# Applicability of some statistical tools to predict optimum adsorption isotherm after linear and non-linear regression analysis

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## Abstract

In any single component isotherm study, determining the best-fitting model is a key analysis to mathematically describe the involved sorption system and, therefore, to explore the related theoretical assumptions. Hence, several error calculation functions have been widely used to estimate the error deviations between experimental and theoretically predicted equilibrium adsorption values ($Q_{e,exp}$ vs. $Q_{e,theo}$ as $X$- and $Y$-axis, respectively), including the average relative error deviation, the Marquardt's percent standard error deviation, the hybrid fractional error function, the sum of the squares of the errors, the correlation coefficient and the residuals. In this study, five other statistical functions are analysed to investigate their applicability as suitable tools to evaluate isotherm model fitness, namely the Pearson correlation coefficient, the coefficient of determination, the Chi-square test, the $F$-test and the Student's $T$-test, using the commonly-used functions as references. The adsorption of textile dye onto *Posidonia oceanica* seagrass fibres was carried out, as study case, in batch mode at 20 °C. Besides, and in order to get an overall approach of the possible utilization of these statistical functions within the studied item, the examination was realized for both linear and non-linear regression analysis. The related results showed that, among the five studied statistical tools, the $\chi^2$ and Student's $T$-tests were suitable to determine the best-fitting isotherm model for the case of linear modelling approach. On the other hand, dealing with the non-linear analysis, despite the Student's $T$-test, all the other functions gave satisfactorily results, by agreeing the commonly-used error functions calculation.
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*Keywords:* Adsorption; Isotherm; Modelling; Error calculation; *Posidonia oceanica*; Textile dye

## 1. Introduction

The removal of dyes from industrial effluents is a field of research receiving increasing attention in the scientific community. The presence of very low concentrations of dyes in effluent is highly visible and undesirable. Moreover, they are very stable and difficult to degrade due to their resistance to heat, light, water and oxidising agents [1]. Among several dye removal technologies including nanofiltration membranes [2], chemical catalysis [3] and microbial degradation [4], the biosorption seems to have a real promising potential to be widely used as an eco-friendly and cheap method for textile effluents decontamination [5–7].

Equilibrium data, commonly known as adsorption isotherms, are the basic requirements for the design of adsorption systems. In order to optimize the design of a specific sorbate/sorbent system to remove dyes from effluents, it is important to establish the most appropriate correlation for the experimental equilibrium data (curves). Previously, some researches were carried out within such issue [8–10]. Many isotherm equations are commonly appearing in the biosorption literature including Langmuir [11], Freundlich [12], Redlich–Peterson [13], Temkin [14] and Elovich [15]. Besides, to sufficiently use the theoretical assumptions behind these mathematical equations, several error deviation functions have been used to adequately measure the goodness of fit of the models, such as the correlation coefficient ($R^2$), the sum of the squares of the errors (SSE), the average relative error deviation (ARED), the Marquardt's percent standard error deviation (MPSED), the hybrid fractional error function (HYBRID) and the residual analysis (RESID).

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The main aim of this research was to investigate, for both linear and non-linear analysis, the applicability of other known statistical tools, namely the Pearson correlation coefficient ($r$), the coefficient of determination ($r^2$), the Chi square test ($\chi^2$), the $F$-test and the Student's $T$-test in determining the best-fitting isotherm model(s). In the present research work, the single component sorption study case was the biosorption of direct Solophenyl Brown textile dye onto Mediterranean *Posidonia oceanica* fibrous biomass in liquid-phase batch system. The theoretically predicted isotherm data were determined using the Microsoft Excel for linear analysis and SPSS 13.0 statistic software for non-linear assessment. The random initial values used to perform the non-linear analysis were the ones deduced from the linear analysis, in order to optimize the iteration procedure. Furthermore, for the special case of three parameters Redlich–Peterson model, the runs were performed using three random initial values (i.e. ± values deduced from the linear analysis).

