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Modeling of phenol adsorption isotherm onto activated carbon by non-linear regression methods: models with three and four parameters

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ABSTRACT

The adsorption equilibrium isotherms of phenol from aqueous solutions onto powdered activated carbon were studied and modeled. The activated carbons CAH1 and CAH2 were obtained by carbonizing the activated rice husks at 500°C and 450°C, respectively, while CAMH was obtained by carbonizing a mixture of 12 g rice husk and 8 g coffee husk at 500°C. In order to determine the best fit isotherm, the experimental equilibrium data were analyzed using four adsorption isotherm models with three-parameters (Redlich–Paterson, Sips, Langmuir–Freundlich, Toth) and three adsorption isotherm models with four-parameter (Fritz–Schlunder, Baudu, Marczewski–Jaroniec) equations. Four error functions, coefficient of determination (R^2), chi-square error function (χ^2), residual root mean square error (RMSE) and HYBRID fractional error function were applied to determine the best fit isotherm. The error analysis showed that the models with three parameters better described the adsorption of phenol data compared with the four-parameter models. All the values of chi-square, HYBRID and RMSE obtained are small, except the Fritz–Schlunder isotherm onto CAH1, this indicate that the adsorption of phenol is favorable onto CAH1, CAH2 and CAMH.

Keywords: Adsorption; Error analysis; Isotherm; Non-linear regression

1. Introduction

Phenol and phenolic derivatives are organic chemicals that appear very frequently in wastewater from almost all heavy chemicals, petrochemical and oil refining industries [1]. Large amounts of waste water are usually generated during the manufacture and processing of organic chemicals. The concentration of these organic materials in wastewater usually exceeds the level for safe discharge into the environment. Phenol and its derivatives are toxic and carcinogenic in nature and are among the primary environmental pollutants [2].

Various methods had been developed for phenol removal from water and wastewater to reduce the risks to the environment, including: physical, chemical and biological [3]. Adsorption, one of the physical methods is generally considered to be an effective method for quickly lowering the concentrations of organic molecules in an effluent [4]. Adsorption was found to be superior to other techniques in terms of its low cost, ease of operation and simplicity of design [5]. Adsorption is defined as a process where a solute is removed from a liquid phase through contact with a solid adsorbent which has a special affinity toward that particular solute. Equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms and characterized by certain constants, the values of which provide information on adsorbent capacity and affinity for some kind of adsorbate. In addition, the quality of the fit up experimental results indicates whether adsorption is monolayer or multilayer,

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on a homogeneous or heterogeneous surface [6]. Isotherm equations do not consider which adsorption mechanisms, such as external mass transfer of solute, intraparticle diffusion or adsorption at sites, are decisive in the adsorption process, but can be used for design of batch adsorption systems [7].

Linear regression has been frequently used to determine the most fitted models and method of least squares has been frequently used for finding the parameters of the models [8–11]. For linear regression to be employed in order to determine isotherm parameters and adsorption capacity, it is first required to convert the non-linear isotherms to linear ones. After drawing a linear regression line, the isotherm parameters are calculated from slope and intercept of the line. The least square method is used to determine isotherm parameters [12–14]. The isotherm model with the best fit for adsorption data is selected on the basis of the determination of the coefficient of determination (R^2) value [15,16]. The most important disadvantage of linear regression is non-fixed error distribution [12,17]. Since the non-linear form of the isotherm is converted to linear form, error structure and distribution are altered and this can influence error variance and normal assumptions of standard least square [13,14]. In addition, in case of isotherms with more than two parameters, we cannot calculate the value of the unknown parameters using linear regression and graphical method [12].

In recent years, non-linear regression in conjunction with error analysis as an alternative to linear regression has been employed to make a model in adsorption studies [12,14]. Several error analysis methods such as coefficient of determination (R^2) [18], non-linear chi-square test error function (χ^2) [19], residual root mean square error (RMSE) [19], hybrid fractional error function (HYBRID) [18,19], have been introduced for non-linear analysis.

The present work describes the investigation of non-linear regressions in the adsorption characteristics of phenol to activated carbon. Three and four parameters isotherms were considered for analyzing the experimental data.

