Description of sigmoid adsorption isotherms of water pollutants by the Aranovich–Donohue equation

Murat Yilmaz\textsuperscript{a}, Mohd Ali Hashim\textsuperscript{b}, and Khim Hoong Chu\textsuperscript{b, c}

\textsuperscript{a}Department of Chemical Engineering, Faculty of Engineering, Osmaniye Korkut Ata University, Osmaniye, Turkey; \textsuperscript{b}Department of Chemical Engineering, Faculty of Engineering, University of Malaya, Kuala Lumpur, Malaysia; \textsuperscript{c}R&D Department, Honeychem Research, Newtown, Wellington, New Zealand

ABSTRACT
In the field of adsorptive water remediation, the Brunauer–Emmett–Teller (BET) equation is often used to describe sigmoid type II isotherm data. A standard type II isotherm is a composite curve that comprises a type I and a type III curve at low and high concentrations, respectively. The BET equation uses a Langmuir-like function to describe the type I curve and a unique function to represent the type III curve. However, the ability of this mathematical combination to fit atypical type II isotherms is somewhat limited. The BET equation has been modified in various ways to improve its data fitting ability. The Aranovich–Donohue equation is an interesting modification in that it has a flexible equation structure that allows users to choose an isotherm model to describe the type I curve at low concentrations. It is shown here that the Aranovich–Donohue–Sips equation featuring the Sips equation is capable of accurately correlating anomalous acid red and acid black dye isotherm data, outperforming the original BET equation. Furthermore, the Aranovich–Donohue–Sips equation can accurately track a phenol isotherm characterized by a type V curve at low concentrations, demonstrating its versatility in fitting nonstandard type II isotherm data.

KEYWORDS
Aranovich–Donohue; Brunauer–Emmett–Teller (BET); multilayer adsorption; type II isotherm; type V isotherm; sigmoid

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INTRODUCTION
Hazardous dyes are frequently found in industrial wastewater streams. The dye pollution problem has stimulated a great deal of research on treatment methods, with adsorption being one of the most heavily investigated technologies. Many synthetic and natural materials have been tested for their ability to remove different classes of dyes from aqueous solutions. The effectiveness of a dye adsorption process is assessed on the basis of experimental data, typically in the form of adsorption equilibrium and kinetics, obtained from small-scale experiments. The measured data are summarized in a concise manner using mathematical models.

Adsorption equilibrium data are usually correlated using empirical or theoretical isotherm equations. The great majority of equilibrium data observed in dye adsorption studies manifest a monotonous approach to an adsorption maximum, designated as type I in the IUPAC classification. These hyperbolic type I curves can be correlated using simple isotherm models, such as the equations of Freundlich and Langmuir. However, an increasing number of studies have reported type II isotherms, which exhibit an inverse sigmoid curve shape. A typical type II isotherm is made up of two parts: a type I curve in the low concentration range and a type III curve at high concentrations. Standard type II isotherms can be modeled using the celebrated equation of Brunauer et al.\textsuperscript{11} commonly known as the BET equation in the gas adsorption literature. More than 20 years ago, Wang et al.\textsuperscript{2} reformulated the BET equation for aqueous phase adsorption and used it to correlate the type II isotherms observed in the case of dye adsorption by sewage sludge particles. The reformulated BET equation was subsequently used by Weng and coworkers to model the type II isotherms for the adsorption of new coccine acid red dye by digested sludge\textsuperscript{3} and sewage sludge ash.\textsuperscript{4} Further studies revealed that methylene blue adsorption by sludge ash and spent activated clay also displayed type II isotherms.\textsuperscript{5,6}

Many subsequent studies on the adsorption of a diverse array of dyes by solid materials have reported type II isotherms, including methylene blue,\textsuperscript{7–9} methyl blue,\textsuperscript{10} reactive blue 5 G,\textsuperscript{11–13} neutral red,\textsuperscript{14} methyl red,\textsuperscript{15} direct red 23,\textsuperscript{16} basic red 46,\textsuperscript{17} basic red 9,\textsuperscript{18} acid red 357,\textsuperscript{19} reactive red BF-4B,\textsuperscript{20} reactive drimarren red,\textsuperscript{21} acid black 210,\textsuperscript{19,22,23} reactive black 5,\textsuperscript{24} crystal violet,\textsuperscript{7} acid orange 7,\textsuperscript{10} acid green 16,\textsuperscript{24} mallow green,\textsuperscript{25} and eosin yellowish.\textsuperscript{26} A macroscopic
type II isotherm is indicative of multilayer adsorption. Indeed, spectroscopic and microscopic studies have provided convincing evidence for the spontaneous formation of dye multilayers at a liquid-solid interface under equilibrium conditions.\[27\]

