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Multiscale estimation of the Freundlich adsorption isotherm

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ABSTRACT: Adsorption plays an important role in water and wastewater treatment. The analysis and design of processes that involve adsorption rely on the availability of isotherms that describe these adsorption processes. Adsorption isotherms are usually estimated empirically from measurements of the adsorption process variables. Unfortunately, these measurements are usually contaminated with errors that degrade the accuracy of estimated isotherms. Therefore, these errors need to be filtered for improved isotherm estimation accuracy. Multiscale wavelet based filtering has been shown to be a powerful filtering tool. In this work, multiscale filtering is utilized to improve the estimation accuracy of the Freundlich adsorption isotherm in the presence of measurement noise in the data by developing a multiscale algorithm for the estimation of Freundlich isotherm parameters. The idea behind the algorithm is to use multiscale filtering to filter the data at different scales, use the filtered data from all scales to construct multiple isotherms and then select among all scales the isotherm that best represents the data based on a cross validation mean squares error criterion. The developed multiscale isotherm estimation algorithm is shown to outperform the conventional time-domain estimation method through a simulated example.

Keywords: *Freundlich isotherm; Multiscale representation; Noise filtering; Parameter estimation*

INTRODUCTION

Toxic heavy metal contamination of water and wastewater is a significant problem. The presence of these metals above the permissible limits in the environment causes severe public health problems (Baily *et al*., 1999; Naseem and Tahir, 2001). Therefore, remediation of such water from these pollutants is essential before it can be reused. Several water purification methods have been utilized, and include chemical precipitation (Henke, 1998), reverse osmosis (Ning, 2002), electro-dialysis, ion exchange and adsorption.

Adsorption is a mass transfer process, which involves the contact of solid (adsorbent) with a fluid containing certain pollutants (Reynolds and Richards, 1995; Alkan and Dogan, 2001; Chien and Shah, 2007; Igwe *et al*., 2008; Shah *et al*., 2009). As a result of the contact, the pollutant binds to the surface of the adsorbent. The adsorption capacity depends on several factors, such as the adsorbent type, its surface area, and its internal porous structure (Abdel-Ghani and Elchaghaby, 2007). Additionally, since the attachment of the pollutant can be physical or chemical, the chemical and physical structures and the electrical charge of the adsorbent can significantly influence the interactions with the adsorbates and thus the effectiveness of pollutant removal. Adsorption processes are characterized by their kinetic and equilibrium isotherms (Chen *et al*., 2010). The adsorption isotherms specify the equilibrium surface concentration of the adsorbate as a function of its bulk concentration. Several mathematical models have been proposed to describe the equilibrium isotherms of adsorption. Some of the most popular models include Langmuir, Freundlich, Redlich-Peterson and Sips. A summary of these isotherms is provided in (Montgomery, 1995). Even though most of these adsorption isotherms are derived based on some theoretical assumptions about the adsorption mechanism, they involve model parameters that need to be estimated from experimental measurements of the process variables (Abdel-Ghani *et al*., 2009). For example, the Freundlich isotherm, which is the focus of this paper, has the following form (Adamson, 1967; Geankoplis, 1993; Seader and Henley, 1998; McCabe *et al*., 2001).

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$$q_e = k_f C_e^{1/n} \quad (1)$$

Where, C_e is the equilibrium liquid phase concentration (mg/L), q_e is the equilibrium solid phase concentration (mg/g), k_f is the sorption capacity, and n is the sorption intensity. In the above Freundlich model, and are model parameters to be estimated using the initial concentration and measurements of the equilibrium concentration, .

Unfortunately, measurements of the equilibrium concentration are usually contaminated with measurement noise due to random errors, human errors, or malfunctioning sensors. The presence of such measurement noise, especially in large amounts, can degrade the accuracy of the estimated isotherm parameters, which in turn limits the ability of the isotherm to accurately predict the adsorption capacity of the process in which the isotherm is used. Therefore, such noise needs to be filtered for improved estimation of the isotherm parameters.

