# Central Composite Design for the Modeling of the Phenol Adsorption Process in a Fixed-Bed Reactor

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The objective of this study is to optimize experimental conditions of the phenol adsorption process onto granular activated carbon by using an experimental design methodology. The process is studied in a fixed-bed reactor. A rotatable and orthogonal central composite design at five levels allowed us to acquire a second-order model with the terms of interaction between the four influential factors chosen. The optimal values of flow rate solution, carbon bed height, temperature, and initial phenol concentration of the solution are equal to 1.67 $mL \cdot s^{-1}$, 14.72 cm, 30 °C, and 94.35 $mg \cdot L^{-1}$, respectively. An optimal adsorption yield (99.33 %) is acquired after only 1 h of experimentation.

## 1. Introduction

Phenols are generally considered to be one of the important organic pollutants discharged into the environment causing an unpleasant taste and odor in drinking water. The major sources of phenol pollution in the aquatic environment are wastewaters from the paint, pesticide, coal conversion, polymeric resin, petroleum, and petrochemical industries. Introducing phenolic compounds into the environment or degradation of these substances results in the appearance of phenol and its derivatives in the environment. Phenols are considered priority pollutants since they are harmful to organisms at low concentrations.[1] Phenols are toxic and mutagenic substances at high concentrations and may be absorbed through the skin. The elimination of these contaminants is thus a major necessity for environmental protection. Several methods were proposed to treat these effluents; the adsorption process from aqueous solutions onto activated carbon is one of the most studied. The adsorption process depends on several parameters such as the pH of the solution, the initial concentration of the solution, the surface area, the particle size, and so forth. The development of mathematical models describing the process has proven to be difficult. The best strategy is to design an experiment. This methodology takes an important place thanks to the development of the computer tool; it allows, from a fewer number of runs, to extract meaningful information on the studied process.[2,3] Although experimental design methodology has largely been employed in various fields, chemistry, agriculture, biology, economy, and so forth, it is only recently becoming increasingly widespread in the adsorption process.[4–9]

This study is a continuation of earlier work accomplished within our research laboratory, on a batch-agitated reactor.[10] Here, we want to compare the results with those obtained in a fixed-bed reactor. The design chosen to carry out our experiments is a central composite design (CCD) at five levels respecting two optimality criteria: orthogonality and rotatability.[2,11,12] Four operating factors were chosen as independent variables, namely, initial phenol concentration, carbon bed height, flow rate, and temperature of the solution. With response surface methodology (RSM), the interaction of possible influencing parameters on phenol adsorption yield can be evaluated with a limited number of planned experiments.[2,13]

## 2. Materials and Methods

The adsorption experiments were carried out in the apparatus shown in Figure 1. For each experiment, a fresh solution of phenol and fresh granular activated carbon were used.

All of the chemicals used in this study were of analytical grade, and distilled water was used to prepare all of the phenol and sulfuric acid solutions ($C_6H_5-OH$; 99 % purity and $H_2SO_4$; 96 % purity). Fractions of solution are sampled at regular intervals over 2 h and analyzed by UV−vis spectrophotometry (Jenway IC 6305) at a wavelength corresponding to the maximum absorbance ($\lambda_{max} = 270$ nm). The pH of the solution has been kept constant at 3[8] by adding the required amount of $H_2SO_4$ solution.

The adsorbent used in this work was a commercial granular activated carbon UP07 which was provided by C.O.G.B. of Bejaia. The physical properties of this adsorbent are shown in Table 1.

Before use, the activated carbon was rinsed with distilled water, filtered, and dried overnight in an oven at 105 °C. The objective of this pretreatment was to eliminate impurities and fine particles.