## 2. Materials and methods

### 2.1. Biomass and dye solution preparation

*P. oceanica* biomass was harvested from Chott Mariem bay (Sousse, Tunisia). The fibres were dried, crushed, and washed thoroughly with distilled water to remove the adhering dirt. They were air dried in an oven at 40 °C for 48 h. After drying, the biosorbent was blended then sieved through a 2 mm mesh size and stored in desiccators.

An accurately weighed quantity of Azo direct dye Solophenyl Brown was dissolved in distilled water to prepare stock solution (500 mg/L). Experimental solutions of the desired concentrations were obtained by successive dilutions. Spectrophotometric scanning of dilute dye solutions was performed and absorbance maximum was identified as 420 nm. Optimum pH range was adjusted between 2 and 2.5 by addition of dilute HCl solution [16].

### 2.2. Batch biosorption protocol

Biosorption equilibrium assays were carried out by adding the dried sorbent (2.5 g) in 250 ml of dye solution with desired concentration and pH at 20 °C in a shaking water bath at 100 rpm. After 24 h (needed time to reach equilibrium [16]), the suspension was filtered and the final concentration of dye in solution was measured using the spectrophotometeric technique. The amount of dye adsorbed onto *P. oceanica* fibres, $Q_e$ (mg/g), was calculated by a mass balance relationship:

$$Q_e = \frac{(C_0 - C_e) \times V}{W} \, (\text{mg/g}) \tag{1}$$

where $C_0$ and $C_e$ are the initial and equilibrium liquid-phase concentrations of dye, respectively (mg/L), $V$ the volume of the solution (L), and $W$ the weight of the dry sorbent used (g).

### 2.3. Best-fitting isotherm model estimation

#### 2.3.1. Commonly used error functions
##### 2.3.1.1. Average relative error deviation (ARED). The main advantage of this error function is the minimization of the fractional error distribution across the entire studied concentration range [17]

$$\text{ARED} = \frac{1}{N} \sum \left| \frac{Q_{e,cal} - Q_{e,exp}}{Q_{e,exp}} \right| \times 100 \tag{2}$$

where $N$ is the number of experimental data points, $Q_{e,cal}$ (mg/g) is the theoretically calculated adsorption capacity at equilibrium and $Q_{e,exp}$ (mg/g) is the experimental adsorption capacity at equilibrium.

##### 2.3.1.2. The sum of the squares of the errors (SSE). This error estimation method is represented by Eq. (3).

$$\text{SSE} = \sum (Q_{e,cal} - Q_{e,exp})^2 \tag{3}$$

Despite its wide use, this function has a major drawback. Indeed, the calculated isotherm parameters derived from such error function will provide a better fit at the higher end of the liquid-phase concentration range. This is because the magnitude of the errors and hence the square of the errors will increase as concentration increases [18].

##### 2.3.1.3. The Marquardt's percent standard deviation (MPSED). This error estimation tool [19] was previously used by many researches in different research fields [20,21]. It is similar to a geometric mean error distribution which was modified to allow for the number of degrees of freedom of the system.

$$\text{MPSED} = \sqrt{\frac{\sum [(Q_{exp} - Q_{cal})/Q_{exp}]^2}{N - P}} \times 100 \tag{4}$$

where $P$ is the number of parameters in each isotherm model.

##### 2.3.1.4. The hybrid fractional error function (HYBRID). This error function (Eq. (5)) was developed [22] in order to improve the fit of the SSE method at low concentration values. In this approach, each SSE value was divided by the experimental solid-phase concentration $Q_e$ value. Furthermore, a divisor was included as a term for the number of degrees of freedom for the system (i.e. the number of data points minus the number of parameters within the isotherm equation).

$$\text{HYBRID} = \frac{1}{N - P} \sum \left| \frac{Q_{e,exp} - Q_{e,cal}}{Q_{e,exp}} \right| \times 100 \tag{5}$$

#### 2.3.2. Alternative statistical functions
##### 2.3.2.1. Correlation coefficient of Pearson (r). The Pearson correlation coefficient (Eq. (6)) is a sampling index, varying from −1 to 1, reflecting the degree linearity of between two

