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Sorption of Congo Red onto Bentonite: An Isothermal Remodelling Exercise

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Editorial

The error distortion caused by the linearized plots of isotherm was avoided by re-analysis of the equilibrium data previously reported for the adsorption of Congo Red onto bentonite by direct nonlinear regression. The data were digitized from the published equilibrium curve and fitted to 19 one-, two- and three-parameter models. The root-mean-square error, the adjusted coefficient of determination, the bias and the accuracy factors, and three information criteria were used to evaluate the model performance. Ranking was based on the diagnostic used: Moreau resulted in the lowest RMSE (0.339 mg g⁻¹) and Hannan-Quinn value, Vieth-Sladek gave the lowest reported BIC, and Freundlich gave the lowest corrected Akaike value. This dependence on the criterion and the small number of equilibrium observations do not justify selection on the basis of goodness of fit alone. The two-parameter Freundlich model provided a close empirical description (RMSE 0.658 mg g⁻¹; adjusted R² 0.99). The Langmuir fit estimated a monolayer capacity of 37.38 mg g⁻¹ (95% confidence interval 24.367-50.393 mg g⁻¹) and an affinity constant of 0.17 L mg⁻¹ (95% confidence interval 0.036-0.310 L mg⁻¹). A Halsey rearrangement of the Freundlich relation gave 26.52 mg g⁻¹, close to the maximum loading observed. These results show that nonlinear fitting improves interpretation of parameters, but mechanistic conclusions remain limited due to sparse data and the uncertainty of parameters.

INTRODUCTION

Congo Red is an anionic diazo dye extensively used in the textile and allied manufacturing industry. Dye residues in wastewater are visible, decrease the penetration of light and may contribute to the chemical load of receiving waters. Many industrial dyes are resistant to conventional biological treatment, hence, adsorption remains an important polishing option when the adsorbent is abundant and cheap [1,2]. Clay minerals are attractive adsorbents because their layered structures, exchangeable ions and surface functional groups can support dye uptake. Vimonses et al. compared the ability of bentonite, kaolin and zeolite under batch conditions and found that among the tested clays, sodium bentonite had the highest Congo Red uptake [1]. The data were analyzed in terms of linearized Langmuir and Freundlich equations for bentonite. Linear transformation alters the weighting and error structure of the observations and thus may bias both parameter estimates and model comparisons [3,4].

Numerous number of equilibrium equations exists, but a small residual error does not prove an adsorption mechanism per se. While sparse curve fitting with flexible three-parameter equations can closely follow the curve, it can also lead to unstable or physically implausible parameters. Hence, information criteria, residual based measures, confidence intervals, parameter constraints and agreement with observed loading should be considered together [3,5,6]. In this work the published bentonite equilibrium curve was reanalyzed by non-linear regression, 19 candidate equations with not more than three fitted parameters were compared, and the uncertainty and physical interpretability of the leading fits examined.

MATERIALS AND METHODS

Source and reconstruction of equilibrium data

Equilibrium concentration (C_e , mg L⁻¹) and equilibrium adsorption capacity (q_e , mg g⁻¹) were reconstructed from Fig. 6a of Vimonses et al. [1] with WebPlotDigitizer version 2.5. The reconstructed values were treated as secondary data; no new adsorption experiment was conducted. This distinction is important because digitization adds uncertainty beyond the experimental error in the source study.

Candidate isotherms and nonlinear fitting

In this study, nineteen equilibrium equations were evaluated: Henry, Langmuir, Jovanovic, Freundlich, Dubinin-Radushkevich, Temkin, Redlich-Peterson, Sips, Toth, Hill, Khan, Brunauer-Emmett-Teller (BET), Vieth-Sladek, Radke-Prausnitz, Brouers-Sotolongo, Fritz-Schlunder III, Unilan, Fowler-Guggenheim, and Moreau. The digitized curve had few number of observations, thus, three adjustable parameters isotherm models were used. The nonlinear forms of the Langmuir and Freundlich equations [7-9] were fitted. For the remaining models [10-15] the original equations and parameter conventions were used.

The solubility term is required by the form of the Dubinin-Radushkevich potential corrected for the aqueous phase, and was interpreted with caution [16,17]. The nonlinear fitting was performed using CurveExpert Professional version 2.6.5. Implicit equations were solved in MATLAB (MathWorks, USA). Model parameters were estimated by least squares. Reported 95% confidence intervals were determined from the covariance of the fitted parameters. A weighting scheme was not reported in the source manuscript.

