Modelling the Effect of Sorbate-Sorbent Interphase on the Adsorption of Pesticides and Herbicides by Historical Data Design

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ABSTRACT

Statistical modelling was employed to analyze the effect of sorbate-sorbent interphase on the adsorption of pesticides and herbicides from aqueous media. The dataset used for this study was sourced from relevant and reputable published papers in the past five years. Sixty-six lines of data were analyzed using response surface methodology (RSM) and historical data design (HDD) on Design expert. Five parameters were considered in the study: adsorbate’s relative molecular mass (RMM), adsorbent specific surface area (SBET), adsorbent effective surface area $eSBET$ (i.e., the portion of the SBET occupied by the sorbate molecules), the water solubility of adsorbate, and adsorbate preferential adsorption (i.e., the ratio of the amount of sorbate on the sorbent to the amount in solution). From the analysis of variance, it was observed that the $SBET$ of the adsorbent was the most significant determinant for the adsorption capacity, $q$ (at a significance level of $p<0.05$). Other significant factors were the RMM, $eSBET$, and the preferential adsorption. Generally, solubility did not show any significant influence on the $q$. The response surface model had an $R^2$ value of 0.9945 and an adjusted $R^2$ value of 0.9927. Conclusively, the $q$ of an adsorbent towards an herbicide or a pesticide increases with increasing $eSBET$ and SBET, irrespective of the sorbate’s solubility and molecular mass.

INTRODUCTION

Pesticides and herbicides (P & H) are substances (or mixtures of various substances) used for the control of pests and herbs, respectively [1]. Their use gained attraction in the 1950s when the so-called ‘green revolution’ commenced [2]. However, the incessant population explosion, dynamics of lifestyle patterns, and technological advancement require the use of P & H in large quantities to cope with the huge demand for agricultural produce in modern times. Therefore, high levels of residual P & H are often found in the environment, which has rendered the chemical being regarded as emerging pollutants because their use has led to soil pollution [3, 4]. Further, P & H often leach from their point of primary application into nearby water bodies, thereby resulting in secondary water pollution and, sometimes, eutrophication [5]. Due to the high toxicity of P & H, these forms of pollution endanger human health via the food chain [6]. Especially, considering the importance of water to the livelihood and survival of man, the presence, quantitation, and eventual mitigation of P & H in our waters cannot be trivialized or ignored [7, 8].

Adsorption by activated carbon [9, 10], biosorbents [11], clay [12], biochar [13, 14], carbon nanotubes [15], and polymers [16, 17] is a proven excellent method in remediating water pollution in general and those of P & H in particular. Therefore, the sorbate-sorbent interphase must be adequately understood because it influences the $q$ of sorbents in aqueous media. Where $q$ is introduced as adsorption capacity.

Within the scope of the authors’ exhaustive search, so far, no study has investigated a historical dataset towards evaluating the effect of sorbate-sorbent interphase on the adsorption of P & H. Thereby, leaving open an interesting knowledge gap, which underlines the novelty of this study. Thence, response surface methodology and historical data design was utilized to investigate the effect of sorbate-sorbent interphase on the adsorption of P & H from aqueous media. The key adsorption properties considered were the adsorbent specific surface area

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(SBET), a derived effective surface area (eSBET) (i.e., the portion of the SBET occupied by the sorbate molecules), the water solubility of adsorbate, and adsorbate preferential adsorption (i.e., the ratio of the amount of sorbate on the sorbent to the amount in solution), and estimated preferential adsorption. The estimation of the eSBET is relevant because the preferential attachment of a sorptive onto a surface depends on the number of active sites with sufficient affinity for the sorptive, irrespective of the SBET. Since most studies reviewed here only reported the SBET without providing (any, adequate, or quantitated) information on the chemical functionalities on the sorbents, the derivation of eSBET came to the fore to inform on the relevance of the active sites on the surface to the q value. Thus, the eSBET value would increase linearly with the relevant chemical surface functionality tethered on the SBET, either naturally or engineered by chemical impregnation [18, 19]. The key index for evaluating the extent of sorbate-sorbent interphase is the mass adsorption capacity (q, mg/g) of the adsorbent for the specific pesticides or herbicides.