Noise removal from data is not a simple task since practical measurements are usually multiscale in nature, meaning that they contain features and noise occupying different locations in time and frequency (Bakshi, 1999). Filtering techniques, however, usually classify noise as high frequency features, and filter the data by removing features with frequency higher than a defined frequency threshold. Since multiscale data may contain correlated noise with low frequency as well as important features with high frequency, noise removal from such data becomes challenging. Thus, multiscale modeling techniques are needed in the estimation of adsorption isotherms to account for this multiscale nature of the data (Nounou and Bakshi, 2000).

Many investigators have used multiscale techniques to improve the accuracy of estimated empirical models. For example, Palavajjhala *et al.* (1996) showed how to use wavelet representation to design wavelet pre-filters for process modeling purposes. Bakshi (1999) has discussed some of the advantages of using multiscale representation in empirical modeling and Bakshi (1998) has enhanced the noise removal ability of the principal component analysis (PCA) model by constructing multiscale PCA models, which he also used in process monitoring. Also, Robertson *et al.* (1998); Nikolaou and Vuthandam (1998) and Nounou (2006) used multiscale representation to reduce collinearity and shrink the large variations in the finite impulse response (FIR) model parameters. Finally, Nounou and Nounou (2005); (2007) and Nounou *et al.* (2009) used wavelet-based representation to enhance the accuracy of the Takagi-Sugeno fuzzy model, the auto regressive with exogenous variable (ARX) model and the Langmuir adsorption isotherm.

The objective of this work is to develop a multiscale estimation algorithm that reduces the effect of measurement noise on the accuracy and prediction of estimated Freundlich isotherm.

MATERIALS AND METHODS

In this section, the problem statement, the theoretical background material and the proposed multiscale modeling methods of the Freundlich isotherm are presented.

Problem statement

Given the initial concentration data $\{C_0(1)\, C_0(2) \ldots C_0(n)\}$and measurements of and the equilibrium concentrations, $\{C_e(1)\; C_e(2) \ldots C_e(n)\}$, which are assumed to be contaminated with additive zero-mean Gaussian noise, i.e.,$C_{e} = \widetilde{C}_{e} + \varepsilon_{e}$, where $\varepsilon_e \sim N(0, s^2)$, it is desired to estimate the parameter, k_f and n, that satisfy the Freundlich isotherm

$$q_e(k) = k_f C_e^{1/n}(k), \quad k \in [1, n] \quad (2)$$

Note that the equilibrium uptake, q_e, is not measured and is calculated as follows (Abdulkarim and Abu Al-Rub, 2004):

$$q_e(k) = \frac{\left(C_o(k) - C_e(k)\right)V}{w} \quad (3)$$

Where, V is the volume of the solution and w is the mass of the adsorbent.

Freundlich model representation

The Freundlich isotherm shown in Eq. 2 is nonlinear and can be linearized as follows:

Defining: $\alpha_1(k) = \ln q_e(k)$, $\alpha_2(k) = \ln C_e(k)$, $a_1 = 1/n$, and $a_2 = \ln k_f$, the linearized isotherm shown in equation (4) can be written in matrix form as follows:

$$\ln q_e(k) = \ln k_f + (1/n) \ln C_e(k) \quad (4)$$

$$\underbrace{\begin{bmatrix} \alpha_1(1) \\ \alpha_1(2) \\ . \\ . \\ \alpha_1(n) \end{bmatrix}}_{Y} = \underbrace{\begin{bmatrix} \alpha_2(1) & 1 \\ \alpha_2(2) & 1 \\ . & . \\ . & . \\ \alpha_2(n) & 1 \end{bmatrix}}_{X} \underbrace{\begin{bmatrix} a_1 \\ a_2 \end{bmatrix}}_{a} \qquad (5)$$

which can be written more compactly as:

$Y=X_a$

Isotherm estimation using least squares regression

The linearized model parameter vector, a, can be estimated using ordinary least squares (OLS) regression by solving the following minimization problem (Frank and Friedman, 1993),

$$\{\hat{a}\} = \arg\min_{a} (Y - Xa)^T (Y - Xa) \qquad (6)$$

Which has the following closed form solution,

$$\hat{a} = \left(X^T X\right)^{-1} X^T Y \qquad (7)$$

Once the parameters a_1 and a_2 are estimated, the original isotherm parameters can be computed as follows:

$$\hat{n} = 1/\hat{a}_1 \text{ and } \hat{k}_f = e^{\hat{a}_2}$$

It can be seen from Eq. 6 that the OLS estimation method relies on minimizing the prediction error of the model output, Y, when estimating the isotherm parameters. This is because it is assumed that the input matrix, *X*, is noise-free. However, if the equilibrium concentration data are noisy, then the matrix, is also noisy, which violates the basic assumption of this approach. Therefore, in the presence of measurement noise in the data, this noise needs to be filtered for improved isotherm parameter estimation. One effective way to do that is through multiscale representation of data, which is presented next.