2. Materials and method

Rice and coffee husks were used as raw materials to produce activated carbons by chemical activation with phosphoric acid followed by carbonization. The activated carbon (CAH1) was obtained by carbonizing the activated rice husks through heating at 500°C for 1 h at 5°C/min. CAH2 was obtained by carbonizing rice husks at 450°C at a heating rate of 5°C/min, while CAMH was obtained by carbonizing a mixture 12 g rice husk and 8 g coffee husk at 500°C [20]. The activated carbons were characterized to have the surface functional groups by Fourier transform infrared (FTIR) spectroscopy, pH at zero point charge (pH_{PZC}), bulk density, moisture content, pH and iodine number.

Adsorption experiments were carried out by mechanical agitation at room temperature. For each run, 20 mL of phenol of known initial concentration (between 50 and 110 mg/L) was treated with a known weight of activated carbon. After agitation for an appropriate length of time, the solution was filtered, and the filtrate analyzed to obtain the concentration of residual phenol using a UV/Vis spectrophotometer (model 6715, Jenway, UK). The amount (q_e) of phenol adsorbed was calculated using Eq. (1):

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

where C_o is the initial concentration of phenol, C_e is its equilibrium concentration, V is the volume of the solution, and m is the mass of the absorbent.

2.1. Adsorpion isotherms

2.1.1. Three-parameter isotherms

2.1.1.1. Redlich–Peterson isotherm The Redlich–Peterson isotherm (Eq. (2)) is a mixture of the Langmuir and Freundlich isotherms. The numerator is from the Langmuir isotherm and has the benefit of approaching the Henry region at infinite dilution [21]. This isotherm model is an empirical isotherm incorporating three parameters. It combines elements from both Langmuir and Freundlich equations; therefore the mechanism of adsorption is a combination and does not follow ideal monolayer adsorption [22].

$$q_e = \frac{AC_e}{1 + BC_e^{\beta}} \tag{2}$$

where A is the Redlich–Peterson isotherm constant (L g^{-1}), B is a constant (L mg^{-1}), β is an exponent that lies between 0 and 1, C_e is equilibrium liquid-phase concentration of the adsorbate (mg L^{-1}), and q_e is equilibrium adsorbate loading on the adsorbent (mg g^{-1}).

2.1.1.2. Sips isotherm Sips isotherm is a combination of the Langmuir and Freundlich isotherms and is given by Eq. (3) [23]:

$$q_e = \frac{q_{ms}K_sC_e^{\beta_s}}{1 + K_sC_e^{\beta_s}} \tag{3}$$

where K_s is the Sips isotherm model constant (L mg^{-1}), β_s is the Sips isotherm exponent, q_{ms} (mg g^{-1}) is the maximum monolayer adsorption by Sips isotherm.

2.1.1.3. Toth isotherm The Toth isotherm is another empirical modification of the Langmuir equation with the aim of reducing the error between experimental data and predicted values of equilibrium data [24]. This model is most useful in describing heterogeneous adsorption systems which satisfies both low and high end boundary of adsorbate concentration [25]. The Toth isotherm model is expressed as in Eq. (4) [25] as follows:

$$q_e = \frac{q_mK_LC_e}{[1 + (K_LC_e)^n]^{1/n}} \tag{4}$$

where K_L is the Toth equilibrium constant, q_m (mg g^{-1}) is the maximum monolayer adsorption capacity predicted by Toth isotherm and n is the Toth model exponent.

2.1.1.4. Langmuir–Freundlich isotherm Langmuir–Freundlich isotherm includes the knowledge of adsorption heterogeneous surfaces. It describes the distribution of adsorption energy onto heterogeneous surface of the adsorbent [26]. Langmuir–Freundlich isotherm can be expressed as in Eq. (5) as follows:

$$q_e = \frac{q_{mLF}(K_{LF}C_e)^{M_{LF}}}{1+(K_{LF}C_e)^{M_{LF}}} \tag{5}$$

where q_{mLF} is the Langmuir–Freundlich maximum adsorption capacity (mg g^{-1}), K_{LF} is the equilibrium constant for heterogeneous solid, and M_{LF} is heterogeneous parameter and it lies between 0 and 1. These parameters can be obtained using the non-linear regression techniques [27].