## 3. Experimental Design Methodology

To obtain a reliable statistical model, prior knowledge of the procedure is generally required. The three steps used in the experimental design included statistical design experiments, estimation of coefficients through a mathematical model with response prediction, and statistical analysis.[2,3,11,12]

In this study, four operating factors, namely, flow rate ($Z_1$), carbon bed height ($Z_2$), temperature ($Z_3$), and initial phenol concentration ($Z_4$), were chosen as independent variables and

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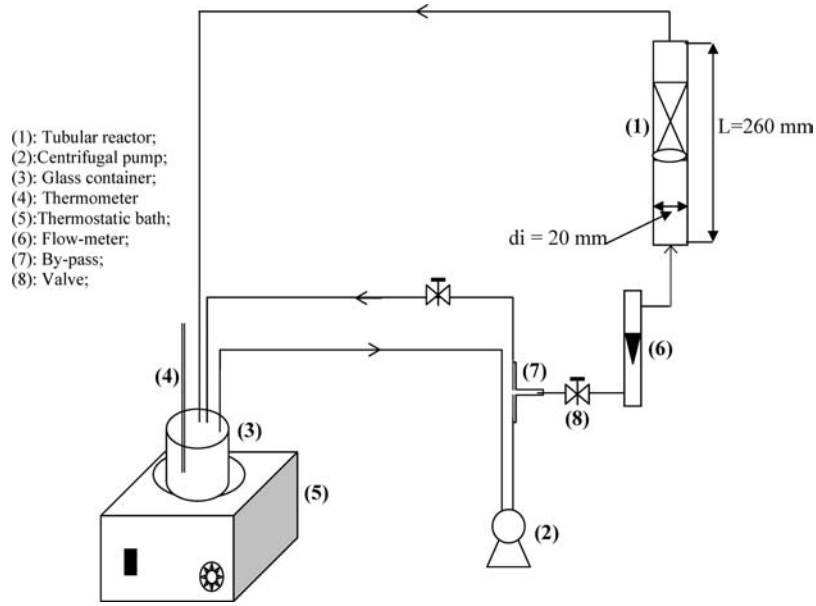


**Figure 1.** Schematic presentation of the experimental setup.

**Table 1. Physical Properties of the Activated Carbon**

| average characteristics | |
|---|---|
| average particle diameter (mm) | 1.00 |
| BET surface area ($m^2 \cdot g^{-1}$) | 632 |
| water content (%) | 0.71 |
| total pore volume ($cm^3 \cdot g^{-1}$) | 0.378 |
| porosity (%) | 73.11 |
| pore diameter ($\mu$m) | 0.7 |

**Table 2. Experimental Range and Levels of Operating Parameters**

| | levels | | | | |
|---|---|---|---|---|---|
| operating factors | −2 | −1 | 0 | 1 | 2 |
| $Z_1$: flow rate ($mL \cdot s^{-1}$) | 0.33 | 0.67 | 1.01 | 1.34 | 1.68 |
| $Z_2$: carbon bed height (cm) | 5 | 8 | 11 | 14 | 17 |
| $Z_3$: temperature (°C) | 20 | 25 | 30 | 35 | 40 |
| $Z_4$: initial phenol concentration ($mg \cdot L^{-1}$) | 10 | 35 | 60 | 85 | 110 |

the phenol adsorption yield as the dependent output response variable which is expressed as (%):

$$y(\%) = \frac{C_o - C_e}{C_o} \cdot 100 \tag{1}$$

where $C_o$ and $C_e$ are initial and equilibrium liquid-phase concentrations ($mg \cdot L^{-1}$), respectively. The experimental ranges are given in Table 2.

The experiments were carried out according to the CCD. It is an optimal design which allows, with a minimum number of experiments, calculation with best precision possible of the effects and interactions of each of the four factors chosen. This design consists of $2^k$ factorial points, augmented by $2k$ axial points, located at a specified distance $\delta$ from the center in each direction on each axis defined by the coded factor levels and $N_o$ center points. $k$ is the number of process factors. Center points, set to the midpoint of each factor range, provide information about the existence of curvature. Axial points allow the estimation of the pure quadratic properties of the model.[2,11–13]

Desirable properties of the design were selected, namely, rotatability and orthogonality. A design is rotatable if the variance of the response is constant for all variables at a given distance from the design center.[2,14] The CCD is rotatable if:

$$\delta = \sqrt[4]{2^k} \tag{2}$$

Orthogonality of design is a requisite for the evaluation of which of the linear, quadratic, and interaction effects are significant. This means that different variable effects can be estimated independently. The rotatable CCD would be nearly orthogonal if:

$$N_o = 4N_f^{0.5} - 2k + 4 \tag{3}$$

where $N_f$ is the number of factorial points ($N_f = 2^k$).