Multiscale data representation

A proper way of analyzing real data requires their representation at multiple scales. This can be achieved by expressing the data as a weighted sum of orthonormal basis functions, which are defined in both time and frequency, such as wavelets (Daubechies, 1988). Wavelets are a computationally efficient family of multiscale basis functions. A signal can be represented at multiple resolutions by decomposing the signal on a family of wavelets and scaling functions. The signals in Figs. 1b, d and f are at increasingly coarser scales compared to the original signal in Fig. 1a. These scaled signals are determined by projecting the original signal on a set of orthonormal scaling functions of the form,

$$\phi_{jk}(t) = \sqrt{2^{-j}}\,\phi\left(2^{-j}t - k\right) \qquad (8)$$

or equivalently by filtering the signal using a low pass filter of length *r*, $h = [h_1 \quad h_2 \quad . \quad . \quad h_r]$, derived from the scaling functions. On the other hand, the signals in Figs. 1c-g, which are called the detail signals, capture the differences between any scaled signal and the scaled signal at the finer scale. These detail signals are determined by projecting the signal on a set of wavelet basis functions of the form,

$$\psi_{jk}(t) = \sqrt{2^{-j}}\,\psi\left(2^{-j}t - k\right) \qquad (9)$$

or equivalently by filtering the scaled signal at the finer scale using a high pass filter of length *r*, $g = [g_1 \quad g_2 \quad . \quad . \quad g_r]$, derived from the wavelet basis functions. Therefore, the original signal can be represented as the sum of all detail signals at all scales and the scaled signal at the coarsest scale as follows,

$$x(t) = \sum_{k=1}^{n2^{-J}} a_{Jk}\phi_{Jk}(t) + \sum_{j=1}^{J}\sum_{k=1}^{n2^{-j}} d_{jk}\psi_{jk}(t) \qquad (10)$$

Where, *j*, *k*, *J* and *n* are the dilation parameter, translation parameter, maximum number of scales (or decomposition depth), and the length of the original signal, respectively (Daubechies, 1988; Strang, 1989).

Fast wavelet transform algorithms of *O(n)* complexity for a discrete signal of dyadic length have been developed (Mallat, 1989). For example, the wavelets and scaling functions coefficients at a particular scale (*j*), d_j and a_j, can be computed in a compact fashion by multiplying the scaling coefficient vector at the finer scale, a_{j-1}, by the matrices, G_j and H_j, respectively.

i.e. $a_j = H_j a_{j-1}$ and $d_j = G_j a_{j-1}$

$$\text{where } H_j = \begin{bmatrix} h_1 & . & h_r & 0 & 0 \\ 0 & h_1 & . & h_r & 0 \\ . & . & . & . & . \\ 0 & 0 & h_1 & . & h_r \end{bmatrix}_{n2^{-j} \times n2^{-j+1}} \quad \text{and} \qquad (11)$$

