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ORIGINAL PAPER

Isotherms for the sorption of zinc and copper onto kaolinite: comparison of various error functions

Sh. Shahmohammadi-Kalalagh · H. Babazadeh

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Abstract In this research, the equilibrium sorption of Zn(II) and Cu(II) by kaolinite was explained using the Freundlich, Langmuir and Redlich–Peterson isotherms, via both linear and non-linear regression analyses. In the case of non-linear regression method, the best-fitting model was evaluated using six different error functions, namely coefficient of determination (r^2), hybrid fractional error function (HYBRID), Marquardt's percent standard deviation (MPSD), average relative error (ARE), sum of the errors squared (SSE) and sum of the absolute errors (EABS). The examination of error estimation methods showed that the Langmuir model provides the best fit for the experimental equilibrium data for both linear and non-linear regression analyses. The SSE function was found to be a better option to minimize the error distribution between the experimental equilibrium data and predicted two-parameter isotherms. In the case of three-parameter isotherm, HYBRID was found to be the best error function to minimize the error distribution structure between experimental equilibrium data and theoretical isotherms. Non-linear method was found to be more appropriate method for estimating the isotherm parameters.

Keywords Adsorption · Freundlich · Langmuir · Redlich–Peterson · Regression analysis

Sh. Shahmohammadi-Kalalagh (✉)
Department of Water Sciences and Engineering,
East Azarbaijan Science and Research Branch,
Islamic Azad University, Tabriz, Iran
e-mail: shahmohammadi_sh@hotmail.com

H. Babazadeh
Department of Water Sciences and Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran

Introduction

Heavy metals in the environment are a source of some concern because of their potential reactivity, toxicity, and mobility in the soil. In order to solve the problems of heavy metal pollution in the environment, it is important to bring pragmatic solutions to the issue. There are several methods for treatment of metal-contaminated effluents such as chemical precipitation (Jüttner et al. 2000; Bose et al. 2002; Wingenfelder et al. 2005), coagulation–flocculation (Ayoub et al. 2001; Semerjian and Ayoub 2003), ultrafiltration (Eckenfelder 2000), flotation (Jokela and Keskitalo 1999; Matis et al. 2003), ion exchange (Eckenfelder 2000) and adsorption (Abdel-Ghani et al. 2009; Tashauoei et al. 2010; Zavvar Mousavi and Seyedi 2011). They have their inherent advantages and limitations in application. Among the numerous treatment technologies developed for the removal of pollutant from the aqueous environment, adsorption is receiving increasing attention in becoming an attractive and promising technology because of its simplicity, cheaper pollution control method, ease of operation and handling (Bhattacharya et al. 2006; Kunda and Gupta 2006; Shahmohammadi-Kalalagh et al. 2011). Clay minerals are copious in nature, cost less, have a high specific surface area and exchange capacity and hence are strong candidates as adsorbents for the removal of heavy metals from wastewaters.

Equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms. Search for the best-fit adsorption isotherm using the method of least squares is the most widely used technique by several researchers (Kumar and Sivanesan 2007; Kumar et al. 2008) to predict the optimum isotherm. As an alternative to the linear transformation, non-linear optimization has also been applied by various researchers (Malek and Farooq

1996; Porter et al. 1999; Ho et al. 2002; Allen et al. 2003; Ng et al. 2003; Ho 2004) to determine isotherm parameter values which require an error function assessment, in order to evaluate the fit of the isotherm to the experimental results for the removal of variety of components from the aqueous environment. Several error deviation functions have been used to predict the optimum isotherm, such as the coefficient of determination (r^2), hybrid fractional error function (HYBRID), Marquardt's percent standard deviation (MPSD), the average relative error (ARE), sum of the errors squared (SSE) and sum of the absolute errors (EABS) (Porter et al. 1999; Ho et al. 2002; Ng et al. 2002; Allen et al. 2003; Wong et al. 2004). However, no detailed studies were available so far comparing the accuracy of these error functions in predicting the isotherm parameters and also the optimum isotherm.

In the present study, a comparison of six different error functions in minimizing the error distribution between the experimental and predicted isotherms is discussed using the experimental data of Zn(II) and Cu(II) onto kaolinite. The three widely used Freundlich, Langmuir and Redlich–Peterson isotherms were used to discuss this issue. The method of least squares was used to predict the isotherm by linear regression method. A trial and error method was used for nonlinear regression to minimize or maximize the objective function using the *solver* add-in function, Microsoft Excel, Microsoft. This study was conducted in Islamic Azad University, Science and Research Branch, Tehran, Iran in Autumn 2011.