The center points are added to allow for the estimation of experimental error and condition uniformity and to take into consideration a check of the lack of fit of the model.[2]

The total number of design points $N$ of a rotatable design is determined from:

$$N = 2^k + 2k + N_o \tag{4}$$

The exploitation of the CCD matrix (Table 3) provides the coefficients of the second-order model:

$$\hat{y} = b_o + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + b_{11}x_{11}^2 + b_{22}x_{22}^2 + b_{33}x_{33}^2 + b_{44}x_{44}^2 \tag{5}$$

where $\hat{y}$ represents the estimated phenol adsorption yield, $b_o$ the average value of the response at the center point of the design, and $b_1$, $b_2$, $b_3$, $b_4$, $b_{12}$, $b_{13}$, $b_{14}$, $b_{23}$, $b_{24}$, $b_{34}$, $b_{11}$, $b_{22}$, $b_{33}$, and $b_{44}$ are the linear, interaction, and quadratic terms, respectively.

For any factor $Z_j$, the transformation from natural to coded values $x_j$ has been performed by considering the following equations:[11]

$$x_j = \frac{Z_j - Z_j^o}{\Delta Z_j} \qquad j = 1, ..., 4$$
$$\Delta Z_j = \frac{Z_j^{max} - Z_j^{min}}{2\delta} \tag{6}$$
$$\text{with } Z_j^o = \frac{Z_j^{max} + Z_j^{min}}{2}$$

$Z_j^{max}$ and $Z_j^{min}$ represent, respectively, the maximum and the minimum level of factor $j$ in the natural unit.

**Table 3. Central Composite Design Matrix and the Results**

| run no. | $x_1$ | $x_2$ | $x_3$ | $x_4$ | $y$ (%) | $\hat{y}$ (%) | $e_i$ (%) |
|---|---|---|---|---|---|---|---|
| 1 | −1 | −1 | −1 | −1 | 85.400 | 85.240 | 0.160 |
| 2 | +1 | −1 | −1 | −1 | 92.570 | 92.608 | −0.038 |
| 3 | −1 | +1 | −1 | −1 | 87.390 | 87.700 | −0.310 |
| 4 | +1 | +1 | −1 | −1 | 93.160 | 92.997 | 0.163 |
| 5 | −1 | −1 | +1 | −1 | 87.800 | 87.395 | 0.405 |
| 6 | +1 | −1 | +1 | −1 | 92.170 | 92.477 | −0.307 |
| 7 | −1 | +1 | +1 | −1 | 94.330 | 93.779 | 0.551 |
| 8 | +1 | +1 | +1 | −1 | 96.670 | 96.791 | −0.121 |
| 9 | −1 | −1 | −1 | +1 | 87.230 | 87.030 | 0.200 |
| 10 | +1 | −1 | −1 | +1 | 95.920 | 96.587 | −0.667 |
| 11 | −1 | +1 | −1 | +1 | 90.730 | 90.539 | 0.191 |
| 12 | +1 | +1 | −1 | +1 | 97.700 | 98.026 | −0.326 |
| 13 | −1 | −1 | +1 | +1 | 86.230 | 86.509 | −0.279 |
| 14 | +1 | −1 | +1 | +1 | 94.170 | 93.781 | 0.389 |
| 15 | −1 | +1 | +1 | +1 | 94.060 | 93.943 | 0.117 |
| 16 | +1 | +1 | +1 | +1 | 98.870 | 99.145 | −0.275 |
| 17 | 0 | 0 | 0 | 0 | 93.910 | 94.402 | −0.492 |
| 18 | 0 | 0 | 0 | 0 | 94.180 | 94.402 | −0.222 |
| 19 | 0 | 0 | 0 | 0 | 94.380 | 94.402 | −0.022 |
| 20 | 0 | 0 | 0 | 0 | 93.710 | 94.402 | −0.692 |
| 21 | 0 | 0 | 0 | 0 | 94.880 | 94.402 | 0.478 |
| 22 | 0 | 0 | 0 | 0 | 93.920 | 94.402 | −0.482 |
| 23 | 0 | 0 | 0 | 0 | 94.640 | 94.402 | 0.238 |
| 24 | 0 | 0 | 0 | 0 | 95.090 | 94.402 | 0.688 |
| 25 | 0 | 0 | 0 | 0 | 94.440 | 94.402 | 0.038 |
| 26 | 0 | 0 | 0 | 0 | 94.750 | 94.402 | 0.348 |
| 27 | 0 | 0 | 0 | 0 | 94.060 | 94.402 | −0.342 |
| 28 | 0 | 0 | 0 | 0 | 94.860 | 94.402 | 0.458 |
| 29 | −2 | 0 | 0 | 0 | 84.150 | 84.687 | −0.537 |
| 30 | +2 | 0 | 0 | 0 | 97.830 | 97.257 | 0.573 |
| 31 | 0 | −2 | 0 | 0 | 88.700 | 88.650 | 0.050 |
| 32 | 0 | +2 | 0 | 0 | 96.460 | 96.473 | −0.013 |
| 33 | 0 | 0 | −2 | 0 | 93.060 | 92.765 | 0.295 |
| 34 | 0 | 0 | +2 | 0 | 95.780 | 96.038 | −0.258 |
| 35 | 0 | 0 | 0 | −2 | 88.360 | 88.630 | −0.270 |
| 36 | 0 | 0 | 0 | +2 | 93.080 | 92.773 | 0.307 |