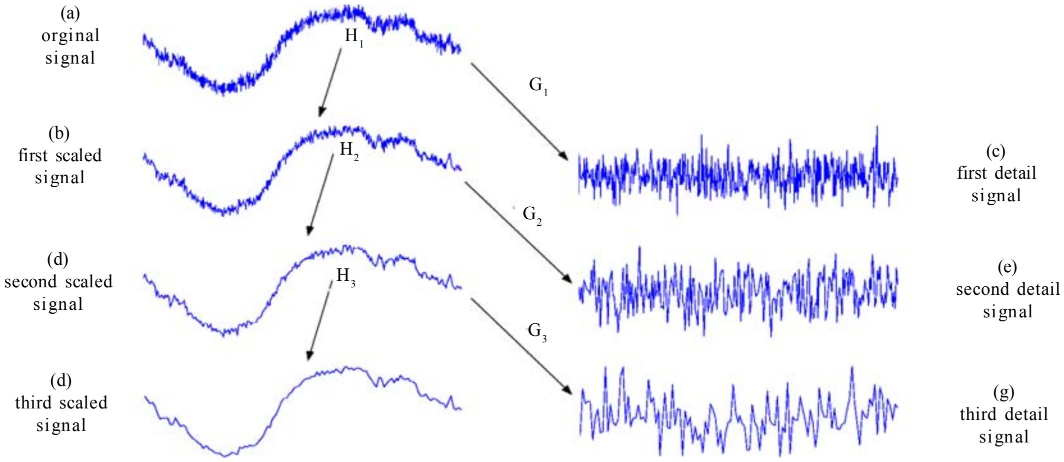

Fig. 1: A schematic diagram of data representation at multiple scales

$$G_j = \begin{bmatrix} g_1 & \cdot & g_r & 0 & 0 \\ 0 & g_1 & \cdot & g_r & 0 \\ \cdot & \cdot & \cdot & \cdot & \cdot \\ 0 & 0 & g_1 & \cdot & g_r \end{bmatrix}_{n2^{-j} \times n2^{-j+1}}$$

Note that the length of the scaling and detail signals decreases dyadically at coarser resolutions (higher *j*). In other words, the length of scaled signal at scale (*j*), is half the length of scaled signal at the finer scale, (*j*-1). This is due to down-sampling, which is used in discrete wavelet transform. Just as an example to illustrate the multiscale decomposition procedure and to introduce some terminology, consider the following discrete signal, Y_o, of length (*n*) in the time domain (i.e., $j = 0$),

$$Y_o = \left[y_o(1) \quad y_o(2) \quad . \quad y_o(k) \quad . \quad y_o(n) \right]^T \tag{12}$$

the scaled signal approximation of Y_o at scale (j), which can be written as:

$$Y_j = \left[y_j(1) \quad . \quad y_j(k) \quad . \quad y_j\left(n2^{-j}\right) \right]^T \tag{13}$$

can be computed as follows:

$$Y_j = H_j Y_{j\text{-}1} = H_j H_{j\text{-}1} \ldots H_1 Y_o \tag{14}$$

Note that this decomposition algorithm is batch, i.e., it requires the availability of the entire data set beforehand. An on-line wavelet decomposition algorithm has also been developed and used in data filtering (Nounou and Bakshi, 1999).

Multiscale data filtering

Multiscale filtering using wavelets is based on the observation that random errors in a signal are present over all wavelet coefficients while deterministic changes get captured in a small number of relatively large coefficients (Cohen *et al.*, 1993; Donoho and Johnstone, 1994; Donoho *et al.*, 1995; Bakshi, 1999; Nounou and Bakshi, 2000). Thus, stationary Gaussian noise may be removed by a three step method (Donoho *et al.*, 1995):

1) Transform the noisy signal into the time-frequency domain by decomposing the signal on a selected set of orthonormal wavelet basis functions;
2) Threshold the wavelet coefficients by suppressing coefficients smaller than a selected value;
3) Transform the thresholded coefficients back into the original domain.

Donoho and coworkers have studied the statistical properties of wavelet thresholding and have shown that for a noisy signal of length n, the filtered signal will have an error within $O(\log n)$ of the error between the noise-free signal and the signal filtered with a priori knowledge about the smoothness of the underlying signal (Donoho and Johnstone, 1994).

Selecting the proper value of the threshold is a critical step in the rectification process, and several methods have been devised. For good visual quality of the filtered signal, the Visushrink method determines the threshold as:

$$t_j = \sigma_j \sqrt{2 \log n} \tag{15}$$

Where, n is the signal length and σ_j is the standard deviation of the errors at scale j, which can be estimated from the wavelet coefficients at that scale by

$$\sigma_j = \frac{1}{0.6745} median\left\{\left|d_{jk}\right|\right\} \tag{16}$$

Other methods for determining the threshold are described in (Nason, 1996). The wavelet coefficients may be thresholded by hard or soft thresholding. Hard thresholding eliminates coefficients smaller than a threshold, whereas soft thresholding also shrinks the larger coefficients towards zero by the value of the threshold. Hard thresholding can lead to better reproduction of peak heights and discontinuities, but at the price of occasional artifacts that can roughen the appearance of the filtered signal, while soft thresholding usually gives better visual filtering quality and fewer artifacts (Coifman and Donoho, 1995). In this work, soft thresholding will be used in filtering adsorption data.