Table 1
Mathematical equation of the used single component isotherm models

| Isotherm model | Equation | Linear expression | Plot |
|---|---|---|---|
| Freundlich | $Q_e = K_F(C_e)^{1/n}$ | $\ln Q_e = \ln K_F + \frac{1}{n}(\ln C_e)$ | $\ln Q_e$ vs. $\ln C_e$ |
| Langmuir | $Q_e = \frac{Q^{\circ} K_L C_e}{1+K_L C_e}$ | $\frac{C_e}{Q_e} = \frac{1}{K_L Q^{\circ}} + \frac{C_e}{Q^{\circ}}$ | $C_e/Q_e$ vs. $C_e$ |
| Redlich–Peterson | $Q_e = \frac{A_{RP} C_e}{1+K_{RP}(C_e)^{\beta}}$ | $\ln\left(\frac{a_{RP}C_e}{Q_e} - 1\right) = \beta \ln(C_e) + \ln K_R$ | $\ln((A_{RP}C_e/Q_e) - 1)$ vs. $\ln(C_e)$ |
| Temkin | $Q_e = \frac{RT}{b} \ln(K_T C_e)$ | $Q_e = B_T \ln(K_T) + B_T \ln(C_e)$ | $Q_e$ vs. $\ln(C_e)$ |
| Elovich | $\frac{Q_e}{Q_m} = K_E Ce \exp\left(-\frac{Q_e}{Q_m}\right)$ | $\ln\left(\frac{Q_e}{C_e}\right) = \ln(K_E Q_m) - \frac{Q_e}{Q_m}$ | $\ln(Q_e/C_e)$ vs. $Q_e$ |

$K_F$: Freundlich isotherm constant $[(mg/g)(L/mg)(1 - n/n)]$; $R$: universal gas constant, 8.314 J/mol; $n$: Freundlich exponent (dimensionless); $T$: absolute temperature in K; $Q^{\circ}$: Langmuir monolayer adsorption capacity (mg/g); $b$: variation of adsorption energy (kJ/mol); $K_L$: Langmuir isotherm constant (L/mg); $K_T$: Temkin equilibrium constant (L/mg); $A_{RP}$: Redlich–Peterson isotherm constant $[(L/mg)\beta]$; $B_T = RT/b$: factor related to the heat of adsorption; $K_{RP}$: Redlich–Peterson isotherm constant (L/g); $K_E$: Elovich equilibrium constant (L/mg); $\beta$: Redlich–Peterson exponent (dimensionless); $Q_m$: Elovich maximum adsorption capacity (mg/g).

dependant data series ($X$ and $Y$).

$$r = \frac{N\left(\sum XY\right) - \left(\sum X\right)\left(\sum Y\right)}{\sqrt{\left[N\sum X^2 - \left(\sum X\right)^2\right]\left[N\sum Y^2 - \left(\sum Y\right)^2\right]}} \quad (6)$$

*2.3.2.2. Coefficient of determination ($r^2$).* The coefficient of determination represents the percentage of variability in the dependent variable that has been explained by the regression line. The value of the coefficient of determination, calculated from Eq. (7), may vary from 0 to 1.

$$r^2 = \frac{S^2}{S_{(XX)}S_{(YY)}} \quad (7)$$

where $S_{(XY)}$ is the sum of squares of $X$ and $Y$, $S_{(XX)}$ is the sum of squares of $X$ and $S_{(YY)}$ is the sum of squares of $Y$.

*2.3.2.3. Chi-square test ($\chi^2$).* The Chi-square statistic test (Eq. (8)) is basically the sum of the squares of the differences between the experimental data and theoretically predicted data from models.

$$\chi^2 = \sum \frac{(Q_{e,exp} - Q_{e,cal})^2}{Q_{e,cal}} \quad (8)$$

*2.3.2.4. Other statistical tools.* In addition to the previously mentioned error equations, two other statistical tests, namely *F*-test and Student's *T*-test, are examined for their suitability to be appropriate functions to predict best-fitting isotherm model.