Model evaluation

Fit quality was evaluated using RMSE, adjusted R², the accuracy factor (AF), the bias factor (BF), the corrected Akaike information criterion (AICc), the Bayesian information criterion (BIC), and the Hannan-Quinn criterion (HQC). For RMSE and the information criteria, smaller values indicate better performance. Values of AF and BF closer to 1 indicate smaller multiplicative error and bias. Because each criterion emphasizes a different aspect of fit or complexity, rankings were interpreted jointly rather than reduced to a single score [5,6].

RESULTS

Fit statistics

The equilibrium data was analyzed by using models such as Moreau, Vieth-Sladek, BET, Radke-Prausnitz, Khan, Toth, Freundlich, Hill, Fritz-Schlunder III, Sips, Temkin, Brouers-Sotolongo, Langmuir, Unilan, Fowler-Guggenheim, Jovanovic, Redlich-Peterson, Henry and Dubinin-Radushkevich (**Table 1**) for finding the best fit through the use of non-linear regression. Except Baudu, Marczewski-Jaroniec, Fritz-Schlunder-IV, Weber-van Vliet, Henry and Dubinin-Radushkevich models, all the other models showed a good fit with data (**Figs. 1-19**). Most equations followed the monotonic increase of q_e but Henry and Dubinin-Radushkevich did not properly describe the curvature. The Moreau equation produced the smallest RMSE (0.339 mg g⁻¹) and HQC (-13.66), closely followed by BET for RMSE (0.345 mg g⁻¹) and HQC (-13.44). Vieth-Sladek had the lowest reported BIC (-11.56), while Freundlich had the lowest AICc (14.549). Freundlich also combined a relatively small RMSE (0.658 mg g⁻¹), adjusted R² of 0.99, BF of 0.993, and AF of 1.046. The rankings therefore changed with the criterion used (**Table 2**).

Table 1. Mathematical models that were used in modelling data [9].

Isotherm	p	Formula	Interpretive class
Henry's law	1	$q_e = HC_e$	Linear partitioning
Langmuir	2	$q_e = \frac{q_{mL}b_L C_e}{1 + b_L C_e}$	Finite homogeneous monolayer
Jovanovic	2	$q_e = q_{mJ}(1 - e^{-K_J C_e})$	Exponential monolayer
Freundlich	2	$q_e = K_F C_e^{\frac{1}{n_F}}$	Empirical heterogeneous surface
Dubinin-Radushkevich	2	Incorrect form $q_e = q_{mDR} \exp \left\{ -K_{DR} \left[RT \ln \left(1 + \frac{1}{C_e} \right) \right]^2 \right\}$ correct form $q_e = q_{mDR} \exp \left\{ -K_{DR} \left[RT \ln \left(\frac{C_s}{C_e} \right) \right]^2 \right\}$	Micropore filling
Temkin	3	$q_e = \frac{RT}{b_T} \{ \ln(a_T C_e) \}$	Coverage-dependent adsorption energy
Redlich-Peterson	3	$q_e = \frac{K_{RP1} C_e}{1 + K_{RP2} C_e^{\beta_{RP}}}$	Hybrid three-parameter relation
Sips	3	$q_e = \frac{K_S q_{mS} C_e^{\frac{1}{n_S}}}{1 + K_S C_e^{\frac{1}{n_S}}}$	Heterogeneous finite-capacity relation
Toth	3	$q_e = \frac{q_{mT} C_e}{(K_T + C_e^{n_T})^{1/n_T}}$	Asymmetric heterogeneous relation
Hill	3	$q_e = \frac{q_{mH} C_e^{n_H}}{K_H + C_e^{n_H}}$	Cooperative/sigmoidal relation
Khan	3	$q_e = \frac{q_{mK} b_K C_e}{(1 + b_K C_e)^{a_K}}$	Generalized saturation relation
BET	3	$q_e = \frac{q_{mBET} \alpha_{BET} C_e}{(1 - \beta_{BET} C_e)(1 - \beta_{BET} C_e + \alpha_{BET} C_e)}$	Multilayer relation
Vieth-Sladek	3	$q_e = \frac{q_{mVS} b_{VS} C_e}{(1 + b_{VS} C_e)^{n_{VS}}}$	Dual-mode relation
Radke-Prausnitz	3	$q_e = \frac{A_{RP} B_{RP} C_e^{\beta}}{A_{RP} + B_{RP} C_e^{\beta-1}}$	Hybrid power-law relation
Brouers-Sotolongo	3	$q_e = q_{mBS} \left(1 - \exp(-K_{BS} C_e^{\frac{1}{n_{BS}}}) \right)$	Stretched-exponential relation
Fritz-Schlunder-III	3	$q_e = \frac{q_{mFS} K_{FS} C_e}{1 + K_{FS} C_e^{n_{FS}}}$	Generalized empirical relation
Fowler-Guggenheim*	3	$q_e = q_{mFG} \frac{K_L C_e e^{\frac{a_{qL}}{C_e}}}{1 + K_L C_e e^{\frac{a_{qL}}{C_e}}}$	Lateral-interaction relation
Moreau	3	$q_e = q_{mM} \frac{b C_e + l b^2 C_e^2}{1 + 2b C_e + l b^2 C_e^2}$	Cooperative binding relation
Unilan	3	$q_e = \frac{q_{mU}}{2b_U} \ln \left(\frac{a_U + C_e e^{b_U}}{a_U + C_e e^{-b_U}} \right)$	Uniform energy distribution
Baudu	4	$q_e = \frac{q_{mB} b_B C_e^{(1+x+y)}}{1 + b_B C_e^{(1+x)}}$	Empirical model accounting for surface heterogeneity and interactions among adsorbed molecules
Marczewski-Jaroniec	4	$q_e = q_{mMJ} \left(\frac{(K_{MJ} C_e)^{n_{MJ}}}{1 + (K_{MJ} C_e)^{n_{MJ}}} \right)^{\frac{m_{MJ}}{n_{MJ}}}$	Generalized heterogeneous-surface model describing a distribution of adsorption-site energies
Fritz-Schlunder-IV	4	$q_e = \frac{A_{FS} C_e^{a_{FS}}}{1 + B_{FS} C_e^{b_{FS}}}$	Four-parameter empirical model for adsorption on energetically heterogeneous surfaces
Weber-van Vliet*	4	$C_e = P_1 q_e^{(P_2 q_e^{P_3} + P_4)}$	Empirical model describing nonlinear adsorption over a broad equilibrium-concentration range

