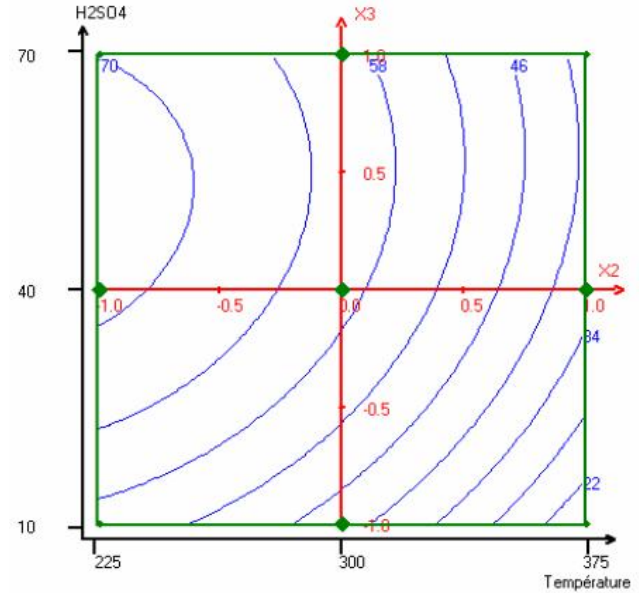


a- Graphical study at 2D

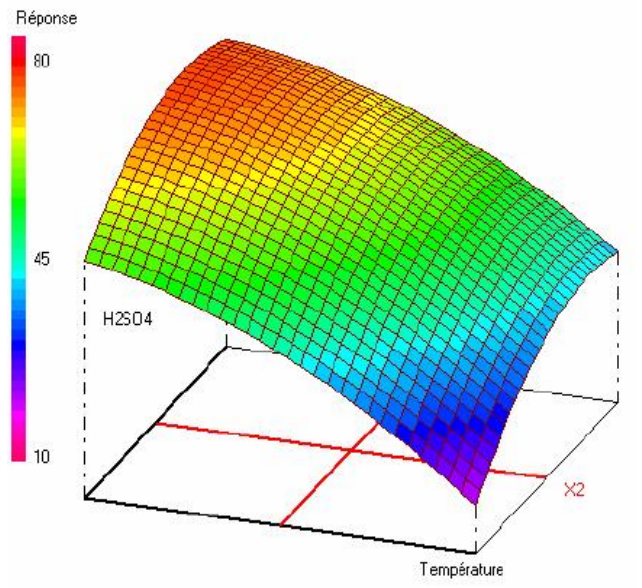


b- Graphical study at 2D

Figure 8 : Isoresponse curves (a) and response surface (b) of methylene blue adsorption capacity: (The combined effect of activation temperature and time at constant percentage of chemical agent (40%)



a- Graphical study at 2D



b- Graphical study at 2D

Figure 9 : Isoresponse curves (a) and response surface (b) of methylene blue adsorption capacity: (the combined effect of activation temperature and percentage of chemical agent at constant activation time (82,5 min)

The results of the ANOVA (TABLE 4) also shows that the experimental value of Scnedecor (Fexp= 38033,55), is largely higher than the critical value of Fisher ($F_{0.001\,(9.6)}$ = 18,69) at a 99.9 % level of confidence with 9 and 6 degrees of freedom. Consequently, the regression is thus very significant and the model is considered corresponding.

The residue analysis of responses Y_1 and Y_2 are shown in Figures 4 and 5. The representations as cloud point depending on the value of the calculated response (Figure 4a and 5a) allow to verify that the experimental variance remains constant regardless of the response values. Indeed, the residues are randomly distributed around zero. The Henry's straight line (Figure 4b and

TABLE 5 : Model validation

Activation time, X_1(min)	Activation temperature, X_2(°C)	Percentage of activating agent (%)	Adsorbent material yield, Y_1(%)		Adsorption capacity, Y_2 (%)	
			Predicted	Experimental	Predicted	Experimental
150,3	237	48	58,14	59,45	77,41	78,65

5b) allow to deduce that the residues follow a normal distribution; in fact, the points are almost aligned in a graph.

The overall quality of regressions is considered good with respect to the values of correlation factors R^2 multilinear and to the random dispersion of residues; this latter result is illustrated by the alignment of the points representing the values of the probability as a function of residues.