2.2. Four-parameter isotherms

2.2.1. Fritz–Schlunder isotherm

Fritz and Schlunder derived an empirical equation (Eq. (6)) which can fit a wide range of experimental results because of the large number of coefficients in the isotherm [28].

$$q_e = \frac{q_{mFS}K_{FS}C_e}{1+q_m C_e^{M_{FS}}} \tag{6}$$

where q_{mFS} is the Fritz–Schlunder maximum adsorption capacity (mg g^{-1}), K_{FS} is the Fritz–Schlunder equilibrium constant (L mg^{-1}), and M_{FS} is the Fritz–Schlunder model exponent.

2.2.2. Baudu isotherm

Bauder observed that the estimation of the Langmuir coefficients, b and q_{ml}, by measurement of tangents at different equilibrium concentrations shows that they are not constants in a broad range [29]. This isotherm model is expressed as in Eq. (7) as follows

$$q_e = \frac{q_m b_0 C_e^{1+x+y}}{1+b_0 C_e^{1+x}} \tag{7}$$

where q_m is the Baudu maximum adsorption capacity (mg g^{-1}), b_0 is equilibrium constant, x and y are Baudu parameters.

2.2.3. Marczewski–Jaroniec isotherm

The Marczewski–Jaroniec isotherm (Eq. (8)) is also known as the four-parameter general Langmuir equation [30]. It is recommended on the basis of the supposition of local Langmuir isotherm and adsorption energies distribution in the active sites on the adsorbent [31].

$$q_e = q_{MMJ}\left(\frac{(K_{MJ}C_e)^{n_{MJ}}}{1+(K_{MJ}C_e)^{n_{MJ}}}\right)^{\frac{M_{Mj}}{n_{MJ}}} \tag{8}$$

where n_{MJ} and M_{Mj} are parameters that characterize the heterogeneity of the adsorbent surface, M_{Mj} describes the spreading of the distribution in the path of higher adsorption energy, and n_{Mj} describes the spreading in the path of lesser adsorption energies.

2.3. Goodness of fit

Goodness of fit is an essentially important parameter that estimates how well the curve fits the experimental data [32]. The following parameters are measured and they judge the goodness of fit:

The coefficient of determination, R^2, is practical as it gives the proportion of the variance of one variable that is predictable from the other variable. It is a measure that allows verify how certain one can be in making predictions from a certain model. Eq. (9) is used to determine R^2 [33] as follows:

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(q_{e,\mathrm{exp}} - q_{e,\mathrm{cal}})^2}{\sum_{i=1}^{n}(q_{e,\mathrm{cal}} - \overline{q_{e,\mathrm{means}}})^2} \tag{9}$$

The traditional methods of determining the isotherm parameters by linear regression appear to give a good fit to experimental data. However, R^2 is based on the non-linear forms of the isotherm equations, but does not represent the errors in the isotherm curves. Non-linear error functions such as the residual RMSE, the chi-square test error function (χ^2) [34] and Hybrid fractional error function (HYBRID) are used to judge the equilibrium model with the optimal magnitude. Their standard equations are as follows in Eqs. (10)–(12) [35].

$$\chi^2 = \sum_{i=1}^{n}\frac{(q_{e,\mathrm{exp}} - q_{e,\mathrm{cal}})^2}{q_{e,\mathrm{cal}}} \tag{10}$$

$$\mathrm{RMSE} = \sqrt{\frac{1}{n-2}\sum_{i=1}^{n}(q_{e,\mathrm{exp}} - q_{e,\mathrm{cal}})^2} \tag{11}$$

and

$$\mathrm{HYBRID} = \frac{100}{n-p}\sum_{i=1}^{n}\frac{(q_{e,\mathrm{exp}} - q_{e,\mathrm{cal}})^2}{q_{e,\mathrm{exp}}} \tag{12}$$

where the subscripts ‘exp’ and ‘cal’ show the experimental and calculated values, n is the number of observations in the experimental isotherm and p is the number of parameters.