Most of the aforementioned dye isotherms have been analyzed using the BET equation. Because of its limited data fitting ability, the BET equation may not be the best model for fitting type II isotherms with intricate curve shapes. A few of the dye isotherms noted above have been correlated using modified versions of the BET equation.\[11,12,19,20\] Among the several modified BET equations, a version proposed by Aranovich and Donohue\[28\] is unique in that it has a flexible equation structure. The Aranovich–Donohue equation admits an unspecified isotherm model, which is used to describe the type I curve at low concentrations. For example, the Aranovich–Donohue equation incorporating the Langmuir equation has recently been shown to be very effective in fitting several adsorption isotherms of water contaminants.\[29\] However, little is known regarding the data fitting ability of other versions of the Aranovich–Donohue equation. To address this research gap, in this paper we evaluate the ability of the Aranovich–Donohue equation featuring the Sips equation to describe type II dye isotherms, making use of literature data. Because the Sips equation can describe sigmoid type V curves, the Aranovich–Donohue–Sips equation is also tested against a phenol isotherm characterized by a type V curve at low concentrations and a type III curve at high concentrations. In the cases examined here, comparisons are made between four isotherm models: the Aranovich–Donohue–Sips equation, the Aranovich–Donohue–Langmuir equation, the original BET equation, and a modified BET equation.

**Isotherm models**

**The BET equation**

The original BET equation has been reformulated by Wang et al.\[21\] for aqueous adsorption systems, given here by Eq. 1, where \( q \) is the adsorbed phase concentration, \( q_m \) is the monolayer adsorption capacity, \( c \) is the liquid phase concentration, and \( b_B \) and \( k_B \) are equilibrium constants. There are three fitting parameters in the BET equation, \( q_m, b_B, \) and \( k_B \), which are adjusted to correlate the relationship between \( q \) and \( c \). Mathematically, Eq. 1 is analogous to the Guggenheim–Anderson–de Boer (GAB) equation for gas phase adsorption.\[30\]

\[
q = \frac{q_mB_C}{1 + (b_B - k_B)c(1 - k_Bc)} \quad (1)
\]

A different reformulated BET equation, frequently used in liquid phase adsorption research,\[31,32\] is given by Eq. 2, where \( c_i \) is the saturation concentration and \( \alpha \) is a dimensionless equilibrium constant. A drawback of Eq. 2 is that it cannot be simplified to the Langmuir isotherm if monolayer adsorption is assumed. In contrast, Eq. 1 reduces to the Langmuir isotherm when \( k_B = 0 \).

\[
q = \frac{q_mC}{1 + (\alpha - 1)(c/c_i)(c_i - c)} \quad (2)
\]

Another reformulated BET equation, given here by Eq. 3, has been used by Yoshida et al.\[33\] to describe the adsorption of an acid dye by cross-linked chitosan fibers. In Eq. (3), \( c_0 \) denotes the initial solute concentration and \( \beta \) is a dimensionless equilibrium constant. However, this formulation is debatable because the value of \( c_0 \) depends simply on the more or less arbitrary choice of the experimenter.

\[
q = \frac{q_mB_C}{1 + (\beta - 1)(c/c_0)(c_0 - c)} \quad (3)
\]

**The Aranovich–Donohue equation**

The original BET equation has been modified by Aranovich and Donohue\[28\] to improve its ability to describe gas adsorption on solid surfaces. In a subsequent paper, they extended the modified model to liquid phase adsorption.\[34\] The form of the Aranovich–Donohue equation for liquid phase adsorption is given by Eq. 4, where \( f(c) \) is a type I isotherm model chosen by users to describe the adsorption of the first molecular layer at low values of \( c \), and \( k_A \) and \( n_A \) are fitting parameters.

\[
q = \left( \frac{f(c)}{1 - k_A c} \right)^{n_A} \quad (4)
\]