Multiscale formulation of Freundlich estimation problem

The main objective here is to reduce the effect of measurement noise in the data on the estimation of the Freundlich isotherm parameters using multiscale filtering. Therefore, the idea is to filter the measured equilibrium concentration data using different decomposition depths, estimate multiple isotherms using the filtered data from these scales, and finally select among all estimated isotherms the one that provides the best prediction.

Denoting the filtered equilibrium concentration data at scale depth (j) as $C_{e,j}(k)$, $k \in [1,n]$, the isotherm obtained using the filtered data at decomposition scale (j) can be expressed as:

$$q_{e,j}(k) = k_{f,j} C_{e,j}^{1/n_j}(k) \tag{17}$$

Where, the equilibrium uptake can be computed using the equilibrium concentration and the initial concentration as follows:

$$q_{e,j}(k) = \frac{\left(C_o(k) - C_{e,j}(k)\right)V}{w} \tag{18}$$

Multiscale Freundlich isotherm estimation

The linearized form of the isotherm shown in Eq. 17 at scale (j) can be written as follows:

$$\ln q_{e,j}(k) = \ln k_{f,j} + \left(1/n_j\right) \ln C_{e,j}(k) \tag{19}$$

Defining: $\alpha_{1,j}(k) = \ln q_{e,j}(k)$, $\alpha_{2,j}(k) = \ln C_{e,j}(k)$, $a_{1,j} = 1/n_j$, and $a_{2,j} = \ln k_{f,j}$, equation (19) can be expressed in matrix form as:

$$\underbrace{\begin{bmatrix} \alpha_{1,j}(1) \\ \alpha_{1,j}(2) \\ . \\ . \\ \alpha_{1,j}(n) \end{bmatrix}}_{Y_j} = \underbrace{\begin{bmatrix} \alpha_{2,j}(1) & 1 \\ \alpha_{2,j}(2) & 1 \\ . & . \\ . & . \\ \alpha_{2,j}(n) & 1 \end{bmatrix}}_{X_j} \underbrace{\begin{bmatrix} a_{1,j} \\ a_{2,j} \end{bmatrix}}_{a_j} \tag{20}$$

Which can be written more compactly as follows:

$$Y_j = X_j a_j \tag{21}$$

The linearized isotherm parameter vector at scale (j), a_j, can be estimated using least squares regression as follows:

$$\hat{a}_j = \left(X_j^T X_j\right)^{-1} X_j^T Y_j \tag{22}$$

Once the parameters $a_{1,j}$ and $a_{2,j}$ are estimated, the original isotherm parameters can be computed as follows:

$$\hat{n}_j = 1/\hat{a}_{1,j} \tag{23}$$

and,

$$\hat{k}_{f,j} = e^{\hat{a}_{2,j}} \tag{24}$$

Multiscale Freundlich isotherm estimation algorithm

The multiscale Freundlich isotherm estimation algorithm can be outlined as follows:

(1) Filter the measured equilibrium concentration data, C_e, using different decomposition depths (j), $j \in [1, J]$

(2) Using the filtered data from each decomposition depth;

(a) Compute the adsorption uptake using the filtered equilibrium concentration data using Eq. 18;

(b) Construct a linearized model of the adsorption isotherm as shown in Eq. 21;

(c) Estimate the linearized model parameters using least squares regression as shown in Eq. 22;

(d) Compute the original model parameters using the estimated linearized model parameters using Eqs. (23) and (24);

(e) Compute the cross validation mean squares error as follows (Nason, 1996).

$$CVMSE(j) = \frac{1}{n}\sum_{k=1}^{n}\left[\left(C_e^*(k) - \hat{C}_e(k)\right)^2\right]$$

where,

$$C_e^*(k) = \frac{1}{2}\left[C_e(k-1) + C_e(k+1)\right] \qquad (25)$$

(3) Select among all scales, the adsorption model with the minimum *CVMSE* as the optimum isotherm.