3. Results and discussion

The properties of activated carbon (CAH1, CAH2 and CAMH) obtained from the characterization are presented in Table 1. The American Water Works Association has set 250 kg m^{-3} as the lower limit on the bulk density for activated carbon [36]. The values of moisture contents are greater than those of commercial activated carbons, which are normally lower than 5% [37]. The minimum iodine number recommended by the American Water Works Association for activated carbon to be used in removal of low molecular weight compounds is

Table 1
Properties of CAH1, CAH2 and CAMH

Parameter	CAH1	CAH2	CAMH
Moisture content (%)	8.86	6.93	8.91
pH	5.20	6.30	3.72
pH_{PZC}	3.0	3.45	2.30
Bulk density (kg m^{-3})	802	826	744
Iodine number (mg g^{-1})	476.25	495.30	590.55

500 mg/g. The iodine number obtained in this work is within this range (Table 1). The lower values of pH and pH_{PZC} show that all the three adsorbents have more acid groups on their surfaces.

Fig. 1 presents the FTIR spectra of the different rice husk activated carbons. The broad absorption bond at 3500–3300 cm^{-1} with a maximum at around 3380 cm^{-1} is characteristic of O-H vibrations of the carbonyls and phenols in the raw material. The 796 and 787 cm^{-1} bands are due to the mode of deformation vibration mode of the C–H plane in differently substituted aromatic rings. The small band around 1,698 cm^{-1} can be attributed to the C=O elongation vibrations of ketones, aldehydes, lactones or carboxyl groups. The spectra of the prepared carbons also show a strong band at 1,600–1,580 cm^{-1} due to the vibrations of the C=C bonds in the aromatic rings. Since the absorption bands of the oxygen and phosphorus compounds overlap in this region, interpretation is difficult. The band around 143 cm^{-1} gives an idea of the relative abundance of the CH_2 and CH_3 groups. The small band at 1,080–1,070 cm^{-1} is attributed to the P+ -O ionized chemical bond in the acid phosphate esters [38] and the symmetric vibrations in the P–O–P (polyphosphate) chains [39].

The simplest method for determination of the isotherm constants for two parameter isotherms is to linearize the model equation and then apply linear regression. Due to the inherent bias from linearization, alternative isotherm parameter sets were determined by non-linear regression. This provides a mathematically rigorous method for determining the isotherm parameters by using the original form of the isotherm parameters [34]. The non-linear approach of three- and four-parameter isotherm models using the four error functions detailed above was adopted, by using a trial and error procedure to fit the data and to determine the isotherm parameters, through the application of the Solver Add-in with Microsoft's spread sheet.

3.1. Three-parameter models

The abilities of the three-parameter equations, Redlich–Peterson, Sips, Toth and Langmuir–Freundlich isotherms, to model the equilibrium adsorption data were examined and presented in Figs. 2–4. Table 2 presents the calculated parameters of the adsorption isotherms and their characterizations obtained using the non-linear fitting analysis. As can be seen from Table 1, the coefficient of determinations for all models are very good (>0.96). Thus, the values of the chi-square test will determine the best model. Among the tested three-parameter equations, the best representation of the experimental results of the adsorption isotherms is obtained using the Langmuir–Freundlich model.

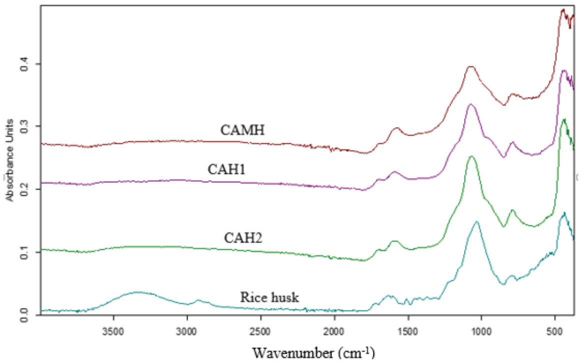

Fig. 1. FTIR spectra of the different rice husk activated carbons.

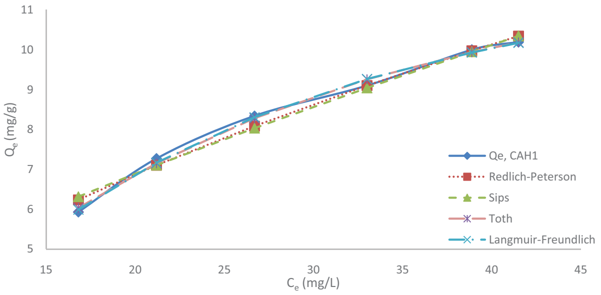

Fig. 2. Comparison of three-parameter isotherm model with experimental data onto CAH1.