According to the CCD, 36 experiments were performed as shown in Table 3.

The 15 unknown coefficients of eq 5 are estimated by a multilinear regression based on the least-squares criterion. The matrix **B** of the coefficients of the model was calculated using relation 7[2,11] by using Excel software:

$$\mathbf{B} = (\mathbf{X}^t\mathbf{X})^{-1}\mathbf{X}^t\mathbf{Y} \tag{7}$$

where **B** is the column matrix of estimated coefficients; $(\mathbf{X}^t\mathbf{X})^{-1}$ the inverse of the dispersion matrix; $\mathbf{X}^t$ the transpose matrix of the effects matrix **X**; and **Y** is the column matrix of observations.

## 4. Results and Discussion

***4.1. Statistical Analysis.*** From a statistical point of view, three tests are required to evaluate the adequacy of the model: the Student's *t* test which is about the significance of factors, the Fisher test, and the *R*-square test.[2,3,11,14]

***4.1.1. Significance of Factors.*** With second-order models, we cannot obtain a dispersion matrix that is perfectly diagonal; at most, a quasi-orthogonality will be searched. Thus, the correlation matrix $\mathbf{X}^t\mathbf{X}$ deprived of its first line and its first column is then diagonal. In this case, the estimated coefficients of the regression model are not correlated; the elimination of a nonsignificant coefficient will not have a consequence on the values of the other coefficients.[14] Their significance can be checked separately by the Student's *t* test.

The coefficient $b_j$ is significantly different from zero if the value $t_j$ of eq 8 is larger than the *t* value given by the table of Student relating to a bilateral test for a level of significance chosen, $\alpha = 0.05$ and $f = 11$ degrees of freedom.

$$t_j = \frac{|b_j|}{\sigma_{bj}}$$
$$\sigma_{bj}^2 = C_{jj}\sigma_{\text{rep}}^2$$
$$\text{and } \sigma_{\text{rep}}^2 = \frac{\sum_1^{N_o}(y_{oi} - \bar{y}_o)^2}{(N_o - 1)} \tag{8}$$

where $b_j$ is the *j*th coefficient of the process parameter; $\sigma_{bj}^2$ the coefficient variance; $C_{jj}$ the diagonal terms of the $(X^tX)^{-1}$ matrix; $\sigma_{\text{rep}}^2$ the replication variance; $y_{oi}$ the observed value of adsorption yield for the $i^{\text{th}}$ central point; $\bar{y}_o$ the average value of adsorption yield for the central point; and $N_o$ the repetition number of the experiments at the center work domain. It was obtained that all individual effects are significant at a 5 % significance level and only the coefficient $b_{33}$ is not significant. Therefore, it is excluded from the regression equation.