RESULTS AND DISCUSSION

In this section, the performance of the multiscale Freundlich isotherm estimation algorithm described earlier is illustrated through a simulated example, where $k_f = 15$ and $n = 5$. Eq. 1 is used to generate data, which are assumed to be noise-free. Then, the equilibrium concentration data are contaminated with zero mean Gaussian noise. Two different levels of noise are used ($\sigma^2 = 10$, 30, and 50), which correspond to C_e approximate signal-to-noise ratios of 127, 56, and 25, respectively. In this example, the “Daubechies-2” wavelet and scaling function filters are used in filtering. To make statistically valid conclusions about the performances of the time domain and multiscale estimation methods, a Monte Carlo simulation of 500 realizations is performed and the results are presented in Tables 1 and 2.

Table 1 compares the estimated parameters and uptake prediction mean squares errors obtained using the time domain and the multiscale estimation algorithms. These mean squares errors are computed with respect to the noise-free values. Table 1 shows that there is a clear advantage of the multiscale algorithm over the time domain method for both the parameters and uptake prediction. Table 2, on the other hand, presents the estimation mean squares errors at multiple scales also for the Freundlich isotherm parameters and predicted uptake. Table 2 shows that there is an improvement in the estimation accuracy at coarser scales up to a certain scale after which the estimation quality deteriorates. That’s why it is

Table 1: Parameter and q_e prediction mean square errors obtained using the time-domain and multiscale estimation methods

Estimation method	$\sigma^2 = 10$			$\sigma^2 = 30$			$\sigma^2 = 50$		
	k_f	n	q_e	k_f	n	q_e	k_f	n	q_e
Time-Domain	1.14	0.232	0.537	5.51	1.249	1.610	12.57	3.272	2.643
multiscale	0.62	0.093	0.175	1.39	0.215	0.485	2.38	0.363	0.740

Table 2: Parameters and q_e prediction mean square errors at multiple scales (numbers in parenthesis indicate the percentage each scale is selected as optimum)

Scale	$\sigma^2 = 10$			$\sigma^2 = 30$			$\sigma^2 = 50$		
	k_f	n	q_e	k_f	n	q_e	k_f	n	q_e
0	1.14	0.232	0.537 (0)	5.51	1.249	1.610 (0)	12.57	3.272	2.643 (0)
1	0.63	0.099	0.248 (3)	1.59	0.259	0.738 (6.2)	2.95	0.493	1.123 (9.2)
2	0.62	0.092	0.168 (97)	1.36	0.211	0.466 (93.6)	2.32	0.354	0.697 (90)
3	0.74	0.110	0.159 (0)	1.46	0.219	0.383 (0.2)	2.58	0.374	0.558 (0.8)
4	1.11	0.153	0.295 (0)	2.06	0.326	0.523 (0)	3.60	0.538	0.723 (0)

important to make a good selection of the estimation scale. Table 2 also lists in parenthesis the percentage each scale is selected as optimum using the cross validation error criterion shown in Eq. 25. These numbers indicate that reasonable approximation of the optimum scale is achieved by the *CVMSE* criterion.

The improvements in parameters estimation and uptake predictions are also illustrated in Figs. 2 and 3 for the case where $\sigma^2 = 30$. Fig. 2 shows histograms of the estimated parameters at different scales and Fig. 3 shows the predictions of the adsorption uptake at different scales. Figs. 2 and 3 show the advantages of multiscale estimation of the Freundlich isotherm, and the reason behind this improvement is the noise removal abilities of multiscale filtering, which is illustrated in Fig. 4 also for the case where $\sigma^2 = 30$.