Note *Implicit equation or function.

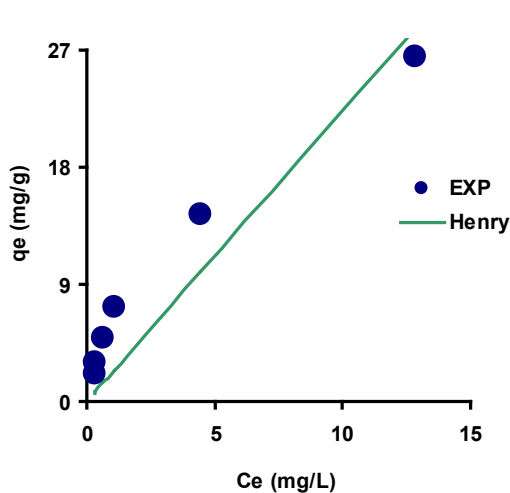


Fig. 1. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Henry.

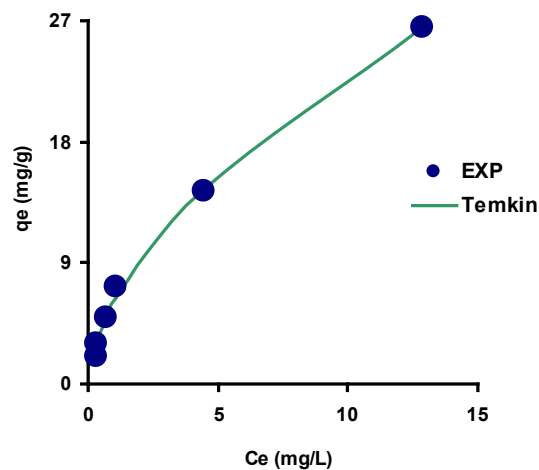


Fig. 4. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Temkin.

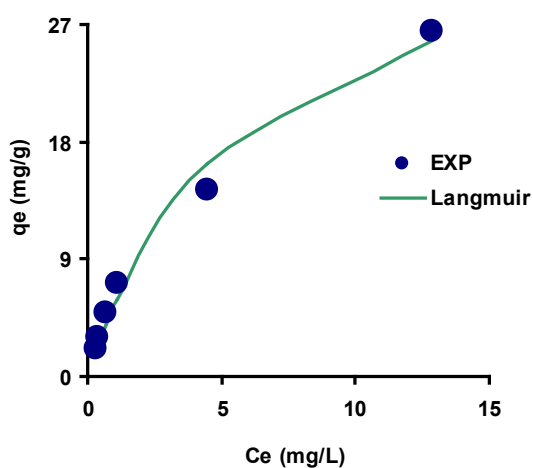


Fig. 2. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Langmuir.