MATERIAL AND METHOD

Response surface methodology (RSM) and historical data design on Design Expert v10.0.1 (Stat Ease Inc., Minneapolis, USA) were used in this study. RSM has been discussed in detail elsewhere [20, 21]. Because the adsorption dataset used in this study was already available (i.e., does not require the pre-designing of experiments), historical data design was selected because of its flexibility. In other words, the adsorption dataset to be analysed was not pre-planned by a Design of experiments (DOE); therefore, it is suitable for the statistical modeling and analysis. This analytical approach affords the researcher the freedom of specifying the number of factors, the number of responses, and the number of lines of results. Historical data design has been previously used for the optimization of biodiesel production [22], machining condition [23], solvent extraction [24], photocatalytic degradation [25], and a host of other applications.

Description of dataset

The dataset used for this analysis was sourced from reliable and relevant open literature. Research papers specifically on herbicide or pesticide adsorption that were published within the past five years were considered. Due to the nature of the analysis, the minimum data specification required from each reviewed papered were those of q and SBET of the adsorbent. Although the amount of adsorbent and the volume of sample solution data were required, they are usually reported in adsorption papers. From the original dataset, 33 papers reported on all the four required information, leading to 66 lines of data for analysis. The base data used for current work is presented in the Supplementary material. Some of the adsorbate (P & H) represented in the dataset includes 2,4-D [26, 27], oxamyl [28], diuron [29], metribuzin [14], dicamba [16], tebuthiuron [17], lindane [30] and a host of others.

Data analysis

The adsorption dataset was entered into Design Expert v10.0.1 (Stat Ease Inc., Minneapolis, USA) under response surface methodology and historical data design for statistical modeling and analysis. Adequate care was taken to ensure that each string of factors corresponded with the assigned response. The designation of factors and response are summarized in Table 1. The molecular mass is the mass of a molecule of the adsorbate (in this case, the pesticide or herbicide), whereas the specific surface area refers to the total surface area of a unit mass of the adsorbent. Moreover, the effective surface area is the surface area of the adsorbent concerning the specific adsorbate. It is unique to each sorbate-sorbent system. The solubility is the chemical property of the adsorbate, which refers to its ability to dissolve in water (specifically for this case). The solubility values reported in the Supplementary material is for 25°C. The preferential adsorption is the adsorbate preference/affinity in getting adsorbed on the surface of the adsorbent, especially in light of other competing adsorbate. The data band shows the range of numeric values for each response and, by consequence, the range for which any model developed from the data is valid.

The design of a wastewater treatment system and equipment dimension that is appropriate to improve the performance of the existing of waste water treatment plant (WWTP) is based on maximum flow 150 m³/day. Secondary data from some literatures to calculate the size of the equipment dimension was also used.

RESULTS AND DISCUSSION

Determination of best fit model

First, the model of best fit for the input data was determined. The Design Expert software uses the sequential model sum of squares where the highest order polynomial whose additional terms are significant (and the model not aliased) is selected as the best fit. A model is said to be aliased when the estimate of an effect includes the influence of one or more other effects (and these are usually high order interactions). Furthermore, a lack-of-fit test is also performed to determine the non-aliased model with the most insignificant lack-of-fit. The lack-of-fit is an error that is observed when the analysis omits one or more critical factors or terms from the process model. The results of the sequential model sum of squares and the lack of fit test are shown in Tables 2 and 3, respectively. The models considered were the linear, two-factor interaction (2FI), quadratic, and cubic.
models. Based on the verdicts presented in Tables 2 and 3, the quadratic model was selected as the model of choice for further analysis.

**Analysis of variance (ANOVA) and model reduction**

The ANOVA was conducted to determine the statistical significance of the quadratic model and all its associated model terms (at p<0.05). From the ANOVA results (Table 4), it was observed that the model is statistically significant, and the lack-of-fit is insignificant. In addition, we observed that the \( S_{\text{BET}} \) of the adsorbent is the most significant, limiting factor for the \( q \) value. Other significant factors were the RMM, e\( S_{\text{BET}} \) and preferential adsorption values. Generally, the solubility of the adsorbate has no statistical significance on the response. In this case, statistical significance refers to an observable relationship between the response (\( q \)) and the factors over the range of data. The effect of each factor may be positive or negative. Such affirmation cannot be determined by ANOVA but by parametric investigations.

