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# Adsorption of Phenolic Compounds from Water on Activated Carbon: Prediction of Multicomponent Equilibrium Isotherms using Single-Component Data

ANABELA LEITÃO* AND ROSALINA SERRÃO
*Laboratory of Separation, Reaction and Environmental Engineering, Faculty of Engineering, Agostinho Neto University, P.O. Box 5532, Luanda, Angola*
aleitao@mstelcom.com

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**Abstract.** Batch-type experiments were carried out to obtain equilibrium isotherms for the adsorption of phenol and $m$-cresol in aqueous solutions on activated carbon. Single solute systems, at 20 and 40°C, were tested for Langmuir, Freundlich and Sips adsorption isotherms in the range of concentrations up to 200 mg/L. Equilibrium data were more closely followed by the Freundlich and Sips equations for all cases. Adsorption isotherms for bisolute systems at 20°C, with two different initial concentrations of phenol and $m$-cresol, were predicted solely on the basis of single solute equilibrium parameters by using the equations of Butler and Ockrent and the IAS theory. The best agreement with the experimental loading values was afforded with the IAS theory based on Sips isotherm for pure compounds. However, this theory is found to be not able to predict with success the binary isotherms in this work where significant displacement of one solute by the other is observed. Chemical interactions in the adsorbed phase, estimated by a modified Butler–Ockrent model, can be responsible for this lack of success of the conventional IAS theory. The predictions based on the IAS theory are compared with the results of some empirical models.

**Keywords:** binary adsorption, phenol and $m$-cresol, prediction of mixture isotherms, IAS theory

## Introduction

Increasing stringent legislation on the purity of drinking water has created a growing interest in the decontamination of water, waste-water and polluted effluents by adsorption processes.

The treatment of waste-waters by activated carbon sometimes involves a multitude of organic pollutants, like phenolic compounds, competing for available adsorption sites on the carbon surface. For multicomponent systems experimental equilibrium data are difficult to obtain. Frequently, the practical way of estimating multicomponent adsorption equilibria is to predict mixture isotherms solely on the basis of single component isotherms. Several methods have been proposed and reported elsewhere (Butler and Ockrent, 1936; Myers and Prausnitz, 1965; Radke and Prausnitz, 1972; Jain and Snoeyink, 1973; Seidel et al., 1986; Srivastava and Tyagi, 1995; Khan et al., 1997; Do, 1998).

Simple and restrictive models were derived by defining the type of equilibrium isotherm in the model. Butler and Ockrent (1936) extended the Langmuir theory to multicomponent competitive adsorption and Jain and Snoeyink (1973) developed a modified Langmuir equation to predict bisolute equilibrium data where some adsorption occurs without competition.

The Ideal Adsorbed Solution (IAS) theory was originally derived by Myers and Prausnitz (1965) using chemical thermodynamics for predicting equilibrium data for gaseous mixtures and then extended by Radke and Prausnitz (1972) for dilute aqueous solutions. This theory has been successfully used to characterize

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*To whom all correspondance should be addressed.

competitive adsorption for activated carbons (Jossens et al., 1978; Fritz and Schluender, 1981; Costa and Rodrigues, 1982; Crittenden et al., 1985).

However, methods based solely on single solute equilibrium data often fail to calculate experimental mixture equilibria accurately (Seidel and Gelbin, 1988) and so several equations, for example those of Fritz and Schluender (1974), Crittenden and Weber (1978), Sheindrof et al. (1982) and Khan et al. (1996, 1997), with additional adjustable parameters have been proposed. But, as the number of solutes in the mixture increases, the number of additional parameters to be determined from the experimental mixture equilibria becomes higher and the use of these equations becomes impracticable.

In our investigation phenol and *m*-cresol have been used to determine the adsorption isotherms for single and bisolute systems from dilute aqueous solutions on activated carbon. The major objective of the present work is to test the predictive performance of two systems of equations: those of Butler and Ockrent (1936) and the IAS theory based on different equilibrium isotherms for the single compounds (Radke and Prausnitz, 1972; Do, 1998).

## Experimental

Batch-type experiments were carried out to obtain adsorption equilibrium data for single and bisolute systems.