The most commonly used type I isotherm equation for \( f(c) \) is the Langmuir equation, as shown in Eq. 5, where \( b_L \) is a constant. In addition to the Langmuir equation, the Sips equation is used for \( f(c) \) in the present study. The resulting equation is given by Eq. 6, where \( b_S \) and \( n_S \) are fitting parameters. The Langmuir version of the Aranovich–Donohue equation, Eq. 5, has four fitting parameters \( (q_m, b_L, k_A, \) and \( n_A) \) and is referred to as the ADL equation, while the Sips version of the Aranovich–Donohue equation, Eq. 6, has five fitting parameters \( (q_m, b_S, n_S, k_A, \) and \( n_A) \) and is called the ADS equation.
\[ q = \frac{q_m b L c}{(1 + b L c)(1 - k A c)^{n_k}} \]  
(5)

\[ q = \frac{q_m (b S c)^{n_k}}{[1 + (b S c)^{n_k}](1 - k A c)^{n_k}} \]  
(6)

**Model fitting and evaluation**

The adjustable parameters of the isotherm models studied in this work were estimated using a nonlinear least-squares fitting procedure. To assess the goodness of fit, the mean absolute percentage error (MAPE) defined by Eq. 7 was calculated for each fit, where \( E_i \) is the \( i \)th experimental value of \( q \), \( M_i \) is the \( i \)th value of \( q \) calculated from the chosen isotherm model, and \( w \) is the number of data points.

\[ \text{MAPE} = \frac{100}{w} \sum_{i=1}^{w} \left| E_i - M_i \right| E_i \]  
(7)

**Results and discussion**

The Aranovich–Donohue equation is evaluated against the isotherm data obtained for the adsorption of acid red 357 and acid black 210 by an activated carbon adsorbent.\(^{19}\) The activated carbon used in the dye adsorption experiments was prepared from chromium-tanned leather shaving wastes. It had a surface area of 800.4 m\(^2\) g\(^{-1}\) and an average pore diameter of 1.27 nm. The batch equilibrium experiments were conducted using a fixed initial dye concentration of 800 mg L\(^{-1}\), a fixed solution volume of 0.1 L, and varying amounts of activated carbon ranging from 0.05 to 7 g L\(^{-1}\). A contact time of 72 h was used for the batch adsorption experiments.

Figure 1 shows the acid red isotherm as reported by Manera et al.\(^{19}\) The dye isotherm exhibits a somewhat intricate type II curve shape. At low solution concentrations, it is represented by a very steep type I curve. The acid red isotherm then displays a rather long plateau, followed by a type III curve, which rises sharply with increasing \( c \). The type II dye isotherm can be viewed as a composite isotherm: a type I curve that transitions to a type III curve at an inflection point.

To demonstrate the limiting modeling power of the BET equation, Eq. 1 was fit to the acid red isotherm. As can be seen in Fig. 1A, the BET fit deviates significantly from the acid red isotherm, failing to track its overall shape. The fit of the acid red isotherm by the ADL equation is also shown in Fig. 1A. It is evident that the ADL fit manifests good agreement with the dye isotherm, tracking closely the entire isotherm shape. Figure 1B shows the fit of the ADS equation, which is very similar to the ADL fit depicted in Fig. 1A. The two forms of the Aranovich–Donohue equation, ADL and ADS, are superior to the BET equation in correlating the atypical shape of the acid red isotherm.

Manera et al.\(^{19}\) applied a modified BET equation to the acid red isotherm, given here by Eq. 8, where \( q_m, n_K, c_1, \) and \( c_2 \) are fitting parameters. Equation 8 is referred to as the Khalfouli equation since it was first reported in the work of Khalfouli et al.\(^{35}\) The data fitting procedure of Manera et al.\(^{19}\) returned the following parameter values: \( q_m = 39.3 \text{ mg g}^{-1}, n_K = 5.2, c_1 = 2.5 \text{ mg L}^{-1}, \) and \( c_2 = 824.4 \text{ mg L}^{-1}. \) Here, a nonlinear regression procedure was used to fit the Khalfouli equation to the acid red isotherm, returning \( q_m = 39.9 \text{ mg g}^{-1}, n_K = 5.1, c_1 = 2.5 \text{ mg L}^{-1}, \) and \( c_2 = 828.4 \text{ mg L}^{-1}. \) Our parameter estimates are comparable to those reported by Manera et al.\(^{19}\) Fig. 1B shows that the Khalfouli fit appears to be marginally inferior to the ADS fit.