## 3. Results and discussion

### 3.1. Adsorption isotherm modelling

The successful representation of the dynamic adsorptive separation of solute from solution onto a sorbent depends upon a good description of the equilibrium separation between the two phases. By plotting solid-phase concentration against residual liquid-phase concentration graphically it is possible to depict the equilibrium adsorption isotherm. In order to optimize the design of a sorption system to remove dyes from aqueous solution, it is important to establish the most appropriate correlation for the

equilibrium curve. There are many theories relating to adsorption equilibrium and among the used models are Freundlich, Langmuir, Redlich–Peterson, Temkin and Elovich. The mathematical equations of these models are illustrated in Table 1 and their associated parameters are given in Table 2 for both linear and non-linear analysis. The fitting presentations are shown in Figs. 1 and 2 for linear and non-linear regression analysis, respectively.

In the following sections, firstly, the best-fitting model will be determined using the most commonly used error functions. Then, the applicability of the proposed statistical tools will be discussed, based on a comparative study between them and the widely used error estimation methods.

### 3.2. Error estimation using commonly used functions

In this section, among the five studied isotherms models, the best-fitting one is determined based on the use of six well-known functions to calculate the error deviation between experimental and predicted equilibrium adsorption data, after both linear and

Table 2
Linear and non-linear Freundlich, Langmuir, Redlich–Peterson, Temkin and Elovich isotherms constants related to the biosorption of Solophenyl Brown textile dye onto raw *P. oceanica* fibres

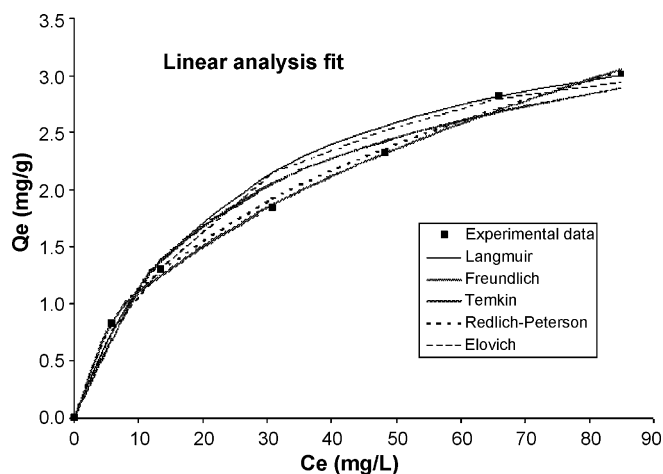|  | Linear method | Non-linear method |
|---|---|---|
| **Freundlich model** | | |
| $K_F$ | 0.353 | 0.361 |
| $n$ | 2.050 | 2.072 |
| **Langmuir model** | | |
| $Q^{\circ}$ | 3.890 | 3.999 |
| $K_L$ | 0.036 | 0.033 |
| **Redlich–Peterson model** | | |
| $A_{RP}$ | 0.552 | 1.189 |
| $K_{RP}$ | 1.023 | 2.806 |
| $\beta$ | 0.598 | 0.548 |
| **Temkin model** | | |
| $B_T$ | 0.828 | 0.829 |
| $K_T$ | 0.388 | 0.388 |
| **Elovich model** | | |
| $Q_m$ | 1.671 | 1.966 |
| $K_E$ | 0.392 | 0.385 |

Fig. 1. Adsorption isotherm modelling of Solophenyl brown dye removal using raw *P. oceanica* fibres using *linear regression analysis* (biomass concentration = 10 g/L, pH 2 and temperature = 20 ± 2 °C).
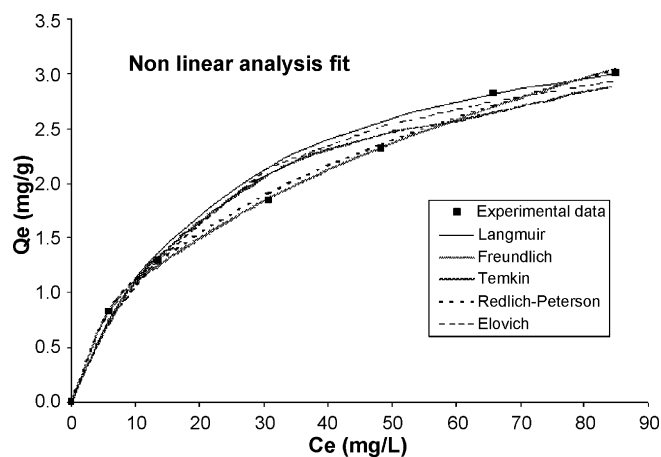