Materials and methods

The materials used in this study are kaolinite (from the Iran china clay industries Co.) and chemical materials such as solutions and reagents. Kaolinite was used as the adsorbent. The chemical composition and some physical properties of kaolinite are presented in Table 1. The molecular structure of kaolinite is sketched in Fig. 1 (Vimonses et al. 2009). Its structural formula is $Al_2Si_2O_5OH_4$. The elements contained in pure kaolinite are arranged to form one silica sheet and one alumina sheet (Sposito 1989). Solutions of selected heavy metals (copper and zinc) were used as the adsorbate for batch adsorption experiments. With reference to the soil profile and groundwater contamination, a low pH for solution was chosen. Accordingly, hydrochloric acid was used to adjust the pH of each solution to 4.5.

Suspensions were prepared on dry solid basis into a uniform powdery texture and mixed with various concentrations of metal solutions. The initial concentrations of Cu and Zn solutions were selected as 50, 200, 500, 1,000, and 2,000 mg/L. These metal concentrations were selected to cover a wide range most commonly found in sewage sludge, some industrial wastes, and municipal solid wastes. The solutions of heavy metals were applied to the kaolinite at 1:10 (solid:solution ratio) (Yong et al. 1992), i.e., 40 g of dry kaolinite and 400 mL of solutions. The mixed solutions are mounted on the stirrer plate during experimental period, to prevent any possible sedimentation and to provide proper agitation. Following agitation, 15 mL aliquots were removed to determine metal concentrations. Aliquots were then centrifuged at 6,000 rpm for 10 min in plastic Nalgene centrifuge tubes equipped with screw-on caps.

Table 1 The chemical composition and some physical properties of kaolinite used in the present study

Constituents	Quantity (%)
Al_2O_3	38.60
SiO_2	45.71
Fe_2O_3	0.38
TiO_2	1.15
CaO	0.10
MgO	0.34
Na_2O	0.31
K_2O	0.10
Loss on ignition	13.31
Cation exchange capacity	8 meq/l00 g
Mean particle size	59×10^{-4} cm
Surface area	12.76 m^2/g
Porosity	0.42
Density	1.49 g/cm^3

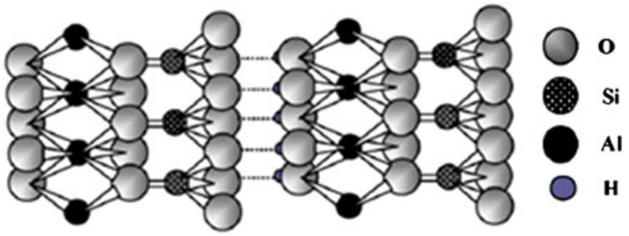

Fig. 1 Molecular structure of kaolinite (Vimonses et al. 2009)

At the end of centrifugation, the supernatant is filtered and stored in 10 ml glass vials prior to atomic absorption (AA) spectroscopy. The total metal content in the filtered solutions was determined by AA spectroscopy in accordance with standard procedures. The total initial metal concentration minus the metal concentration in the supernatant was taken as the metal adsorbed by kaolinite. Experiments were conducted (without replication) at room temperature (20 ± 1 °C), over a range of metal concentrations, keeping the initial pH constant [The kaolinite under consideration (hydrated poorly crystalline (PX)), is relatively chemically inert and reacts with acids and bases only under extreme conditions. Also, it is water processed, which reduces soluble salt contents to extremely low level (Sposito 1989). On the other hand, the adjusted pH is near the point of zero charge (PZC) value of kaolinite and it seems that no considerable change in pH during the experiment may be found].

Data evaluation

The amount of Zn(II) and Cu(II) adsorbed by the kaolinite was determined using a mass balance equation expressed as in Eq. (1):

$$q_{\mathrm{e}} = \frac{V(C_0 - C_{\mathrm{e}})}{m} \tag{1}$$

where q_{e} is metal concentration on the kaolinite (mg/g) at equilibrium, C_{e} is metal concentration in solution (mg/L) at equilibrium, C_0 is initial metal concentration in solution (mg/L), V is volume of initial metal solution used (L), and m is mass of kaolinite used (g).