\[ q = \frac{n_K q_m}{[(c_1/c)^{n_k} - (c_1/c_2)^{n_k} + 1](1 - (c/c_2)^{n_k})} \]  
(8)

The acid black isotherm reported by Manera et al.\(^{19}\) is presented in Fig. 2. The overall shape of the acid black isotherm is analogous to that of the acid red isotherm. At
low solution concentrations, the acid black isotherm exhibits a highly favorable type I curve. The plateau is quite long and located at a high level of \( q \sim 500 \text{ mg g}^{-1} \). The plateau of the acid red isotherm depicted in Fig. 1 is located at \( q \sim 200 \text{ mg g}^{-1} \). In contrast to the acid red isotherm, the type III curve of the acid black isotherm at high concentrations displays a more gradual rise in \( q \) with increasing \( c \).

Figure 2A presents the BET fit of the acid black isotherm, which is in good agreement with the type I curve of the isotherm at low concentrations, but cannot capture the gradual curvature of the type III curve defined by the last three data points. In contrast, both the ADL and ADS fits, shown in Fig. 2A and 2B, respectively, accurately track the entire shape of the acid black isotherm, including the shape of the type III curve at high concentrations. Because the last three data points of the acid black isotherm were considered to have large errors, Manera et al.\(^ {19} \) did not fit the Khalboufi equation to the dye isotherm. Figure 2B shows that the Khalboufi fit of the acid black isotherm is satisfactory, but the fitted curve cannot trace the curvature of the type III curve defined by the last three data points. The Khalboufi fit is therefore similar to the BET fit but is inferior to the ADL and ADS fits. The resulting parameter estimates obtained by the model fits discussed above are summarized in Table 1. With the exception of the \( b_b \) parameter of the BET equation for the acid red isotherm, all parameters of the four isotherm equations can be estimated with reasonable standard errors.

To complement the visual analyses of the model fits described above, here we compare the model fits using the MAPE statistical metric. For the acid red isotherm, the MAPE metric increases in the following manner: ADS (2.7%) < ADL (4.6%) < Khalboufi (5.4%) < BET (17.4%). It is at once evident that the ADS equation is the best-performing model, while the BET equation is the worst-performing one. In the case of the acid black isotherm, the MAPE indicator can be arranged in the following ascending order: ADL (5.7%) < ADS (6.7%) < BET (7.0%) < Khalboufi (8.7%). Therefore, the ADL equation is the best-performing model, while the Khalboufi equation is the worst-performing one.

The two versions of the Aranovich–Donohue equation (ADL and ADS) are more effective than the BET and Khalboufi equations in describing the two dye isotherms. Since the four isotherm equations have different numbers of fitting parameters, the Akaike Information Criterion (AIC) test, which penalizes models with multiple fitting parameters, provides a more objective statistical evaluation of the model fits. In brief, the AIC test

![Figure 2](image)

**Figure 2.** Comparison of model fits and acid black isotherm data reported by Manera et al.\(^ {19} \) (a) BET (Eq. 1) and ADL (Eq. 5) fits. (b) ADS (Eq. 6) and Khalboufi (Eq. 8) fits.
endorse the model with the largest Akaike weight among a cohort of competing models. We refer readers to the book by Motulsky and Christopoulos[36] for the method of calculating the Akaike weight. In the case of the acid red isotherm, the descending order of the Akaike weights for the four model fits is the following: ADL (89%) > Khalfaooui (9%) > ADS (2%) > BET (0%). In the case of the acid black isotherm, the Akaike weight decreases in the following order: ADL (49%) > BET (25%) > Khalfaooui (17%) > ADS (9%). Evidently, the AIC test favors the ADL equation, which uses four fitting parameters to provide highly accurate fits of the two dye isotherms. Although equally accurate, the ADS equation is penalized by the AIC test for having five fitting parameters.