***4.1.2. Reliability of the Model.*** The test of reliability for the predicting equation has been carried out by the Fisher's variance ratio test known as the *F*-test.[2] This test compared the residual variance with the replication variance. The *F*-ratio is given by the following form:

$$F = \frac{\sigma_{\text{res}}^2}{\sigma_{\text{rep}}^2}$$
$$\text{and}$$
$$\sigma_{\text{res}}^2 = \frac{\sum_i (y_i - \hat{y}_i)^2}{N - p} \tag{9}$$

where $\sigma_{\text{res}}^2$ is the residual variance; $\sigma_{\text{rep}}^2$ the replication variance; *N* the total number of observations ($N = 36$); *p* the number of

**Table 4. Analysis of Variance for the Second-Order Model[a]**

| source of variations | SS | *f* | MS | *F*-value |
|---|---|---|---|---|
| residual error | 4.82 | 22 | 0.22 | 1.09 |
| experimental error | 2.21 | 11 | 0.20 | |

[a] SS represents the sum of squares, $f$ = degrees of freedom, and MS the mean squares.

coefficients in the regression equation ($p = 14$); $y_i$ the observed value of adsorption yield for the $i$th observation, and $\hat{y}_i$ the estimated value of adsorption yield for the $i^{th}$ observation.

The tabulated $F$ value for the 5 % significance level and degrees of freedom $f_1$ and $f_2$ ($f_1 = N - p = 22$ and $f_2 = N_0 - 1 = 11$) is between 2.57 and 2.61. Table 4 gives the results of the variance analysis.

It was found that the estimated value of $F$ is much less than this interval. The two variances are then statistically equal: the adjustment error between the real model and the postulated model is negligible in front of the experimental error. Hence, it can be concluded that the established predicting equation gives an excellent fit to the observed data. The coefficient of determination $R^2$ was 99.99 %, therefore indicating a high degree of correlation between the response and the independent variables in two responses (experimental and predicted values). Figure 2 demonstrates the strong correlation between $\hat{y}$ (predicted values) and $y$ (experimental values), which is very good for the goodness of fit.

The model equation for phenol adsorption on activated carbon obtained after performing 36 experiments and discarding the insignificant effects is as follows:

$$\hat{y} = 94.40 + 3.14x_1 + 1.96x_2 + 0.82x_3 + 1.04x_4 - 0.52x_1x_2 - 0.57x_1x_3 + 0.55x_1x_4 + 0.99x_2x_3 + 0.26x_2x_4 - 0.67x_3x_4 - 0.86x_1^2 - 0.46x_2^2 - 0.93x_4^2 \quad (10)$$

The analysis of the regression equation shows that the principal effects of the four selected factors have an influence on the phenol adsorption yield. The flow rate ($x_1$) has the strongest effect on the response since the corresponding coefficient ($b_1 = 3.14$) is larger than the coefficients of the other investigated factors. The positive sign of this coefficient indicates that an increase in the flow rate improves the adsorption yield. This effect is explained by the decrease of the diffusional boundary layer thickness surrounding the adsorbent, with the increase of the flow rate which favors the orientation of the phenol molecules toward the most energetic sites. The corresponding coefficient ($b_3 = 0.82$) of temperature ($x_3$) is the lowest of the four factors. This result is in agreement with those found in the kinetic study carried out in a closed agitated reactor.[14] This indicates the exothermic nature of the process. The quadratic effect ($x_3^2$) of the temperature is negligible.