Results and discussion

Linear regression analysis

The linear least-squares method to the linearly transformed isotherm equations was widely applied to confirm the experimental data and isotherms using coefficient of determination (Ho et al. 2005; Kumar and Sivanesan 2006). The widely used linearized form of Freundlich, Langmuir, and Redlich–Peterson isotherms which are found to be the best-fit expressions in representing most of the experimental equilibrium data is shown in Table 2. The Freundlich, Langmuir, and Redlich–Peterson constants can be obtained from the slope and intercept of plot between $\ln(q_{\mathrm{e}})$ versus $\ln(C_{\mathrm{e}})$, $C_{\mathrm{e}}/q_{\mathrm{e}}$ versus C_{e} and $\ln(AC_{\mathrm{e}}/q_{\mathrm{e}}-1)$ versus $\ln(C_{\mathrm{e}})$, respectively. In the case of Redlich–Peterson isotherm, the constants were obtained by maximizing the r^2 value using a trial and error procedure using the *solver* add-in with Microsoft's spreadsheet, Microsoft excel. The way of obtaining the isotherm constants was explained in Table 2.

Figures 2 and 3 show the experimental equilibrium data and the predicted theoretical isotherms using linear method for the sorption of Zn(II) and Cu(II) onto kaolinite,

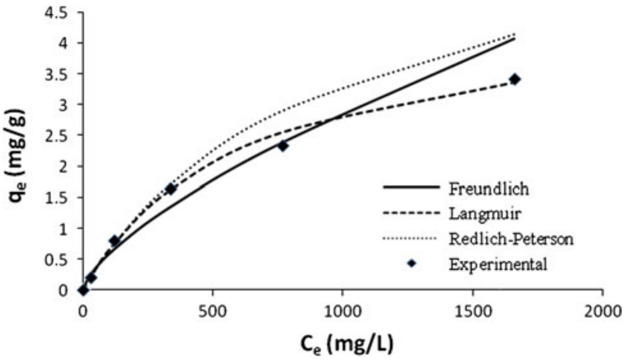

Fig. 2 Adsorption isotherms for Zn(II) onto kaolinite by linear method

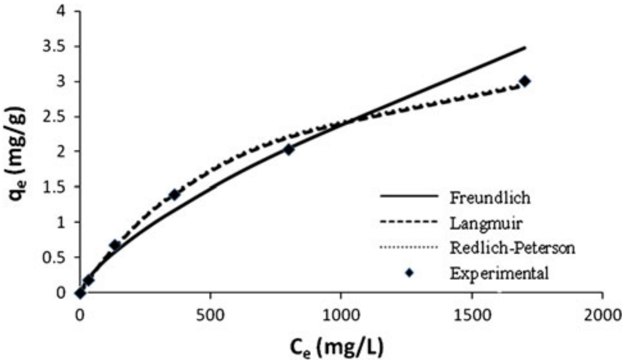

Fig. 3 Adsorption isotherms for Cu(II) onto kaolinite by linear method

Table 3 Isotherm constants for Zn(II) and Cu(II) onto kaolinite by linear method

	Zn(II)	Cu(II)
Freundlich		
K_{F} (mg/g) (L/g)^n	0.0236	0.0194
1/n	0.6944	0.6976
r^2	0.9700	0.9780
Langmuir		
K_{L} (L/mg)	0.0016	0.0014
q_{m} (mg/g)	4.6250	4.1649
r^2	0.9942	0.9945
Redlich–Peterson		
A (L/g)	0.0071	0.0060
B (L/mg$^{1-1/A}$)	0.0015	0.0015
G	0.9595	0.9944
r^2	0.9927	0.9934

Table 2 Isotherms and their linear forms (q_{m}: mg/g; K_{L}: L/mg; K_{F}: (mg/g)(L/g)^n; A: L/g; B: L/mg$^{1-1/A}$)

Isotherm	Non-linear form	Linear form	Plot
Freundlich	$q_e = K_{\mathrm{F}} C_{\mathrm{e}}^{1/n}$	$\ln(q_{\mathrm{e}}) = \ln(K_{\mathrm{F}}) + \frac{1}{n}\ln(C_{\mathrm{e}})$	$\ln(q_{\mathrm{e}})\mathrm{vs.}\ln(C_{\mathrm{e}})$
Langmuir	$q_{\mathrm{e}} = \frac{K_{\mathrm{L}} q_{\mathrm{m}} C_{\mathrm{e}}}{1+K_{\mathrm{L}} C_{\mathrm{e}}}$	$\frac{C_{\mathrm{e}}}{q_{\mathrm{e}}} = \frac{1}{K_{\mathrm{L}} q_{\mathrm{m}}} + \frac{1}{q_{\mathrm{m}}} C_{\mathrm{e}}$	$\frac{C_{\mathrm{e}}}{q_{\mathrm{e}}}\mathrm{vs.}C_{\mathrm{e}}$
Redlich–Peterson	$q_{\mathrm{e}} = \frac{AC_{\mathrm{e}}}{1+BC_{\mathrm{e}}^{g}}$	$\ln\left(A\frac{C_{\mathrm{e}}}{q_{\mathrm{e}}} - 1\right) = \ln(B) + g\ln(C_{\mathrm{e}})$	$\ln\left(A\frac{C_{\mathrm{e}}}{q_{\mathrm{e}}} - 1\right)\mathrm{vs.}(C_{\mathrm{e}})$