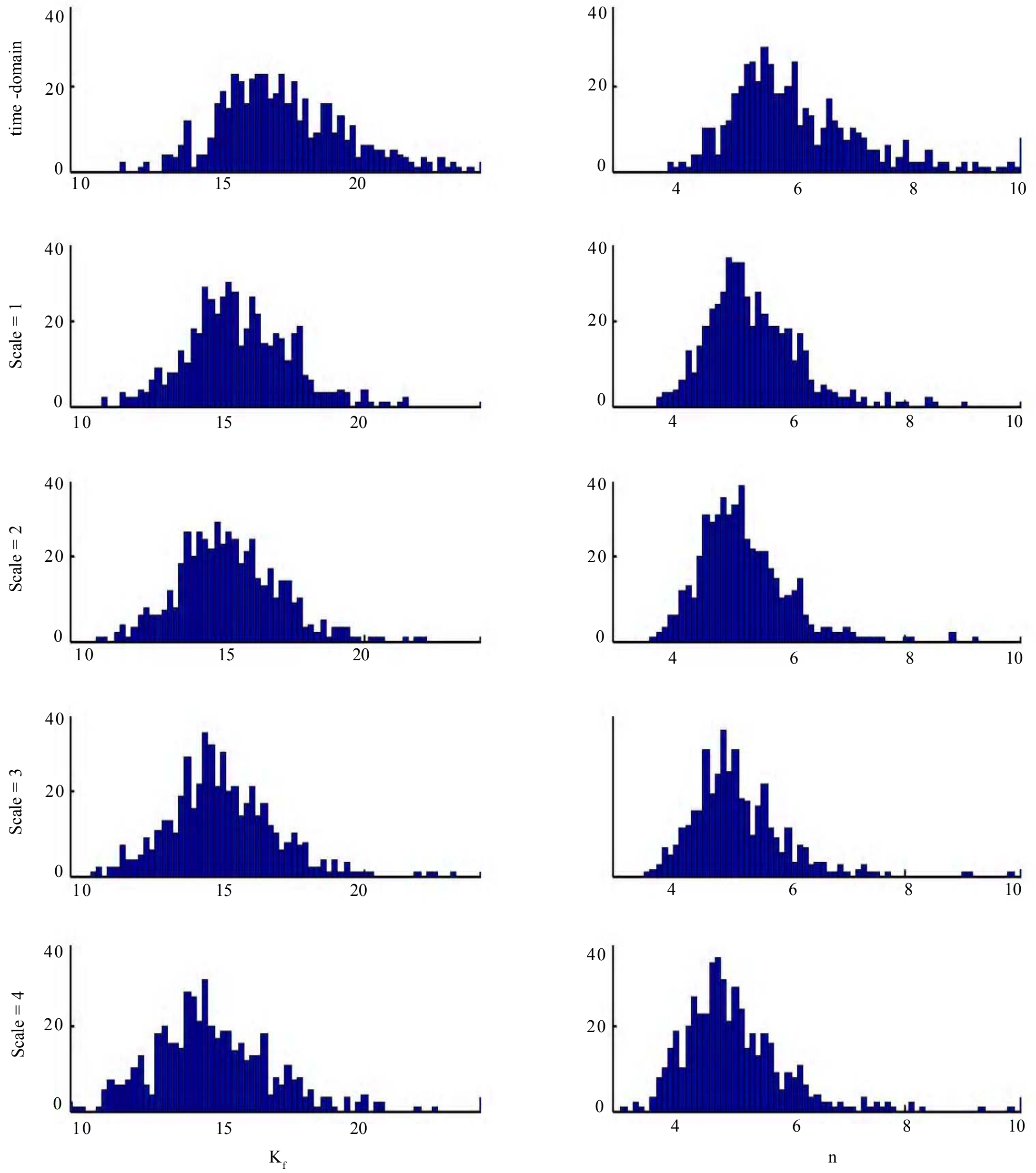

Fig. 2: Comparison between the estimated isotherm parameters at multiple scales for the case where $\sigma^2 = 30$