**Table 5. Optimum Values of the Process Parameter for Maximum Adsorption Yield**

| | optimum values | |
|---|---|---|
| parameters | coded units ($x_i$) | natural units ($Z_i$) |
| flow rate | 1.956 | 1.67 mL·s$^{-1}$ |
| carbon bed height | 1.241 | 14.72 cm |
| temperature | −0.167 | 29.17 °C |
| phenol initial concentration | 1.374 | 94.35 mg·L$^{-1}$ |

The design of experiments for phenol adsorption yield also exhibits the existence of interactions between the various factors studied. This result shows the weakness of conventional methods which do not take into account these effects.

***4.2. Process Optimization.*** Optimization consists in finding the whole of the values of the operational variables which involves an optimal adsorption yield. The localization of the optimum point can be obtained in various manners.[2,11,12] Having a model, the layout of the surface contours plot remains the easiest method to interpret. These curves are significant to visualize the behavior of the phenomenon studied, also, to choose the zone of the work study which allows a better output. The established model is written in the following matrix form:

$$\hat{y} = b_o + x_k^t b_k + x_k^t \mathbf{B} x_k \quad (11)$$

where $b_k$ is the matrix column of the coefficients of the first order terms; **B** is the central matrix; and $x_k^t$ is the vector transposed of the punctual coordinates in the work domain considered.

The matrix $B$ is built as follows:

$$\mathbf{B} = \begin{bmatrix} b_{11} & \frac{1}{2}b_{12} & \frac{1}{2}b_{13} & \frac{1}{2}b_{14} \\ \frac{1}{2}b_{12} & b_{22} & \frac{1}{2}b_{23} & \frac{1}{2}b_{24} \\ \frac{1}{2}b_{13} & \frac{1}{2}b_{23} & b_{33} & \frac{1}{2}b_{34} \\ \frac{1}{2}b_{14} & \frac{1}{2}b_{24} & \frac{1}{2}b_{34} & \frac{1}{2}b_{44} \end{bmatrix} \quad (12)$$

Optimal values of the process parameters were first calculated in coded units by using relation 13 and then converted to natural units by eq 6. The results are shown in Table 5.

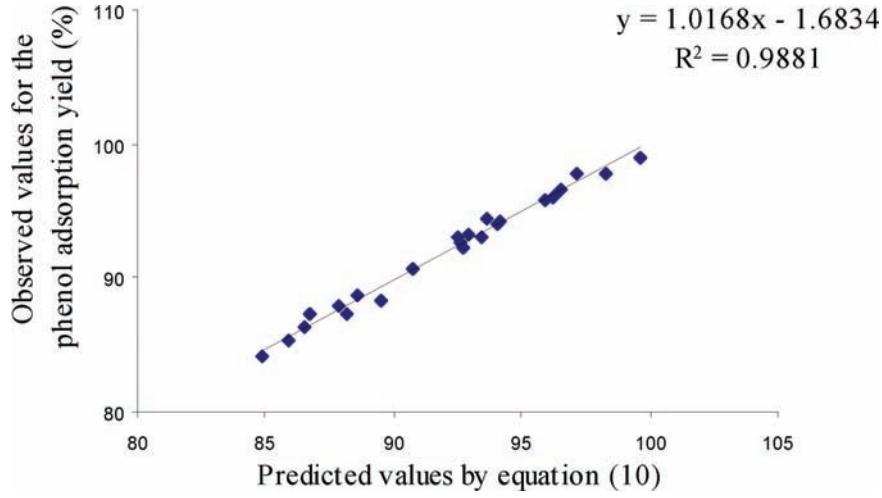


**Figure 2.** Experimental values and values predicted by eq 10.

$$x_s = -\frac{1}{2}\mathbf{B}^{-1}b_k \quad (13)$$

The optimal adsorption yield obtained by the model reaches 99.33 %; this result closely agrees with the adsorption yield of 98.87 % obtained by the experiment (in run 16). The response surface and contour plots given in Figures 3 and 4 are drawn by using the "Matlab 7.0" software in the plane concentration or carbon bed height when the remaining variables (flow rate and temperature) are kept at their optimal values. These curves allow us to determine the region of the work domain where the phenol adsorption yield is optimum.[15] The desired objective is to eliminate a great quantity of phenol by using a minimal