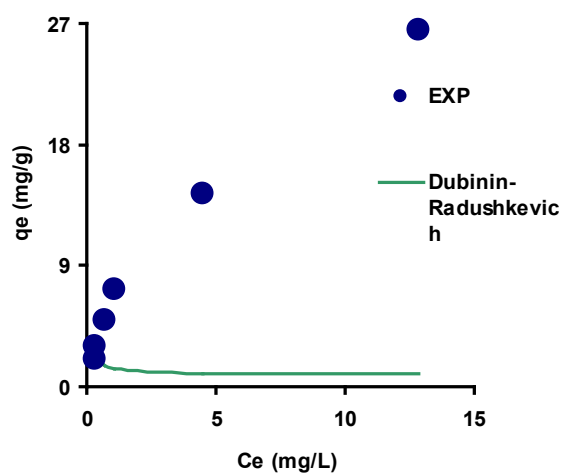


Fig. 5. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Dubinin-Radushkevich.

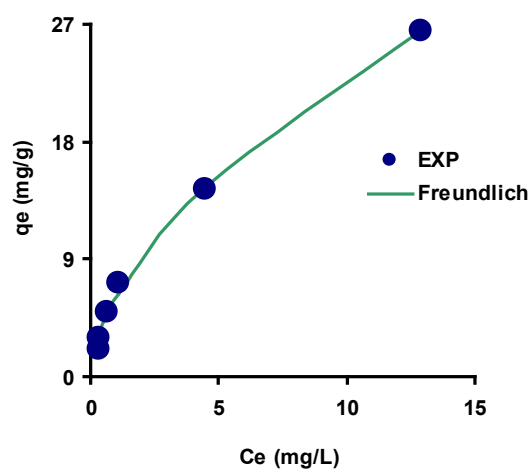


Fig. 3. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Freundlich.

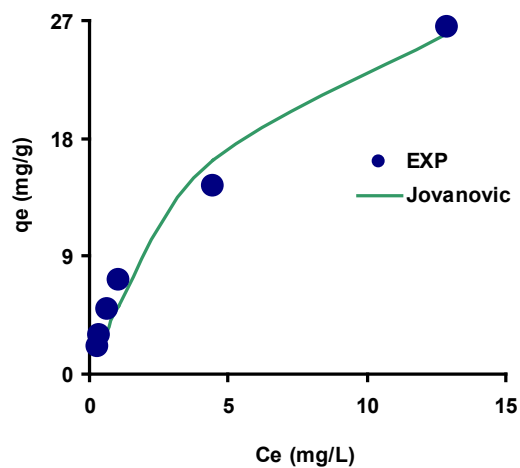


Fig. 6. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Jovanovic.

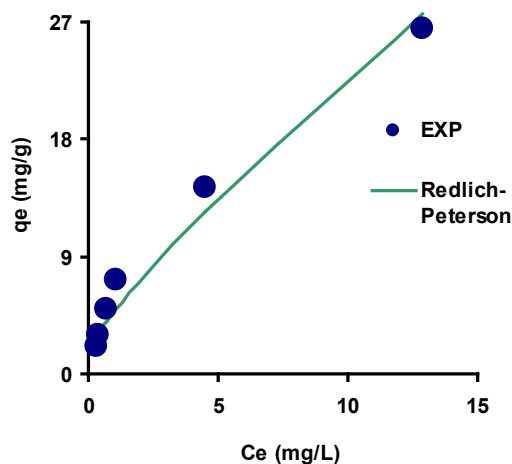


Fig. 7. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Redlich-Peterson.

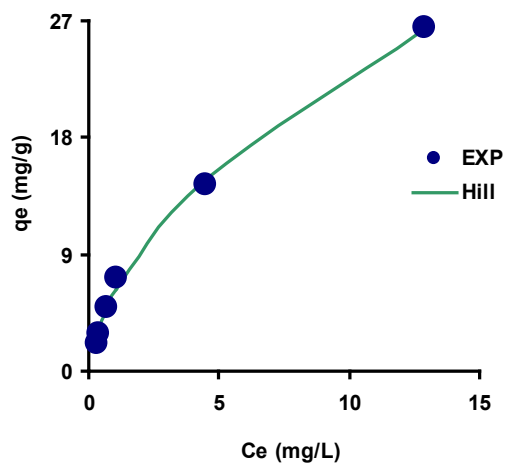


Fig. 10. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Hill.