respectively. The predicted isotherm constants for the Zn(II) and Cu(II) and their corresponding r^2 values by the linear method were shown in Table 3. From Table 3, the relatively lower r^2 values for Freundlich isotherm when compared to Langmuir and Redlich–Peterson isotherm suggest that it may not be appropriate to use this model in representing the equilibrium uptake of Zn(II) and Cu(II) by kaolinite. From Table 3, it can be observed that Redlich–Peterson and Langmuir isotherms very well represent the experimental equilibrium data with a higher r^2 value (>0.99).

Figure 3 shows that the Redlich–Peterson and Langmuir isotherms overlapped each other, and seemed to be the best-fitting models for the experiment results with the same values of coefficient of determination (Table 3). Thus, Langmuir isotherm is a special case of Redlich–Peterson isotherm when constant g was unity ($g \approx 1$). The similar findings have been reported by other researchers as well (Kumar and Sivanesan 2007; Subramanyam and Das 2009).

Previously, several research reports suggested non-linear method as a better way to obtain the isotherm parameters as sometime linearization of non-linear experimental data may distort the error distribution structure of isotherm (Ho 2004; Kumar 2006). Based on any of the error measures, better fits can be obtained for any isotherm using non-linear isotherm. Thus, in the present study, non-linear regression method was used to determine the optimum isotherm out of the three widely used isotherms (Freundlich, Langmuir, and Redlich–Peterson) studied.

Non-linear method

Due to the inherent bias resulting from linearization, non-linear regression was applied to determine alternative isotherm parameter sets. In the case of non-linear method, previous research studies reported that the predicted isotherms were found to be varying with the error function used while minimizing the error distribution between the experimental and predicted isotherms (Porter et al. 1999; Ho et al. 2002; Ng et al. 2002; Allen et al. 2003; Wong et al. 2004). Thus, in order to analyze the impact of various error functions on the predicted isotherms, six error functions r^2, HYBRID, MPSD, ARE, SSE and EABS were

Table 4 Different error functions used for equilibrium isotherm analyses

Error function	Abbreviation	Definition/expression	Reference
The coefficient of determination	r^2	$\frac{\sum_{i=1}^{n}\left(q_{e,calc}-\bar{q}_{e,exp}\right)_i^2}{\sum_{i=1}^{n}\left(q_{e,calc}-\bar{q}_{e,exp}\right)_i^2+\sum_{i=1}^{n}\left(q_{e,calc}-q_{e,exp}\right)_i^2}$	Ho (2004)
The hybrid fractional error function	HYBRID	$\frac{100}{n-p}\sum_{i=1}^{n}\left[\frac{\left(q_{e,exp}-q_{e,calc}\right)^2}{q_{e,exp}}\right]_i$	Allen et al. (2003)
Marquardt's percent standard deviation	MPSD	$100\sqrt{\frac{1}{n-p}\sum_{i=1}^{n}\left(\frac{q_{e,exp}-q_{e,calc}}{q_{e,exp}}\right)_i^2}$	Allen et al. (2003)
The average relative error	ARE	$\frac{100}{n}\sum_{i=1}^{n}\left\lvert\frac{q_{e,exp}-q_{e,calc}}{q_{e,exp}}\right\rvert_i$	Allen et al. (2003)
The sum of the errors squared	SSE	$\sum_{i=1}^{n}\left(q_{e,calc}-q_{e,exp}\right)_i^2$	Ng et al. (2002)
The sum of the absolute errors	EABS	$\sum_{i=1}^{n}\left\lvert q_{e,calc}-q_{e,exp}\right\rvert_i$	Ng et al. (2002)

n is the number of experimental data points, p is the number of parameters in each isotherm model, $q_{e,cal}$ (mg/g) is the theoretically calculated adsorption capacity at equilibrium and $q_{e,exp}$ (mg/g) is the experimental adsorption capacity at equilibrium