Based on the limiting factors, the derived model (whose ANOVA is presented in Table 4) is given in Equation (1). The range of validity for each factor is \( 100<x<500 \), \( 0<x<3000 \), \( 0<x<1 \), \( 0<x<20 \) and \( 0<x<50 \).

\[
q = -516.99 + 3.967A - 0.5529B - 1.26 \times 10^{4}C + 12650D + 17.62E + 2.554AB + 53.68AC - 64.64AD - 0.1146AE + 2.125 \times 10^{2}BC + 2.743BD + 1.37 \times 10^{-2}BE + 2.105 \times 10^{7}CD - 4156CE - 2519DE - 7.23 \times 10^{-3}A^2 - 1.399 \times 10^{-5}B^2 + 1.343 \times 10^{-7}C^2 - 296.6D^2 + 0.2674E^2
\]

(1)

Next in the modelling of the data was model reduction. Here, the insignificant terms in the model are sequentially removed whilst monitoring the ANOVA result and the model summary statistics (\( R^2 \), adjusted \( R^2 \)). This is done to improve the overall accuracy of the model. The first term removed from the model was the single solubility term ‘\( D \)’. Upon recalculation, all the remaining terms remained significant except BE, C\(^2\), and D\(^2\). Therefore, by expunging those 3 terms, the final model (Equation (2)) had all terms significant.

\[
q = -348.93 + 2.914A - 0.4809B - 5.396 \times 10^{4}C + 11.13E + 2.159 \times 10^{-3}AB + 2515AC - 1.338AD - 0.0571AE + 2.145 \times 10^{2}BC + 1.779BD - 1.55 \times 10^{5}CD - 1427CE + 416ADE - 5.92 \times 10^{-3}A^2 - 5.31 \times 10^{-6}B^2 + 0.0978E^2
\]

(2)

Note, the current model is a non-hierarchical polynomial regression model (and it excludes hierarchically inferior terms). Thus, it may be used only within the range of validity for each factor. This is because all analysis within the software is based on the coded equation. This means that these expressions cannot serve as a truly accurate predictor but can be used only by investigating the relationship between the factors and their respective responses.

**Model diagnostics**

The R-squared value is the variance of the experimental variables that is predictable by the correlation/model, and the closer the \( R^2 \) is to unity (1), the better the model [22].

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### Table 1. Designation of factors and response

| Designation          | Data                                     | Unit         | Data band       |
|----------------------|------------------------------------------|--------------|-----------------|
| Factor 1             | Relative molecular mass (RMM)            | g/mol        | 100 < x < 500   |
| Factor 2             | Specific surface area (\( S_{\text{BET}} \)) | m\(^2\)/g    | 0 < x < 3000    |
| Factor 3             | Effective surface area (e\( S_{\text{BET}} \)) | mol/m\(^2\) | 0 < x < 1      |
| Factor 4             | Solubility                               | mol/l        | 0 < x < 20      |
| Factor 5             | Preferential adsorption                  | Dimensionless | 0 < x < 50      |
| Response             | Mass adsorption capacity                 | mg/g         |                 |

### Table 2. Results of the sequential model sum of squares

| Source                | Sum of Squares | df  | Mean Square | F Value | p-value | Verdict   |
|-----------------------|----------------|-----|-------------|---------|---------|-----------|
| Mean vs Total         | 4.967e+6       | 1   | 4.967e+6    | 5.65    | 0.0002  |           |
| Linear vs Mean        | 2.171e+6       | 5   | 4.342e+5    | 5.65    | 0.0002  |           |
| 2FI vs Linear         | 4.513e+6       | 10  | 4.513e+5    | 233.00  | < 0.0001| Suggested |
| Quadratic vs 2FI      | 64592          | 5   | 12918       | 18.02   | < 0.0001| Suggested |
| Cubic vs Quadratic    | 32260          | 33  | 977.59      | 4.513e+5| 0.0002  | Aliased   |
| Residual              | 0.000          | 12  | 0.000       |         |         |           |
| Total                 | 1.175e+7       | 66  | 1.780e+5    |         |         |           |
TABLE 3. Results of the lack-of-fit test

| Source         | Sum of Squares | df  | Mean Square | F Value | p-value | Verdict      |
|----------------|----------------|-----|-------------|---------|---------|--------------|
| Linear         | 4.607e+6       | 58  | 79434       | 52.39   | 0.0189  |              |
| 2FI            | 93820          | 48  | 1954.6      | 1.29    | 0.5340  |              |
| Quadratic      | 29227          | 43  | 679.72      | 0.45    | 0.8803  | Suggested    |
| Cubic          | -3032.7        | 10  | -303.26     | -0.20   | 1.0000  | Aliased      |
| Pure Error     | 3032.7         | 2   | 1516.3      |         |         |              |