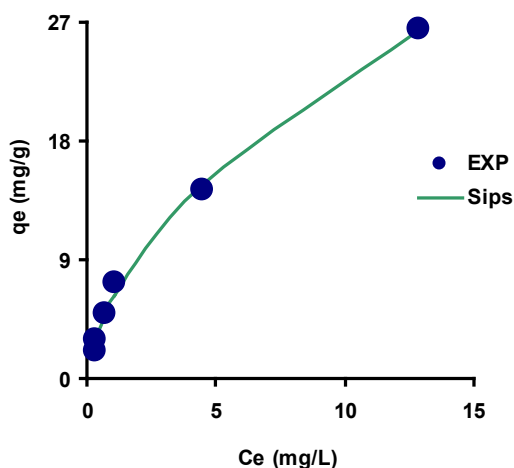


Fig. 8. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Sips.

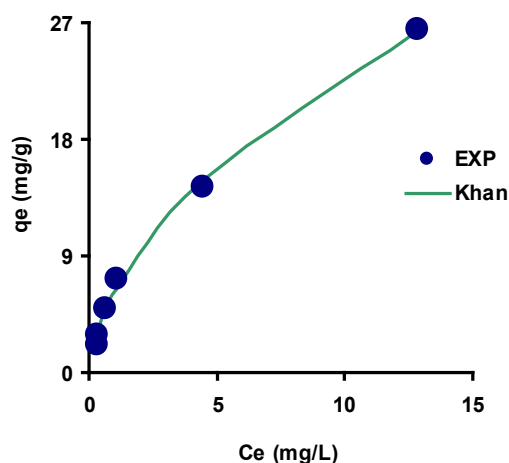


Fig. 11. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Khan.

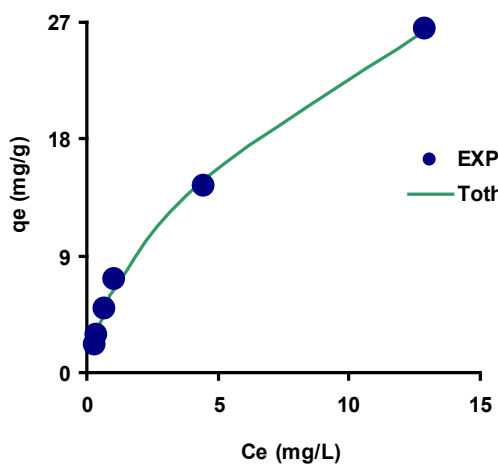


Fig. 9. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Toth.

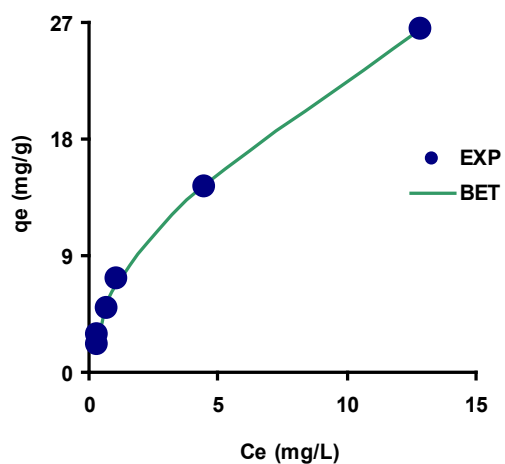


Fig. 12. Fitting Congo Red adsorption data onto bentonite using the isotherm model of BET.

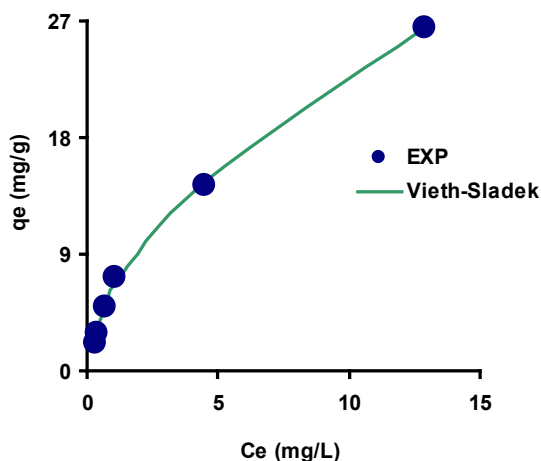


Fig. 13. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Vieth-Sladek.