Table 5 Isotherm error deviation data related to the sorption of Zn(II) and Cu(II) onto kaolinite using six commonly used functions

Error functions	r^2		ARE		MSPD		HYBRID		EABS		SSE	
	Zn(II)	Cu(II)	Zn(II)	Cu(II)	Zn(II)	Cu(II)	Zn(II)	Cu(II)	Zn(II)	Cu(II)	Zn(II)	Cu(II)
Linear form												
Freundlich	0.9700	0.9780	16.0501	12.7778	22.7330	18.2112	7.1882	4.3217	1.1855	0.8449	0.5377	0.2873
Langmuir	0.9942	0.9945	4.5388	3.2230	7.1530	6.0292	0.8502	0.5608	0.3498	0.2730	0.0537	0.0332
Redlich–Peterson	0.9927	0.9934	11.5951	3.2487	23.7515	7.1405	15.1690	0.9773	1.4314	0.2730	0.8685	0.0394
Non-linear form												
Freundlich	0.9790	0.9877	17.1684	14.6649	26.7441	26.4800	5.2506	2.8057	0.8369	0.5192	0.2155	0.0822
Langmuir	0.9930	0.9945	5.2232	5.5862	8.1126	8.0508	0.8870	0.5955	0.4069	0.3075	0.0471	0.0286
Redlich–Peterson	0.9930	0.9945	5.2740	7.0924	9.9817	12.1690	1.3347	1.1769	0.4100	0.3885	0.0472	0.0375

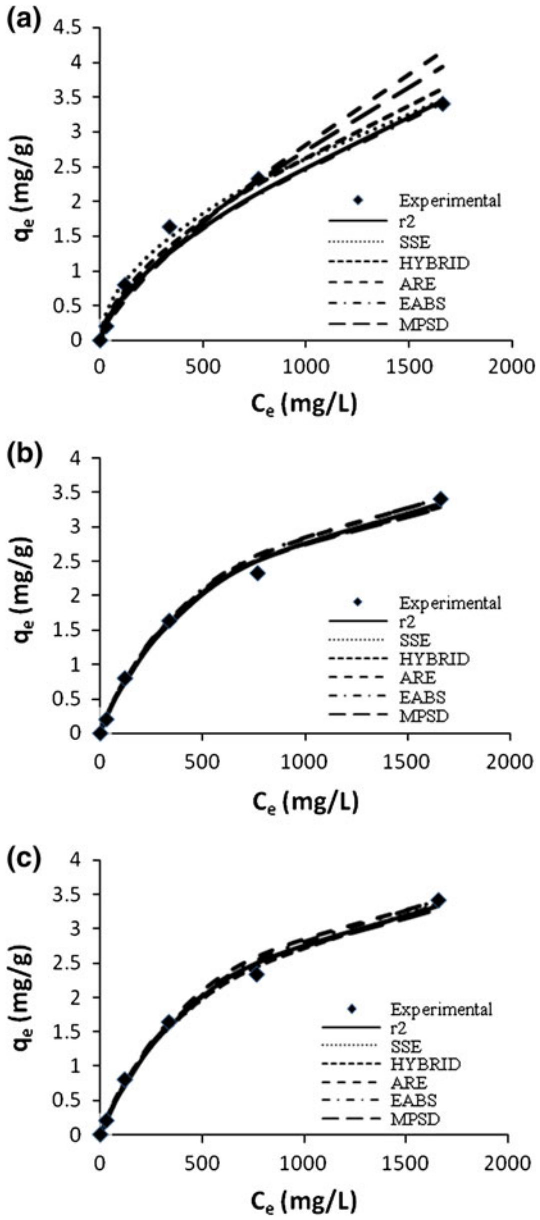

Fig. 4 Isotherm plots for Zn(II) onto kaolinite using various error analysis methods: **a** Freundlich isotherm, **b** Langmuir isotherm and, **c** Redlich–Peterson isotherm