TABLE 4. Analysis of variance (ANOVA) for quadratic model (Partial sum of squares - Type III)

| Source                             | Sum of Squares | df  | Mean Square | F Value | p-value      | Verdict     |
|------------------------------------|----------------|-----|-------------|---------|--------------|-------------|
| Model                              | 6.749e+6       | 20  | 3.374e+5    | 470.70  | < 0.0001     | significant |
| A-Relative molecular mass          | 5000.8         | 1   | 5000.1      | 6.98    | 0.0113       |             |
| B-Specific surface area            | 2.937e+6       | 1   | 2.937e+6    | 4096.7  | < 0.0001     |             |
| C-Effective surface area           | 7644.0         | 1   | 7644.01     | 10.66   | 0.0021       |             |
| D-Solubility                       | 1323.6         | 1   | 1323.6      | 1.85    | 0.1810       |             |
| E- Preferential adsorption         | 4372.5         | 1   | 4372.5      | 6.10    | 0.0174       |             |
| AB                                 | 47104          | 1   | 47104       | 65.71   | < 0.0001     |             |
| AC                                 | 8585.5         | 1   | 8585.5      | 11.98   | 0.0012       |             |
| AD                                 | 31232          | 1   | 31232       | 43.57   | < 0.0001     |             |
| AE                                 | 16787          | 1   | 16787       | 23.42   | < 0.0001     |             |
| BC                                 | 2.933e+6       | 1   | 2.933e+6    | 4091.1  | < 0.0001     |             |
| BD                                 | 9453.8         | 1   | 9453.8      | 13.19   | 0.0007       |             |
| BE                                 | 717.11         | 1   | 717.11      | 1.00    | 0.3226       |             |
| CD                                 | 1307.8         | 1   | 1307.8      | 1.82    | 0.1836       |             |
| CE                                 | 11511          | 1   | 11511       | 16.06   | 0.0002       |             |
| DE                                 | 4773.7         | 1   | 4773.7      | 6.66    | 0.0132       |             |
| A<sup>2</sup>                      | 25401          | 1   | 25401       | 35.43   | < 0.0001     |             |
| B<sup>2</sup>                      | 2525.7         | 1   | 2525.7      | 3.52    | 0.0670       |             |
| C<sup>2</sup>                      | 796.97         | 1   | 796.97      | 1.11    | 0.2973       |             |
| D<sup>2</sup>                      | 783.25         | 1   | 783.25      | 1.09    | 0.3015       |             |
| E<sup>2</sup>                      | 12210          | 1   | 12210       | 17.03   | 0.0002       |             |
| Residual                           | 32260          | 45  | 716.90      |         |             |             |
| Lack of Fit                        | 29228          | 43  | 679.72      | 0.45    | 0.8803       | not significant |
| Pure Error                         | 3032.7         | 2   | 1516.3      |         |             |             |
| Cor. Total                         | 6.781e+6       | 65  |             |         |             |             |

The adjusted $R^2$ value is one that has been adjusted for the number of predictors in the correlation/model. The new model had an $R^2$ and adjusted $R^2$ values of 0.9945 and 0.9927, respectively. Such high reliability is impressive, considering that 66 lines of data (from nine different adsorbents against 32 different adsorbate) were used for the analysis. To achieve such high values for the coefficient of determination when a large pool of data is analysed is rare. This is an indicator of the accuracy of the model in predicting the statistical relevance of the factors. The parity between the model prediction and the actual values are shown in Figure 1. Obviously, most of the 66 data points fell on the parity diagonal with negligible few outliers, further suggestive of the accuracy of the model for the intended purpose.
Figure 1. Parity plot of model predictions against actual results

Figure 2. Effect of specific surface area and molecular mass on the mass adsorption capacity

Figure 3. Effect of solubility and effective surface area on the mass adsorption capacity

Figure 4. Effect of preferential adsorption and effective surface area on the mass adsorption capacity

**Parametric studies**

Besides, the evaluation of the statistical significance of the factors on the mass adsorption capacity, the other purpose of this paper is to conduct a parametric study. In parametric investigations, the relationship between the process parameters is evaluated in relation to the process response. Five parameters are considered in this study as factors affecting the sorbate-sorbent interactions for pesticides and herbicides. They are the adsorbate molecular mass, adsorbent specific surface area, adsorbent effective surface area, adsorbate solubility, and adsorbate preferential adsorption. The adsorbate solubility has already been shown to be a statistically insignificant factor but will be discussed also. The values on the y-axis of Figures 2-4 must be ignored as they are not the exact values because the model is a non-hierarchical polynomial regression model (and it excludes hierarchically inferior terms). The plots are chosen in such a way that all factors are discussed, and all key factor interactions are highlighted.