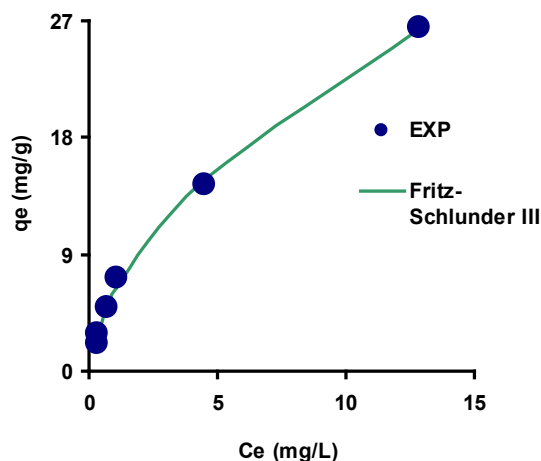


Fig. 16. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Fritz-Schlunder III.

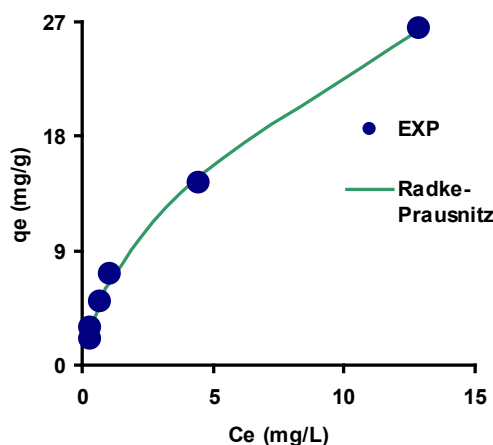


Fig. 14. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Radke-Prausnitz.

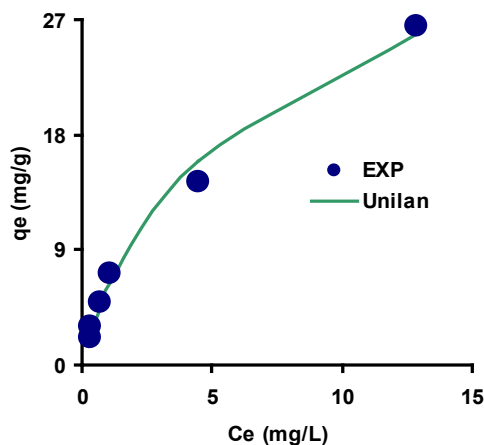


Fig. 17. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Unilan.

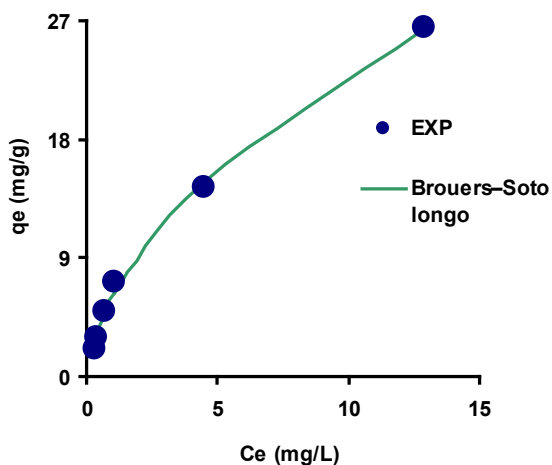


Fig. 15. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Brouers-Sotolongo.

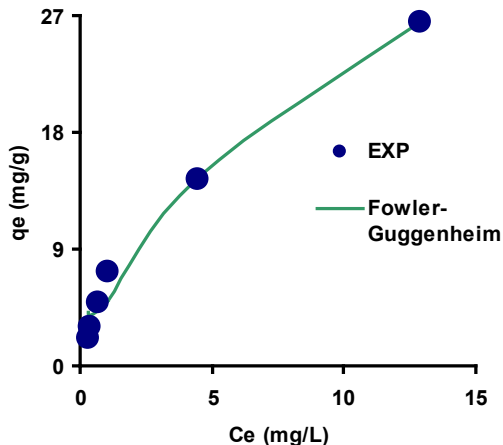


Fig. 18. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Fowler Guggenheim.

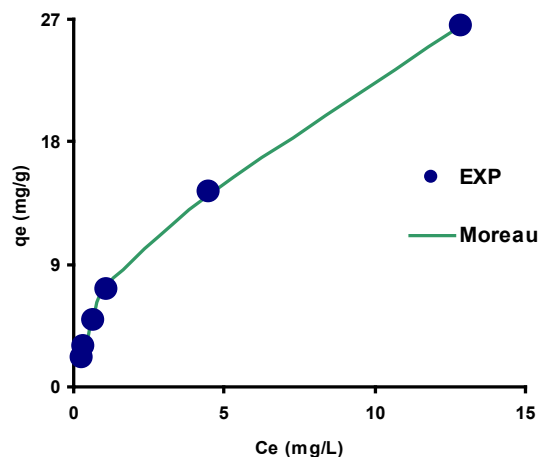


Fig. 19. Fitting Congo Red adsorption data onto bentonite using the isotherm model of Moreau.