Fig. 2. Adsorption isotherm modelling of Solophenyl brown dye removal using raw *P. oceanica* fibres using *non-linear regression analysis* (biomass concentration = 10 g/L, pH 2 and temperature = 20 ± 2 °C).

non-linear analysis. Hence, according to Table 3, is seems that the Redlich–Peterson model is the most suitable model to satisfactorily describe the studied biosorption phenomenon. Indeed, the highest $R^2$ value and the lowest ARED, MSPED, HYBRID, RESID and SSE values were found when modelling the equilibrium data using the Redlich–Peterson, for both linear and non-linear regression analysis (cf. Table 3).

By the way, a brief comparison between linear and non-linear analysis was carried out to investigate, among these six error functions, the most suitable one(s) for each regression approach, taking the best-fitting model (i.e. Redlich–Peterson) as a study case. The results had shown that, for the linear analysis, the regression coefficient ($R^2$) is the highest, which makes it more adequate as error estimation tool to determine the best isotherm fit for linear regression. For non-linear analysis, the rest of the error functions (i.e. ARED, MSPED, HYBRID, RESID and SSE) seem to be the most appropriate. Such trend was previously proven by other researchers [18,24].

Therefore, based on these mentioned results (used as reference for the next section), the useful error estimation statistical tools should point out the Redlich–Peterson as best-fitting model

after both linear and non-linear analysis. In that context, it has to be stressed that since the Redlich–Peterson is a three-parameter model while Freundlich, Langmuir, Temkin and Elovich are two-parameter models, only the results obtained with the error functions which take into account different numbers of the model parameters (MSPED and HYBRID) seem more adequate and essentially meaningful.

### 3.3. Error estimation using alternative statistical tools

For the linear analysis approach, the results, given by Table 4, showed that both $\chi^2$ and Student's $T$-tests are suitable to adequately determine the best-fitting isotherm model. Indeed, among the five investigated tools, only those statistical tests were able to point out the Redlich–Peterson model as the most appropriate one to satisfactory fit the studied biosorption isotherm curves.

Moving to the case of non-linear regression analysis, except the Student's $T$-test, all the other statistical functions ($r$, $r^2$, $\chi^2$ test and $F$-test) were suitable to determine the most appropriate fitting-model (cf. Table 4), which is also the Redlich–Peterson. Such tendency (i.e. more statistical functions are valid for

Table 3
Isotherm error deviation data related to the biosorption of Solophenyl Brown textile dye onto raw *P. oceanica* fibres using six commonly used functions

| Error functions | $R^2$ | ARED | MPSED | HYBRID | SSE | RESID |
|---|---|---|---|---|---|---|
| Linear approach | | | | | | |
| Freundlich | 0.9971833 | 2.9900154 | 0.2825892 | 0.0998208 | 0.0210730 | 0.026 |
| Langmuir | 0.9764732 | 7.0764936 | 1.0105635 | 1.2765482 | 0.1620227 | 0.110 |
| Redlich–Peterson | **0.9980743** | **2.9567853** | **0.2666462** | **0.0888752** | **0.0199709** | **0.021** |
| Temkin | 0.9692536 | 8.7652330 | 1.4352287 | 1.5982735 | 0.2870381 | 0.178 |
| Elovich | 0.9642114 | 8.9187342 | 1.4704153 | 1.6610253 | 0.3026738 | 0.233 |
| Non-linear approach | | | | | | |
| Freundlich | 0.9937448 | 2.3327362 | 0.2748083 | 0.0943995 | 0.0190207 | 0.019 |
| Langmuir | 0.9745945 | 7.7474731 | 1.1050843 | 1.5265142 | 0.0939763 | 0.094 |
| Redlich–Peterson | **0.9948648** | **2.0826036** | **0.2558520** | **0.0818253** | **0.0185504** | **0.018** |
| Temkin | 0.9691891 | 8.2910673 | 1.2805516 | 1.6566301 | 0.1644019 | 0.114 |
| Elovich | 0.9594594 | 8.5513292 | 1.3871165 | 1.7201772 | 0.1742385 | 0.150 |

The highest $R^2$ and the lowest ARED, MPSED, HYBRID, SSE and RESID values are in bold.