Adsorbent material yield

To investigate the effects of the three factors on the Adsorbent material yield, the response surface methodology was used, and two and three-dimensional plots were drawn. The Adsorbent material yield percent response surface graphs was shown in Figures. 6 and 7

The examination of the response surface corresponding to this model shows that:

b_2 coefficient (0.807) is the most important factor in the model, therefore the temperature (X2) is the most influential factor on the mass yield. Indeed the graphical study (Figure 6) shows that the increase in temperature indicates a significant improvement in mass yield of the adsorbent that exceeds 64% for the values of temperature around the 300 ° C which is then of the order of 40% for the temperature around the 225 and 375 ° C. Under these conditions, the temperature at 300 ° C remains the most suitable for a better development of the adsorbent material yield as the variation of temperature is substantially linear as a function of mass yield.

The influence of the activation time (X1) on the mass yield of the adsorbent material is less than the activation temperature (b1 = 0.420). The mass yield remains maximum throughout the time interval 15-150min (Figure. 6). This can be explained by a given time of 15 min, proves to be sufficient for maximum mass yield.

The influence of percentage of chemical activating agent on adsorbent material yield (b_3 = -1.445) is less than that of the temperature. The negative sign of b_3 proves that a decrease in percentage of chemical activating agent results an improvement of mass yield (Figure 7).

The isoresponse curves and the response surface (Figure 7) show clearly that the highest yield (64%) is achieved with a percentage of chemical activating agent = 30% and an activation temperature = 300 ° C.

Adsorption capacity on methylene blue

The effects of the three factors on the methylene blue adsorption were shown in Figure 8 and 9.

b_1 coefficient (- 0.567) of the time (X_1) in the model is more important than the temperature (X_2) (b_2 = - 17.60), it is therefore the most influential factor on the adsorption capacity. Indeed the graphical study shown in figure 8 indicates that the increase in the time resulting a significant improvement of the methylene blue adsorption capacity that exceeds 80% for the values of the time in the vicinity of 150 min. Under these conditions, the time at 150 min is the most appropriate for a better development of the adsorption capacity of the adsorbent material as the time variation is substantially linear as a function of the adsorption capacity.

The influence of the temperature of activation (X2) on the adsorption capacity of the adsorbent material is less than that of the activation time; the adsorption capacity is maximal at the temperatures in the vicinity of 225 °C (Figure. 8).

The influence of percentage of chemical activating agent (b3 = 8.809) on the adsorption capacity is very important compared to that of the temperature. The positive sign of b3 proves that an increase in of percentage of chemical activating agent results an improvement of the adsorption capacity (Figure 9).

The isoresponse curves and the response surface (Figure 9) show clearly that the maximum adsorption capacity (80%) is achieved with a percentage of chemical activating agent = 60% and an activation temperature = 225 °C.

Process optimization

In the production of adsorbent material, one of the main aims of this study was to find the optimum process parameters relatively high product yields are expected for economical feasibility and the most impor-

tant property of adsorbent material is its adsorption capacity. Therefore, the adsorbent material produced should have a high mass yield and also a high adsorption capacity for economical viability. However, to optimize both these responses under the same condition is difficult because the interest region of factors is different. When Y_1 increases, Y_2 will decrease and vice versa. Therefore, in order to compromise between these two responses, the function of desirability was applied using Design-Expert software NEMRODW. The experimental conditions with the highest desirability were selected to be verified. The adsorbent material was prepared under the experimental conditions given in TABLE 5, together with the predicted and experimental values for methylene blue adsorption capacity and adsorbent material yield. The optimal adsorbent material was obtained using preparation condition as: 150.3 min activation time, 237 °C activation temperature, and 48 % of H_2SO_4: chemical activating agent, which resulted in 58.14% of mass yield and 77.41% of methylene blue adsorption capacity. It was observed that the experimental values obtained were in good agreement with the values predicted from the models, with relatively small errors between the predicted and the actual values, which was 2.2% and 1.58%, respectively, for adsorbent material yield and methylene blue adsorption capacity.

CONCLUSION

The residues of marine sponges are a good precursor for the optimization of adsorbent materials with interesting characteristics (great adsorption capacity and high mass yield).

The response surface methodology is an appropriate tool to study optimization of the activation process to prepare adsorbents materials to be used in a given technological process.

In the present paper, this optimization was carried out to obtain adsorbent materials from marine sponges residues with suitable characteristics for use in water treatments.

The optimal adsorbent material was obtained using 237°C activation temperature, 150.3 min activation time and 48% of chemical activating agent, resulting in 58.14% of mass yield and 77.41 % of methylene blue adsorption capacity. The experimental values obtained for the methylene blue adsorption capacity and mass yield were found to agree satisfactorily with the values predicted by the models.

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