Table 2. Error function analysis for the fitting of the isotherm of Congo Red onto bentonite.

Model	p	RMSE	adjR ²	AICc	BIC	HQC	BF	AF
Henry	1	3.632	0.86	24.384	0.00	15.55	0.519	1.979
Langmuir	2	1.427	0.97	23.838	16.18	4.17	0.891	1.171
Freundlich	2	0.658	0.99	14.549	5.42	-5.12	0.993	1.046
Temkin	3	0.760	0.99	46.549	-3.87	-3.95	0.993	1.046
Dubinin-Radushkevich	2	14.789	-291.32	51.894	-2.08	32.23	0.090	11.153
Jovanovic	2	1.729	0.96	26.136	33.48	6.47	0.835	1.249
Redlich-Peterson	3	2.078	0.93	58.616	7.72	8.12	0.913	1.165
Sips	3	0.731	0.99	46.080	9.99	-4.42	0.987	1.045
Toth	3	0.657	0.99	44.808	-2.54	-5.69	0.985	1.037
Hill	3	0.731	0.99	46.080	-3.82	-4.42	0.987	1.045
Khan	3	0.628	0.99	44.266	-2.54	-6.23	0.985	1.034
BET	3	0.345	1.00	37.064	-4.36	-13.44	0.993	1.010
Vieth-Sladek	3	0.432	1.00	39.760	-11.56	-10.74	0.992	1.017
Radke-Prausnitz	3	0.628	0.99	44.266	-8.86	-6.23	0.985	1.034
Brouers-Sotolongo	3	0.733	0.99	46.108	-4.36	-4.39	0.987	1.045
Fritz-Schlunder III	3	0.674	0.99	45.110	-2.52	-5.39	0.985	1.039
Unilan	3	1.215	0.98	52.175	-3.51	1.67	0.943	1.099
Fowler-Guggenheim	3	1.858	0.94	57.274	3.55	6.77	0.964	0.964
Moreau	3	0.339	1.00	36.843	8.65	-13.66	0.984	0.984

Note:
 RMSE Root mean Square Error
 adjR² Adjusted Coefficient of determination
 p no of parameters
 AF Accuracy factor
 BF Bias factor
 BIC Bayesian Information Criterion
 AICc Adjusted Akaike Information Criterion
 HQC Hannan-Quinn information criterion

Parameter estimates

The Langmuir model estimated q_m at 37.38 mg g⁻¹ (95% CI 24.367-50.393 mg g⁻¹) and b at 0.17 L mg⁻¹ (95% CI 0.036-0.310 L mg⁻¹). The large range of the capacity is a result of the weak definition of the saturation region. For Freundlich the value of K_F was 6.082 in the composite units corresponding and n was 1.74. As Freundlich has no finite asymptote, its capacity cannot be read directly from K_F . Application of the Halsey rearrangement at the highest equilibrium concentration gave 26.52 mg g⁻¹, close to the greatest loading observed, but not equal to a fitted saturation capacity.

Several three-parameter equations were close fits by visual inspection, but their intervals showed weak identifiability. Khan, Radke-Prausnitz and Toth included confidence limits crossing zero for at least one nominally positive parameter (Table 3). Such estimates are not physically interpretable in the absence of constrained fitting and should not be preferred solely because their residual errors are small.

Table 4. Isothermal models' constants for the top seven models.

Model	Unit	Value	(95% confidence interval)
BET	q_{mBET} mg g ⁻¹	15.764	12.751 to 18.777
	a_{BET}	0.65	0.401 to 0.899
	b_{BET}	0.03	0.03 to 0.04
Vieth-Sladek	q_{mVS} mg g ⁻¹	11.27	6.140 to 16.397
	b_{VS}	0.86	0.194 to 1.516
	K_{VS}	1.252	0.888 to 1.616
Khan	q_{mK} mg g ⁻¹	3.412	-6.671 to 13.494
	b_K	3.82	-12.372 to 20.005
	a_K	0.47	0.280 to 0.666
Radke-Prausnitz [#]	b	0.49	0.128 to 0.848
	B_{RP} mg g ⁻¹	8.29	-1.720 to 18.306
	A_{RP} L g ⁻¹	23.63	-57.158 to 104.410
Freundlich [#]	K_F (mg g ⁻¹ .L mg ⁻¹) ^{1/nF}	6.082	5.157 to 7.006
	n_F L mg ⁻¹	1.74	1.533 to 1.937
Langmuir	q_{mF} mg g ⁻¹	26.520	
	q_{mL} mg g ⁻¹	37.38	24.367 to 50.393
	b_L L mg ⁻¹	0.17	0.036 to 0.310
Toth	q_{mT} mg g ⁻¹	7.389	1.486 to 13.292
	K_T L mg ⁻¹	0.30	-1.046 to 1.641
	n_T	0.70	0.507 to 0.892