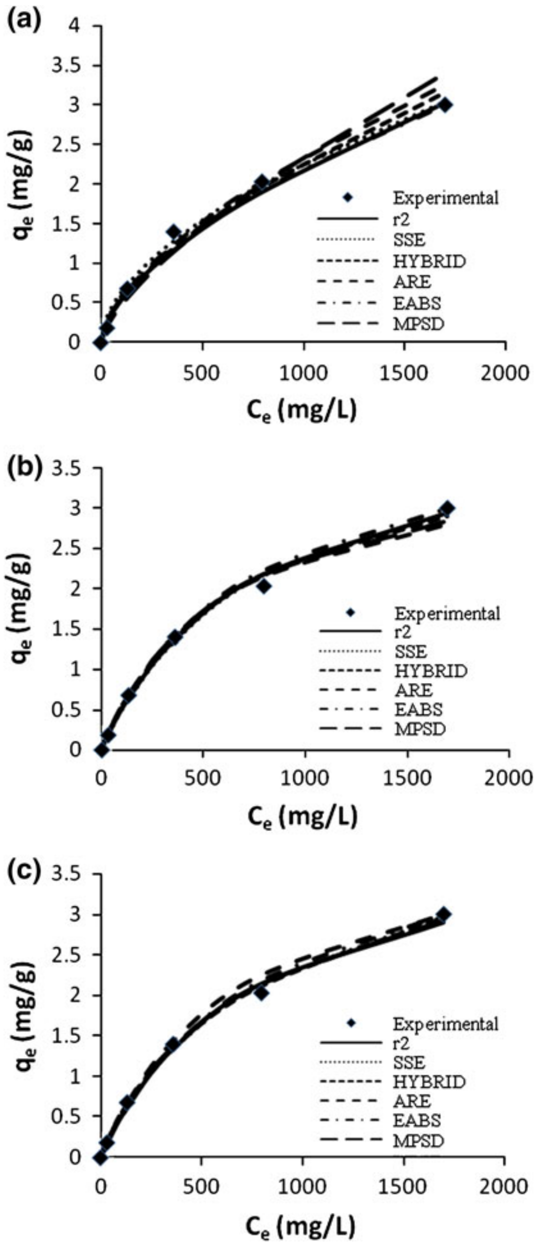

Fig. 5 Isotherm plots for Cu(II) onto kaolinite using various error analysis methods: **a** Freundlich isotherm, **b** Langmuir isotherm and **c** Redlich–Peterson isotherm

examined and in each case, the isotherm parameters were determined by minimizing the respective error function across the concentration range studied, using the *solver* add-in for Microsoft Excel. The explanations of various error functions used in the present study are given in Table 4.

By comparing the results of the values of the error functions (Table 5), it is found that the Langmuir model is the most suitable model to satisfactorily describe the studied sorption phenomenon. Indeed, the highest r^2 value and the lowest ARE, MSPD, HYBRID, EABS, and SSE values were found when modelling the equilibrium data using the Langmuir, for both linear and non-linear regression analyses (cf. Table 5).

Figure 4a–c shows the experimental and predicted Freundlich, Langmuir, and Redlich–Peterson isotherms for Zn(II) onto kaolinite by minimizing the error distribution between the experimental and the theoretical isotherms using various error functions explained in Table 4. From Fig. 4a–c, it is observed that the predicted theoretical isotherm was found to be varying with respect to the error functions used. A similar observation was found for Cu(II) onto kaolinite (Fig. 5a–c). The variation in the predicted isotherms was found to be more pronounced for Langmuir and Redlich–Peterson isotherms (Figs. 4b, c; 5b, c).

The calculated isotherm parameters and the corresponding error functions for the minimum error distribution between the experimental equilibrium data and predicted isotherm for Zn(II) and Cu(II) are given in Tables 6 and 7, respectively. From Tables 6 and 7, other than r^2, all the other error functions studied corresponding to the minimized deviations between the experimental equilibrium data and predicted isotherms. In the case of r^2, the Redlich–Peterson isotherm was found to be the best-fit isotherm followed by Langmuir isotherm.

Figure 6a, b shows the experimental equilibrium data and predicted isotherms for the sorption of Zn(II) onto kaolinite by maximizing r^2 function and minimizing the MPSD error function, respectively. From Fig. 6a, b, it can be observed that Redlich–Peterson isotherm exactly overlaps the Langmuir isotherm. Thus, Langmuir isotherm is a special case of Redlich–Peterson isotherm when constant g was unity. A similar observation was observed for the sorption of Cu(II) onto kaolinite (Fig. 7a, b). A similar trend was observed for the predicted isotherms by minimizing the error functions HYBRID, ARE, EABS, and SSE for both Zn(II) and Cu(II) (not shown).