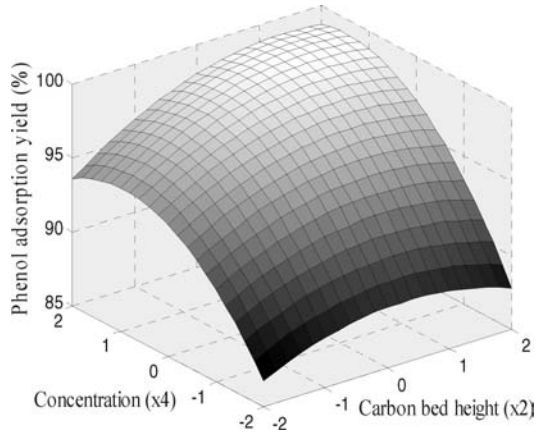


**Figure 3.** Response surface plot for phenol adsorption yield in the plane concentration or carbon bed height at optimal values of flow rate and temperature ($x_1 = 1.953$ and $x_3 = -0.168$).

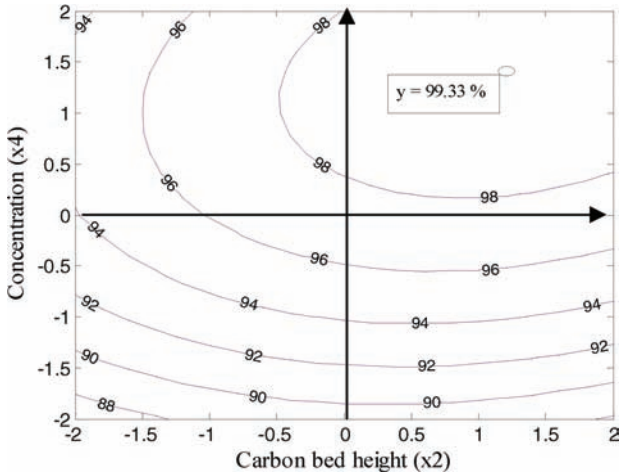


**Figure 4.** Response contour plot for phenol adsorption yield in the plane concentration or carbon bed height at optimal values of flow rate and temperature ($x_1 = 1.953$ and $x_3 = -0.168$).

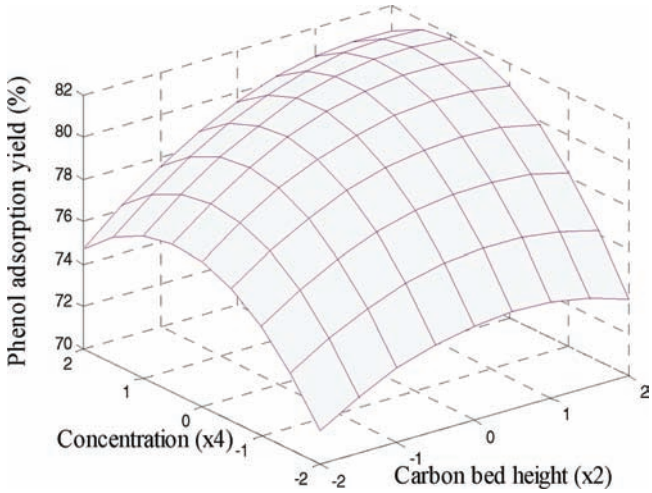


**Figure 5.** Response surface plot for phenol adsorption yield in the plane concentration or carbon bed height at $x_1 = -2$ and $x_2 = -2$.