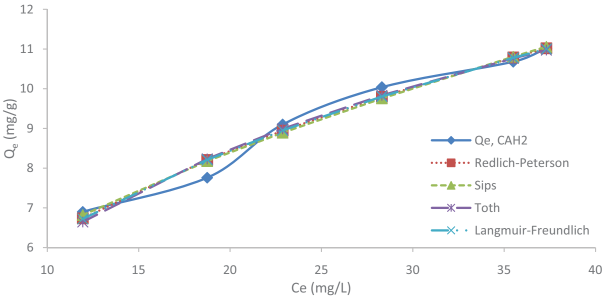

Fig. 3. Comparison of three-parameter isotherm model with experimental data onto CAH2.

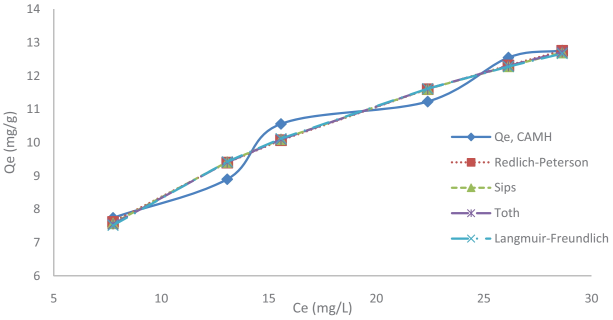

Fig. 4. Comparison of three-parameter isotherm model with experimental data onto CAMH.

Table 2
Optimum isotherm parameters and their statistical comparison values for three-parameter models

No	Models	Constants	Values	R^2	RMSE	χ^2	HYBRID
CAH1							
1	Redlich–Peterson	A (L g^{-1})	7.4042	0.9840	0.2293	0.0293	0.9908
		B (L mg^{-1})	5.1911				
		β	0.4591				
2	Sips	q_{ms} (mg g^{-1})	20.4851	0.9469	0.1677	0.0155	0.5170
		k_s (L mg^{-1})	0.0292				
		β_s	0.9469				
3	Toth	Q (mg g^{-1})	12.6758	0.9957	0.1196	0.0076	0.2431
		K_L	0.0320				
		n	2.0338				
4	Langmuir–Freundlich	q_{mLF} (mg g^{-1})	13.2110	0.9964	0.1101	0.0058	0.1929
		K_{LF}	0.0527				
		M_{LF}	1.5433				
CAH2							
1	Redlich–Peterson	A (L g^{-1})	8.5357	0.9758	0.2835	0.0381	1.3091
		B (L mg^{-1})	3.2145				
		β	0.5971				
2	Sips	q_{ms} (mg g^{-1})	22.6044	0.9728	0.3014	0.0449	1.5381
		k_s (L mg^{-1})	0.0706				
		β_s	0.7149				
3	Toth	Q (mg g^{-1})	35.6543	0.9738	0.2970	0.0435	1.4909
		K_L	0.0853				
		n	0.4103				
4	Langmuir–Freundlich	q_{mLF} (mg g^{-1})	59.5674	0.9757	0.2842	0.0386	1.3249
		K_{LF}	0.0014				
		M_{LF}	0.5068				
CAMH							
1	Redlich–Peterson	A (L g^{-1})	13.1143	0.9637	0.4172	0.0685	2.3072
		B (L mg^{-1})	3.3348				
		β	0.6389				
2	Sips	q_{ms} (mg g^{-1})	26.2091	0.9605	0.4376	0.0762	2.5829
		k_s (L mg^{-1})	0.1062				
		β_s	0.6488				
3	Toth	Q (mg g^{-1})	83.7902	0.9627	0.4250	0.0716	2.4136
		K_L	0.3786				
		n	0.2382				
4	Langmuir–Freundlich	q_{mLF} (mg g^{-1})	27.6769	0.9607	0.4350	0.0752	2.5415
		K_{LF}	0.0267				
		M_{LF}	0.6259				