In order to check which error function minimizes the error distribution between the experimental and theoretical isotherms, another statistical-term coefficient of non-determination, K^2, was used (Kumar et al. 2008). The coefficient of non-determination, K^2, was defined as:

$$K^2 = \frac{\text{Unexplained variance}}{\text{Total variance}} = 1 - \frac{\text{Explained variance}}{\text{Total variance}} = 1 - r^2 \tag{2}$$

The coefficient of non-determination is a much more useful measure of the linear or non-linear co-variation of two variables. The K^2 will be very much useful to derive any conclusion about the extent of the relationship between

Table 6 Predicted isotherm by minimizing the error distribution using five different error functions for Zn(II) adsorption onto kaolinite

Error functions	r^2	SSE	HYBRID	ARE	EABS	MPSD
Freundlich						
K_F	0.0321	0.0657	0.0377	0.0158	0.0321	0.0217
$1/n$	0.6305	0.5348	0.6157	0.7515	0.6292	0.7012
OF[a]	0.9790	0.0730	4.1212	14.6520	0.8343	21.9003
Langmuir						
K_L	0.0015	0.0015	0.0017	0.0015	0.0016	0.0017
q_m	4.6710	4.6723	4.4700	4.7584	4.7081	4.4516
OF[a]	0.9930	0.0471	0.7695	4.3092	0.3098	6.8767
Redlich–Peterson						
A	0.0070	0.0082	0.0085	0.0073	0.0070	0.0075
B	0.0015	0.0054	0.0058	0.0014	0.0015	0.0018
g	1.0000	0.8557	0.8522	1.0152	0.9957	0.9876
OF[a]	0.9930	0.0256	0.5144	4.2233	0.3520	6.8683

[a] Objective function for the minimum error distribution between experimental and predicted isotherms

Table 7 Predicted isotherm by minimizing the error distribution using five different error functions for Cu(II) adsorption onto kaolinite

Error functions	r^2	SSE	HYBRID	ARE	EABS	MPSD
Freundlich						
K_F	0.0311	0.0479	0.0297	0.0312	0.0393	0.0185
$1/n$	0.6152	0.5581	0.6277	0.6251	0.5829	0.7009
OF[a]	0.9877	0.0406	2.4697	13.9834	0.3976	17.8239
Langmuir						
K_L	0.0013	0.0012	0.0014	0.0016	0.0013	0.0015
q_m	4.2721	4.3305	4.0920	3.8166	4.3367	3.9100
OF[a]	0.9945	0.0279	0.5325	2.8802	0.2402	5.0934
Redlich–Peterson						
A	0.0054	0.0063	0.0069	0.0061	0.0054	0.0064
B	0.0013	0.0047	0.0066	0.0015	0.0013	0.0038
g	1.0000	0.8509	0.8189	0.9912	0.9912	0.8852
OF[a]	0.9945	0.0141	0.2250	2.9557	0.2868	4.1140

[a] Objective function for the minimum error distribution between experimental and predicted isotherms

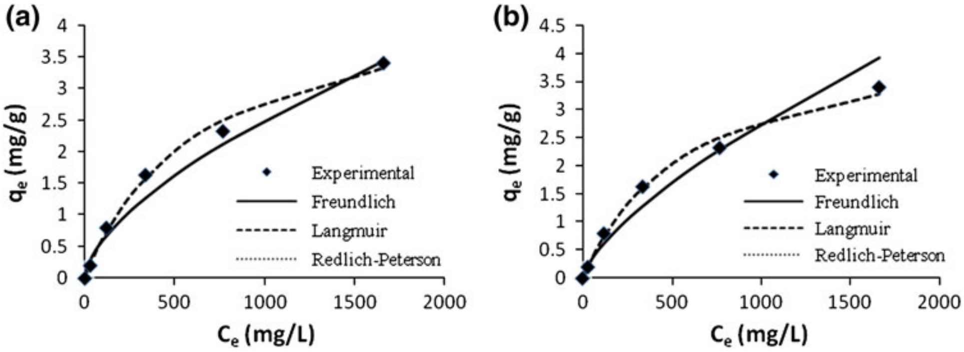

Fig. 6 **a** Isotherms by maximizing r^2 function for Zn(II) onto kaolinite. **b** Isotherms by minimizing MPSD function for Zn(II) onto kaolinite