Table 4
Isotherm error deviation estimation related to the biosorption of Solophenyl Brown textile dye onto raw *P. oceanica* fibres using alternative statistical tools

| Error functions | Pearson correlation coefficient ($r$) | Determination coefficient ($r^2$) | $\chi^2$ test | $F$-test | Student's $T$-test |
|---|---|---|---|---|---|
| Linear approach | | | | | |
| Freundlich | **0.997406857** | **0.994820439** | 0.99999959 | **0.99153374** | 0.97211901 |
| Langmuir | 0.985191583 | 0.970602455 | 0.99991061 | 0.94541803 | 0.86732529 |
| Redlich–Peterson | 0.997333718 | 0.994674544 | **0.99999967** | 0.98088104 | **0.99953293** |
| Temkin | 0.969172236 | 0.984465457 | 0.99992877 | 0.97244531 | 0.99759685 |
| Elovich | 0.941782248 | 0.970454660 | 0.99985131 | 0.95319407 | 0.98482737 |
| Non-linear approach | | | | | |
| Freundlich | 0.99742910 | 0.994864802 | 0.99999970 | 0.99132575 | **0.99988687** |
| Langmuir | 0.98913874 | 0.978395449 | 0.99991556 | 0.91985839 | 0.97979819 |
| Redlich–Peterson | **0.99749378** | **0.994993848** | **0.99999973** | **0.99322045** | 0.99695100 |
| Temkin | 0.981932135 | 0.964190718 | 0.99993503 | 0.96921681 | 0.99913016 |
| Elovich | 0.970456610 | 0.941782248 | 0.99985131 | 0.95319407 | 0.98482737 |

The highest $r$, $r^2$, $\chi^2$, $F$-test and Student's $T$-test values are in bold.

non-linear than linear analysis) could be explained by the difference between experimental and predicted equilibrium data using linear and non-linear methods. Indeed, the transformation of non-linear to linear expression distorts the experimental error, creating therefore an inherent error estimation problem which limits the validity of the studied tools. Moreover, the linear analysis method assumes the scatter of points around the line follows a Gaussian distribution and the error distribution is the same at every value of the equilibrium liquid-phase residual concentration (i.e. *X*-axis). Nevertheless, such behaviour is practically impossible with equilibrium isotherm models (since isotherm models had non-linear shape) as the error distribution gets altered after transforming the data to linear [23,24]. Unlikely, non-linear regression method would avoid such errors, making this analysing technique the most appropriate to obtain more realistic isotherm parameters.

## 4. Conclusion

In this research, equilibrium biosorption of direct textile dye by marine *P. oceanica* fibres has been carried out. The related results have been modelled using Freundlich, Langmuir, Redlich–Peterson, Temkin and Elovich equations, via both linear and non-linear regression analysis. The best-fitting model was, firstly, evaluated using six different error functions. The examination of all these error estimation methods showed that the Redlich-Peterson model provides the best fit for the experimental equilibrium data (i.e. highest $R^2$ and lowest ARED, MSPED, HYBRID, RESID and SSE values). In that context, it has to be mentioned that MSPED and HYBRID are the most suitable and meaningful tools to predict best-fitting equation when investigated models do not contain the same number of inner parameters.

Secondly, the applicability of five statistical tools to satisfactorily determine best-fitting isotherm model was investigated, for both linear and non-linear analysis. The related results showed that the $\chi^2$ and Student's $T$ tests could be useful, with $R^2$, in the case of linear analysis. On the other hand $r$, $r^2$, $\chi^2$ and $F$-test seem to be adequate to point out the best-fitting isotherm model after a non-linear regression approach.

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