The values of the maximum adsorption capacity obtained using the Langmuir–Freundlich equation are higher than those calculated by the Langmuir model which are 16.38 and 17.54, respectively, for CAH2 and CAMH and lower than that of CAH1 19.23 mg g^{-1} [40]. The values of β, *n* and M_{LF} obtained using Redlich–Peterson, Toth and Langmuir–Freundlich isotherms have a difference of 1, which means that the adsorption of phenol by this adsorbent cannot be reduced to the Langmuir isotherm. The high values of R^2 obtained indicate the adequacy of all these models for the adsorption of phenol in aqueous solution. These models are thus linked to the adsorption of materials having a heterogeneous surface. According to Ho et al. [6], the small values of RMSE and χ^2 indicate a better model fitting. The fitness of the models to explain the equilibrium data was in the order: Langmuir–Freundlich > Toth > Redlich–Peterson > Sips for the adsorption onto CAH1 activated carbon. On CAH2 activated carbon, the values of RMSE and χ^2 are similar and make it difficult to have a unique equilibrium. The same result is observed for the CAMH absorbent. In this study, all the values of RMSE, HYBRID and chi-square (χ^2) are very small and indicate a favorable adsorption of phenol onto the different adsorbents.

3.2. Four-parameter models

The adsorption data were analyzed according to the non-linear form of the four-parameter isotherm models. An appropriate fitting of the experimental results of adsorption isotherms was obtained using the four-parameter model of Fritz–Schlunder, Baudu and Marczewski–Jaroniec (Figs. 5–7) onto different activated carbons. Table 3 presents the calculated parameters of the adsorption isotherms and their characterizations obtained using the non-linear fitting analysis. A high R^2 value is obtained using Marczewski–Jaroniec model with 0.9837 and the lowest value is 0.8177 from Fritz-Schlunder model. All these values concern only CAH1 activated carbon. For the adsorbent CAH2 and CAMH, the values of R^2 are between 0.96 and 0.98. According to the values of RMSE and χ^2, the Fritz–Schlunder model presents poor results, these results and the value of R^2 conclude that this model cannot describe the adsorption of phenol onto CAH1. An excellent description of the experimental results is obtained using the Marczewski–Jaroniec model. On the basis of the average percentage error values (Table 3), the equation of Marczewski–Jaroniec seems better than that of Fritz–Schlunder and Baudu. The values of the maximum adsorption capacity obtained using all the three four-parameter isotherms are lower than those calculated from the Langmuir and Elovich models and lower than the theoretical values.

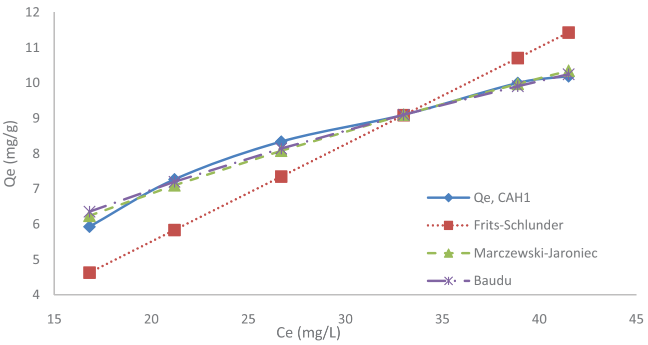

Fig. 5. Comparison of four-parameter isotherm model with experimental data onto CAH1.

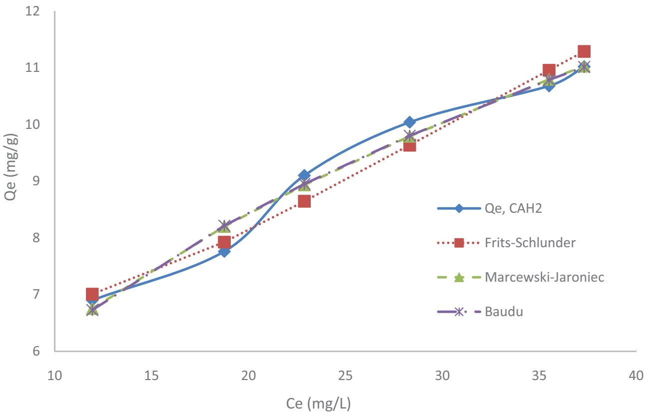

Fig. 6. Comparison of four-parameter isotherm model with experimental data onto CAH2.