Note: [#]Isotherms that have no direct way in estimating the maximum adsorption capacity (mg g⁻¹).

DISCUSSION

The main finding shows that there is not a single equation that satisfies all error function, but rather the instability of model ranking across the diagnostics. BET and Moreau gave the least residual error, Vieth-Sladek the least reported BIC and Freundlich the least AICc. With few observations the residual accuracy of three-parameter models can be improved while the parameters are poorly identified, and this concern is reinforced by the negative lower confidence bounds obtained for some nominally positive constants. Hence, a fitted curve should be taken as a description of the performance, but not as proof of multilayer adsorption, cooperativity or a specific energy distribution, and this is the common norm in many adsorption isotherm studies with points lower than 10 would give poor mechanistic interpretation [3,8]. Freundlich offers a sparse empirical description of the bentonite curve. He concurs with the conclusion of the original study that bentonite equilibrium was better described by Freundlich than by a linearized Langmuir plot [1]. Its exponent $n_F = 1.74$ indicates a sublinear increase over the measured range. However, the equation does not tend to a finite capacity, and therefore the 26.52 mg g⁻¹ value calculated by rearrangement is a concentration-dependent estimate and not an independently fitted maximum.

The model Langmuir provides a finite and readily comparable capacity, but the estimate of 37.38 mg g⁻¹ has a wide 95% interval. Because there is only one observation in the data set near the upper end of the concentration range, the asymptote is extrapolated and is sensitive to small changes in that point. Thus this value should be considered a model based estimate rather than a measured saturation capacity. This caution is also applicable to the present bentonite result when compared with high capacity engineered adsorbents that may have extremely different pH, temperature, dose, ionic strength and concentration ranges [18-20]. The model Dubinin-Radushkevich fit was very poor and should not be used to infer adsorption energy or to assign physical vs. chemical uptake. The widely used potential term for aqueous systems is not dimensionally consistent in the absence of adsorbate solubility, and the traditional energy threshold has been questioned [16,17]. In addition, the Temkin equation must be dimensionally consistent and its approximate concentration range [15]. These limitations illustrate the need to separate assessments of mathematical fit from mechanistic validity. In this remodelling analysis, values were reconstructed

from a published figure, rather than from the original numerical dataset. The limited number of observations limits discrimination among similarly shaped equations and diminishes confidence-interval estimation. Experimental weights were not available and variability could not be replicated, therefore residual independence could not be tested reliably, and external validation was not possible. Future work should fit the original replicate-level data, report residual plots and parameter correlations, apply physically justified constraints, and add concentrations that define the approach to saturation.

CONCLUSION

In this study, the use of a direct nonlinear regression avoided the need to transform the bentonite equilibrium data and showed a strong dependence of model ranking on the chosen diagnostic. With this sparse dataset, no model can be claimed as best in all circumstances as each of Moreau, Vieth-Sladek, Freundlich and BET were each supported by at least one reported statistic. Freundlich gave a good description empirically with two parameters. Langmuir estimated $q_m = 37.38 \text{ mg g}^{-1}$ and $b = 0.17 \text{ L mg}^{-1}$ with wide confidence intervals. The results support nonlinear, multi-criterion evaluation and cautious interpretation of fitted parameters, in particular in case of weak sampling of the saturation region.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this article.

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AUTHORS' CONTRIBUTIONS

The conceptualization of the study was led by I.A.S. Data analysis was conducted by I.A.S. with the remaining authors contributing to the review and editing.

DATA AVAILABILITY STATEMENT

The data analysed in this study were reconstructed from the published equilibrium figure cited as reference [1].

AI USAGE DECLARATION

During the preparation of this work, the authors used OpenAI ChatGPT solely for language editing, grammatical correction, formatting support, and refinement of readability. The authors reviewed and edited the resulting text and take full responsibility for the accuracy, integrity, originality, and final content of the publication.

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