From Figure 2, it was observed that adsorbent having higher $S_{BET}$ would exhibit a superior $q$ value irrespective of the sorptive’s RMM. Although the chemistry of the sorbate-sorbent interface plays an important role in adsorption, the $S_{BET}$ remains the primary determining factor for P & H adsorption. The initial concentration of the adsorbate is another important determining factor identified in this study with respect to P & H control. Further, it was noticed that the $q$ increases with $eS_{BET}$ regardless of the sorptive’s solubility (Figure 3). This was expected because the $eS_{BET}$ is a function of the active sites on the $S_{BET}$ with an adequate affinity towards a specific adsorbate. Such specificity means that if the adsorbent has a quite $S_{BET}$, then more available active sites for adsorption of that specific adsorbate are expected. Therefore, other physical properties (such as pore volume and pore size distribution), and chemical properties (amount and affinity of surface chemical groups) of the adsorbent, as well as the kinetic diameter and orientation of the adsorbate often influence the $q$ values.

The effect of dimensionless preferential adsorption (Table 1) on the adsorption capacity was barely noticed, as depicted in Figure 4, as the case was for solubility (Figure 3). This was unsurprising as solubility was not statistically significant to the response surface model.
from the ANOVA. It must be emphasized that the observations from these discussions are specifically for P & H with RMM between 100 and 500 g/mol (Table 1). Thence, further inferences and wide generalizations outside this domain are not guaranteed.

CONCLUSIONS

Response surface methodology and historical data design were successfully used to investigate the effect of sorbate-sorbent interphase on the adsorption of pesticides and herbicides from aqueous media. Five parameters were considered in the study: adsorbate molecular mass, adsorbent specific surface area, adsorbent effective surface area, adsorbate solubility, and adsorbate preferential adsorption. Several important conclusions can be drawn from the study:

i. The results of the sequential model sum of squares and the lack of fit test revealed that the quadratic model is the best fit.

ii. From the analysis of variance (ANOVA), it was observed that the specific surface area of the adsorbent is the most significant factor affecting the adsorption capacity (at a significance level of $p<0.05$). Other significant factors were the molecular mass, effective surface area, and the preferential adsorption.

iii. The adsorbate’s water solubility does not have a significant effect on the adsorption capacity. The model was carefully reduced to eliminate all non-significant terms before diagnostics, and parametric investigations were conducted.

iv. The model had an $R^2$ value of 0.9945 and an adjusted $R^2$ value of 0.9927, which was quite impressive for 66 lines of data.

An adsorbent with a high specific surface area would exhibit high adsorption capacity irrespective of the relative molecular mass. The adsorption capacity of the adsorbent to the herbicide or pesticide increases at the higher effective surface area, and this holds for all domains of solubility. The effect of solubility and preferential adsorption on the adsorption capacity was barely noticed.

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چکیده
از مدل آماری برای تجزیه و تحلیل تأثیر اینترفاز جاذب مواد در جذب سموم و عفون‌کشی‌ها از محیط‌های آبی استفاده شد. جمعه داده‌های مورد استفاده برای این مطالعه از مقالات معتبر منتشر شده در پنج سال گذشته تهیه شدند. شصت و شش خط داده با استفاده از روش سطح پاسخ (RSM) و طراحی داده‌های مورد استفاده از روش سطح پاسخ (RSM) و طراحی داده‌های مورد استفاده از سطح خاص جاذب (SBET) مورد تجزیه و تحلیل قرار گرفت. پنج پارامتر در این مطالعه در نظر گرفته شد: توده مولکولی نسبی جاذب (q0), حلالیت (C0), رابطه جذب ترجیحی (α), و جذب ترجیحی (β). به طور حتمی، حلالیت تأثیر قابل توجهی در حقوق کشف نمی‌کند. عوامل مهم دیگر شامل مقدار SBET و مقدار مولکولینی یک کیلو گرم (M) می‌باشد.

کلمات کلیدی: بیشینه‌سازی، پاسخ‌گویی، فیزیولوژی، سموم، عفون‌کشی‌های آبی.