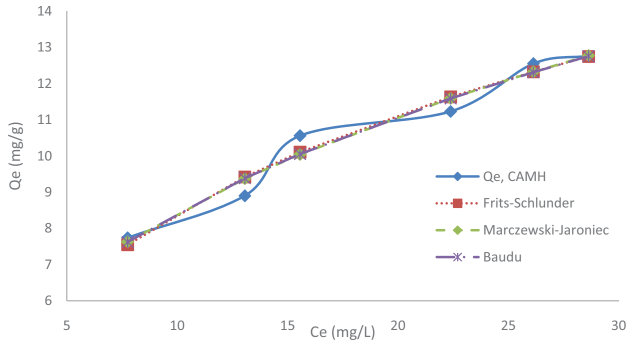

Fig. 7. Comparison of four-parameter isotherm model with experimental data onto CAMH.

4. Conclusion

The equilibrium adsorption of phenol onto activated carbon was explained using non-linear methods with three and four parameters. Often, non-linear methods are used for estimating the adsorption characteristics of certain adsorption systems, due to simplicity in estimation. For three parameter models, the Langmuir–Freundlich isotherm was found to be the best model, with $R^2 = 0.9964$, $\chi^2 = 0.0058$ and RMSE = 0.1101 onto CAH1 activated carbon. For four parameters, Marczewski–Jaroniec was the best model with $R^2 = 0.9837$, $\chi^2 = 0.0300$ and RMSE = 0.2321 onto CAH1. The present investigations showed that non-linear method is a better way to obtain the isotherm parameters. All the values of chi-square, HYBRID and RMSE obtained are small, this indicate that without the Fritz–Schlunder isotherm onto CAH1, the adsorption of phenol is favorable onto CAH1, CAH2 and CAMH, and that all theses activated carbons have a heterogeneous surface.

Table 3
Optimum isotherm parameters and their statistical comparison values for four-parameter models

No	Models	Constants	Values	R^2	RMSE	χ^2	HYBRID
CAH1							
1	Fritz–Schlunder	q_{mFS} (mg g^{-1})	6.9769	0.8177	1.3004	1.0320	44.3142
		K_{FS} (L mg^{-1})	0.0395				
		q_m	–0.0194				
		M_{FS}	–0.6255				
2	Baudu	q_m (mg g^{-1})	1.6404	0.9800	0.2430	0.0351	1.8503
		b_0	0.9886				
		x	0.0794				
		y	0.4966				
3	Marczewski–Jaroniec	q_{MMJ} (mg g^{-1})	2.5534	0.9837	0.2321	0.0300	1.5220
		K_{MJ}	0.2484				
		n_{MJ}	4.1511				
		M_{MJ}	0.5596				
CAH2							
1	Fritz–Schlunder	q_{mFS} (mg g^{-1})	2.0711	0.9612	0.3733	0.0593	2.8931
		K_{FS} (L mg^{-1})	0.0356				
		q_m	–1.1993				
		M_{FS}	–0.1275				
2	Baudu	q_m (mg g^{-1})	2.4913	0.9755	0.2858	0.0389	2.0056
		b_0	0.9779				
		x	0.4275				
		y	0.4123				
3	Marczewski–Jaroniec	q_{MMJ} (mg g^{-1})	3.4467	0.9761	0.2819	0.0375	1.9261
		K_{MJ}	0.3463				
		n_{MJ}	4.8951				
		M_{MJ}	0.4307				
CAMH							
1	Fritz–Schlunder	q_{mFS} (mg g^{-1})	5.4156	0.9630	0.4257	0.0719	3.6521
		K_{FS} (L mg^{-1})	0.8823				
		q_m	0.9345				
		M_{FS}	0.6985				
2	Baudu	q_m (mg g^{-1})	3.4716	0.9640	0.4156	0.0681	3.4221
		b_0	2.5496				
		x	0.8956				
		y	0.3882				
3	Marczewski–Jaroniec	q_{MMJ} (mg g^{-1})	3.3127	0.9645	0.4141	0.0676	3.3900
		K_{MJ}	0.8890				
		n_{MJ}	3.7451				
		M_{MJ}	0.3944				

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