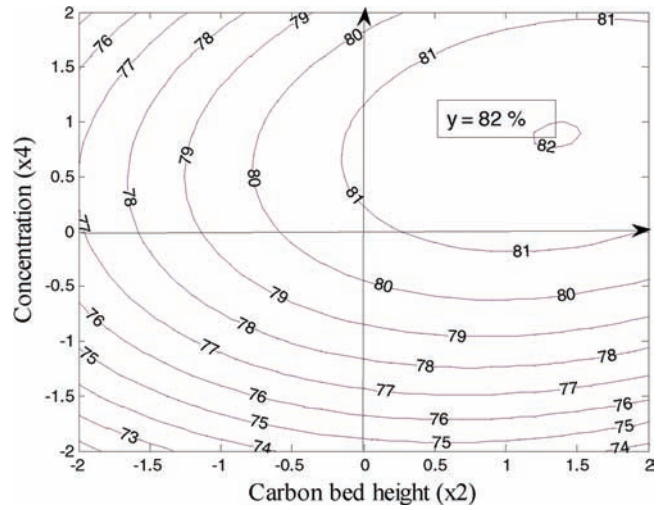


**Figure 6.** Response contour plot for phenol adsorption yield in the plane concentration or carbon bed height at $x_1 = -2$ and $x_2 = -2$.

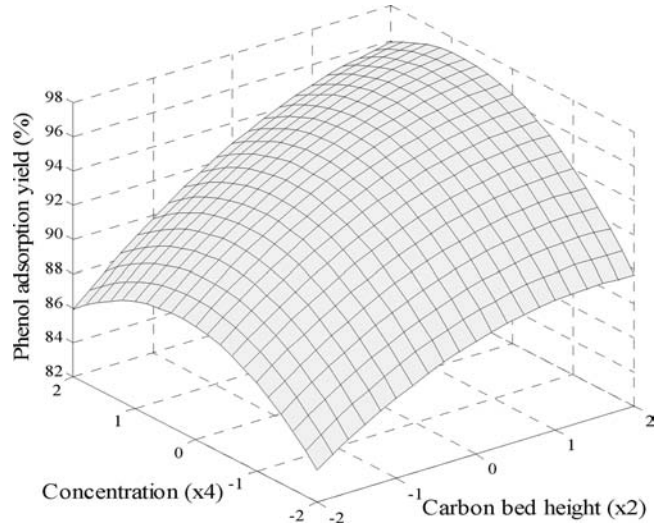


**Figure 7.** Response surface plot for phenol adsorption yield in the plane concentration or carbon fixed bed at $x_1 = 0$ and $x_2 = 0$.

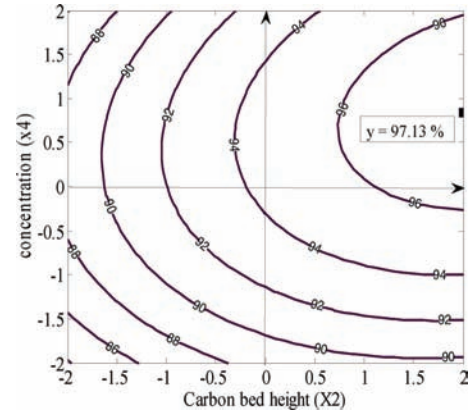


**Figure 8.** Response contour plot for phenol adsorption yield in the plane concentration or carbon fixed bed at $x_1 = 0$ and $x_2 = 0$.

quantity of activated carbon. We see that these figures confirm well the coordinates of the optimal point obtained.

Figures 5 and 6 show well that for low levels (−2) of the flow rate and the temperature which correspond respectively to 0.33 $mL \cdot s^{-1}$ and 20 °C, the largest adsorption yield does not exceed 82 %, whatever the bed height and initial concentration of phenol used.

In Figures 7 and 8 we can see that, for medium values of temperature and flow rate (level 0), the phenol adsorption yield is important but corresponds to a great quantity of activated carbon.

## 5. Conclusion

The experimental design methodology was used successfully for modeling the phenol adsorption process on commercial activated carbon in a fixed-bed reactor. A significant adsorption yield is obtained after an equilibrium time of only 60 min. The temperature has a weak influence on the adsorption yield in the interest domain considered; the optimal adsorption yield is obtained at 29.17 °C. The increase in the flow rate has a positive effect on the percent removal of phenol, and its optimal value is 1.67 $mL \cdot s^{-1}$. The observation of surface contour plots in the plane carbon bed height or initial phenol concentration allows choosing the favorable and economical conditions driving to a satisfactory phenol adsorption yield.

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