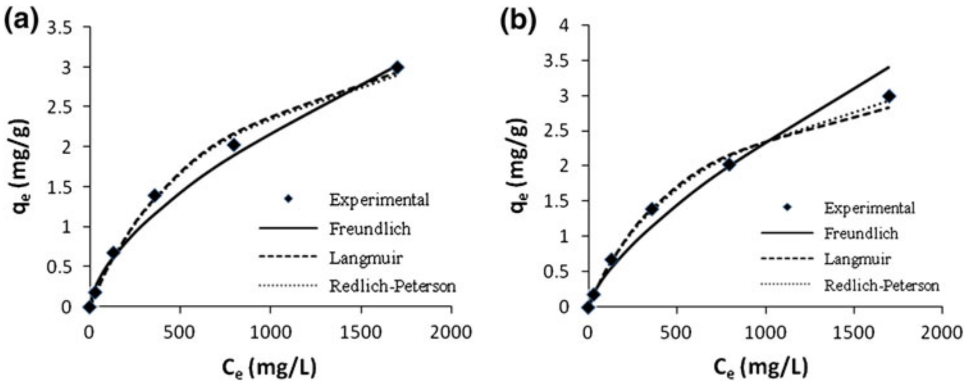

Fig. 7 **a** Isotherms by maximizing r^2 function for Cu(II) onto kaolinite. **b** Isotherms by minimizing MPSD function for Cu(II) onto kaolinite

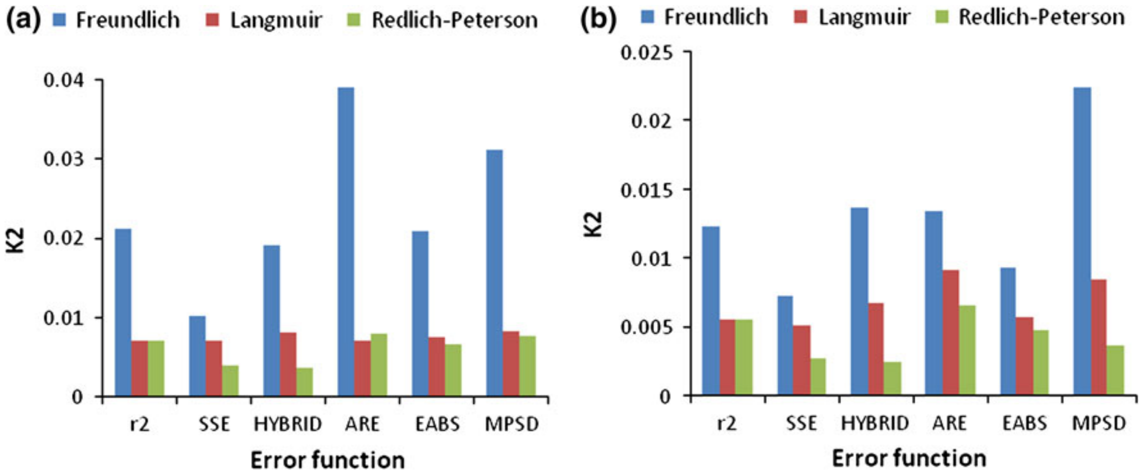

Fig. 8 Coefficient of non-determination for isotherms of **a** Zn(II), **b** Cu(II) onto kaolinite

the transformed experimental data and the predicted isotherms (Kumar et al. 2008). Figure 8a, b shows the calculated K^2 values for the isotherms predicted by minimizing or maximizing the various error functions for Zn(II) and Cu(II) onto kaolinite, respectively. From Fig. 8a, b, it is observed that SSE function least unexplain the two-parameter isotherm, suggesting this function as the best function to minimize the error distribution between the experimental and predicted two-parameter isotherms, namely the Freundlich and Langmuir isotherms. Thus, for the studied system, SSE was found to be the best error function to be used in predicting the optimum isotherm if the objective is to select the best-fit isotherm out of two-parameter isotherms. In the case of three-parameter Redlich–Peterson isotherm, it can be observed that HYBRID was found to be the best error function in minimizing the error distribution between the experimental equilibrium data and the predicted isotherms (Fig. 8a, b). Thus, the results of HYBRID can be useful in predicting the optimum isotherm based on the three-parameter Redlich–Peterson isotherm.

Conclusion

A comparison of linear and non-linear regression methods in selecting the optimum isotherm was made to the experimental equilibrium data of Zn(II) and Cu(II) sorption by kaolinite. The coefficient of determination (r^2) was used to select the best-fit linear theoretical isotherm. In the case of non-linear regression method, six error functions were used to predict the parameters involved in the two- and three-parameter isotherms and also to predict the optimum isotherm. The examination of all these error estimation methods showed that the Langmuir model provides the best fit for the experimental equilibrium data (i.e. highest r^2 and lowest SSE, HYBRID, ARE, EABS, and MPSD values). For two-parameter isotherms, a SSE function and for three-parameter isotherms, a HYBRID function were found to be a better option to minimize the error distribution between the experimental and predicted isotherms. The coefficient of non-determination was found to be more useful statistical term in identifying the best error function while selecting the optimum isotherm.

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