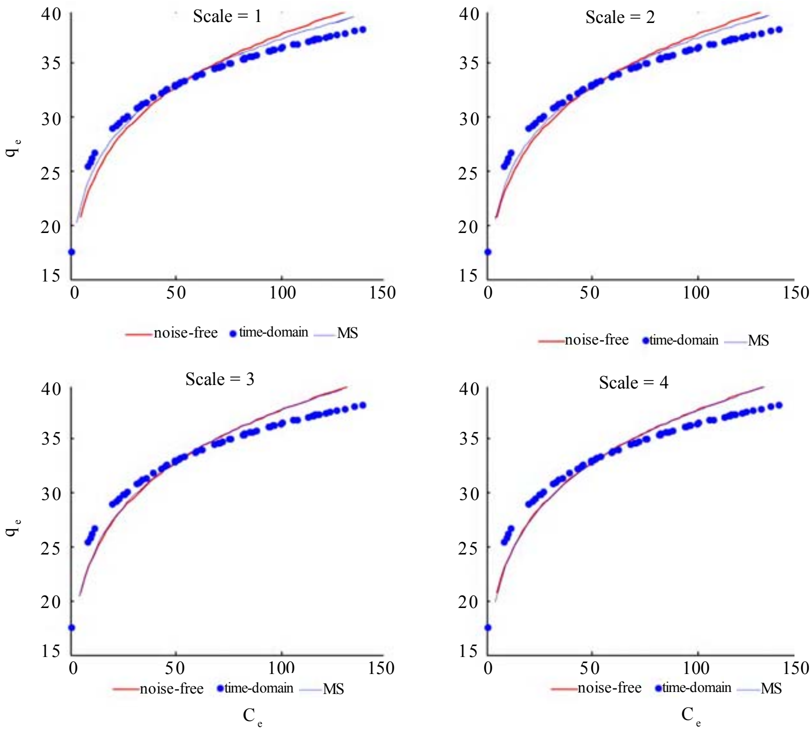

Fig. 3: Comparison between the uptake prediction at multiscale and its prediction in the time-domain for the case where $\sigma^2 = 30$

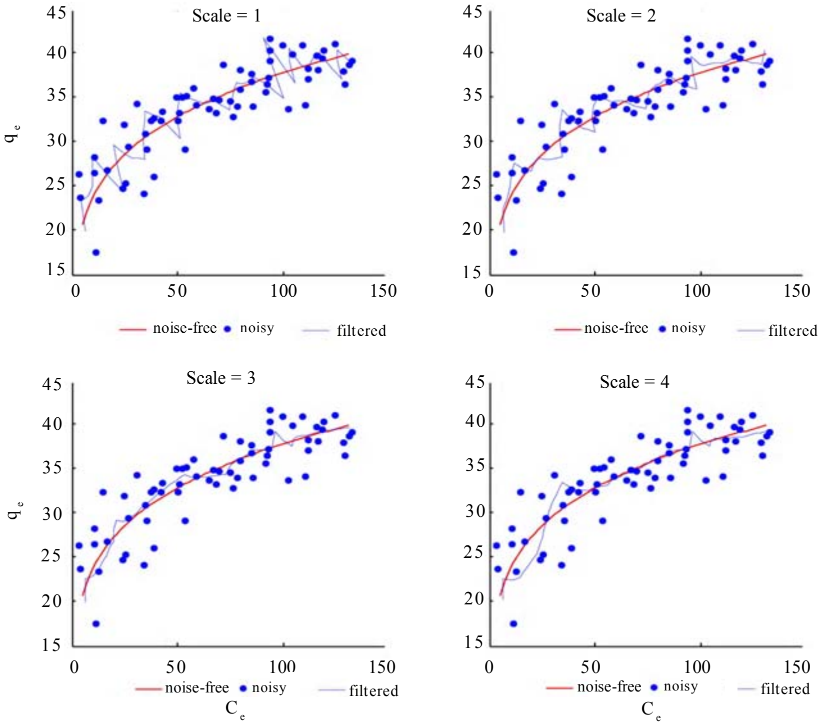

Fig. 4: Multiscale filtering of adsorption data at different scales for the case where $\sigma^2 = 30$

CONCLUSION

In this research, a new multiscale algorithm is developed to improve the estimation and prediction accuracies of the Freundlich adsorption isotherm from noisy measurements. The algorithm relies on denoising the data using multiscale filtering at different decomposition depths, constructing multiple isotherms at all scales, and then selecting among all scales the optimum Freundlich isotherm based on a cross validation error criterion. The performance of the developed multiscale estimation algorithm is illustrated through a simulated example that shows its advantages over the time-domain estimation method.

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