The ADS equation can be used to correlate experimental isotherms that do not conform to the shape of a standard type II curve. The Sips equation predicts a sigmoid type V curve when the exponent \( n_s \) is greater than unity. As can be seen in Table 1, the values of \( n_s \) for the ADS fits of the acid red and acid black isotherms are greater than unity. Although not visible in Figs. 1A and 2B, the ADS fits are, in fact, made up of a type V curve at low concentrations and a type III curve at high concentrations. A composite isotherm comprising a conspicuous type V curve at low concentrations and a type III curve at high concentrations has been reported by Yılmaz et al.[37] Fig. 3A shows this isotherm, which was measured for the adsorption of phenol by a modified bentonite adsorbent. As can be seen in Fig. 3A, the phenol isotherm is well described by the ADS equation. The ADL fit, however, shows significant deviations at low solution concentrations. It is obvious that the phenol isotherm exhibits a sigmoid type V curve in the low concentration range. An enlarged view of the type V curve in the solution concentration range of 0–400 mg L\(^{-1}\) is shown in Fig. 3B. The ADS equation can track the sigmoid shape of the type V curve, but the ADL equation is clearly inadequate because the Langmuir equation is confined to tracing hyperbolic type I curves. As a result, the ADS equation is much more effective than the ADL equation in correlating the overall shape of the phenol isotherm. The resulting parameter estimates for the ADS and ADL fits are listed in Table 2.

Figure 4 shows the fits of the BET and Khalfaooui equations to the phenol isotherm. The resulting parameter estimates are summarized in Table 2. As can be seen in Fig. 4A, the BET fit is very poor, missing most of the data points. By contrast, Fig. 4A shows that the Khalfaooui fit can track the overall shape of the phenol isotherm, including the type V curve in the low concentration range. However, Fig. 4B shows that there are significant deviations between the Khalfaooui fit and experimental data at \( c < 100 \) mg L\(^{-1}\). The ADS fit depicted in Fig. 3B seems to be more effective than the Khalfaooui fit shown in Fig. 4B in capturing the overall shape of the type V curve in the low concentration range.

The Aranovich–Donohue equation has been applied to several gas adsorption systems.[38–41] It has been used, in the form of ADL, to describe a limited number of liquid phase adsorption systems. The earliest example is the work of Aranovich and Donohue,[34] who used the ADL equation to describe the adsorption of organic compounds by carbon adsorbents. The ADL equation has been used by Liang et al.[42] to describe Pb(II) adsorption on a layered double hydroxide adsorbent. A good fit was obtained, but the experimental isotherm exhibited a typical type II curve shape, which could have been described equally well by the BET equation. Liang et al.[42] attributed the adsorption process to a mechanism of surface-induced precipitation of Pb(II), resulting in a multilayer adsorption/precipitation process. It is conceivable that such an adsorption/
precipitation mechanism could be operative in the acid red and acid black adsorption systems studied by Manera et al.\textsuperscript{19} In more recent studies, the ADL equation has been applied to several ion-exchange\textsuperscript{43–46} and water contaminant\textsuperscript{29} adsorption data sets. However, little to no research has examined the ability of the ADS equation to fit type II isotherm data.

### Conclusions

This work has demonstrated the effectiveness of the Aranovich–Donohue equation in fitting the experimental isotherms of acid red, acid black, and phenol. The Aranovich–Donohue equation has a flexible structure, as it allows users to select an isotherm model to describe the curve shape in the low concentration range, which is typically type I. Therefore, isotherm models such as Langmuir, Freundlich, Toth, and Sips may be used in the Aranovich–Donohue equation. In this work, the Sips isotherm was selected for the Aranovich–Donohue equation. The Aranovich–Donohue–Sips equation was found to fit the acid red and acid black isotherms with intricate curve shapes to a significant degree of precision. In contrast, the performance of the original BET equation was poor due to its inflexible functional form. The phenol isotherm, comprising a type V curve at low concentrations and a type III curve at high concentrations, was also well described by the Aranovich–Donohue–Sips equation. Because of its ability to fit both type I and type V curves in the low concentration range, the Sips equation is recommended for use in the Aranovich–Donohue equation.

### Disclosure statement

The authors declare that they have no known competing financial interests.

### ORCID

Khim Hoong Chu [http://orcid.org/0000-0003-4012-0667](http://orcid.org/0000-0003-4012-0667)
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