The adsorbent was activated carbon Nuchar WA (apparent density 0.27 g/cm$^3$, porosity 0.85 and BET surface area 800 m$^2$/g (Leitão, 1987)). This carbon was subjected to contact with boiling water under vacuum prior to use.

Purity grades of phenol and *m*-cresol (BDH) were 99.5% and 98% respectively. A 500 ppm stock solution of each solute was prepared every 2 weeks from which working solutions were then prepared by dilution with distilled water. 100 cm$^3$ of equal solutions, with the desired phenol and *m*-cresol concentrations, were fed to 250 cm$^3$ jacketed narrow neck bottles, with stoppers, kept at constant temperature (20 or 40°C) and placed in a shaker. Different amounts of activated carbon 18/20 (mesh size) were then dropped to each bottle. After 15 days, phenol and *m*-cresol liquid equilibrium concentrations in each bottle were measured by HPLC with a UV absorption detector. Before analysis by HPLC, each sample was filtered with a 0.2 $\mu$m membrane filter after previous filtration with standard filter paper to remove the activated carbon powder.

## Results and Discussion

### *Single Solute Isotherms*

Prior to the study of multicomponent adsorption, we measured the single solute adsorption isotherms of phenol and *m*-cresol, at 20 and 40°C, starting from an initial solute concentration of 200 mg/L in the aqueous phase. The experimental data were correlated by the Langmuir, Freundlich and Sips (Langmuir-Freundlich) equations in the range of concentrations up to 200 mg/L.

The best values of the unknown parameters of those equations were obtained by minimizing the sum of squares of deviations between experimental and calculated values of the solid equilibrium concentrations ($q^*$). Table 1 lists the parameter values for the three isotherms and the average relative error (*ERM*) of each equilibrium representation defined by

$$\mathrm{ERM} = \frac{100}{N}\sqrt{\sum_{i=1}^{N}\left(1 - \frac{q^*_{i,\mathrm{cal}}}{q^*_{i,\mathrm{exp}}}\right)^2} \tag{1}$$

We note from the above table that the Freundlich and Sips equations follow the experimental data more

*Table 1.* Parameters values for different isotherms for single solute adsorption.

| | | Langmuir $q^* = \frac{QbC^*}{1+bC^*}$ | | | Freundlich $q^* = KC^{*1/n}$ | | | Sips $q^* = \frac{Q(bC^*)^{1/n}}{1+(bC^*)^{1/n}}$ | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Solute | $T$ (°C) | $Q$ (mg/g) | $b$ (L/mg) | ERM (%) | $K$ (L$^{1/n}$/(g mg$^{1/n-1}$)) | $n$ | ERM (%) | $Q$ (mg/g) | $b$ (L/mg) | $n$ | ERM (%) |
| Phenol | 20 | 142.4 | 0.103 | 1.99 | 52.10 | 5.155 | 0.82 | 176.5 | 0.06515 | 1.901 | 1.10 |
| | 40 | 163.0 | 0.055 | 1.96 | 39.37 | 3.759 | 1.16 | 176.5 | 0.04709 | 1.262 | 1.35 |
| *m*-Cresol | 20 | 142.4 | 0.207 | 2.20 | 66.49 | 6.452 | 1.40 | 176.5 | 0.16063 | 2.784 | 1.80 |
| | 40 | 148.9 | 0.126 | 3.81 | 55.31 | 5.051 | 1.12 | 176.5 | 0.09128 | 1.745 | 2.00 |

closely than the Langmuir one. Additionally, the carbon affinity is higher for *m*-cresol and the Sips parameter *n* is greater than unity for both systems. Considering that the larger is this parameter the more heterogeneous is the system, it also can be seen that the system is more heterogeneous for *m*-cresol and the heterogeneity of both systems decreases as temperature increases.

The experimental and calculated equilibrium data are depicted in Fig. 1(a) and (b) at the temperatures of 20 and 40°C. The experimental data can be best fitted with the Freundlich isotherm and for the sake of clarity only the calculated data by this equation are shown.

### *Bisolute Isotherms*

Adsorption isotherms were measured at 20°C for bisolute systems with two different initial concentrations of phenol and *m*-cresol, $C_{o1} = C_{o2} = 200$ mg/L and $C_{o1} = C_{o2} = 100$ mg/L. The measured equilibrium concentrations of these compounds in the liquid phase as a function of the mass of activated carbon added to the system are displayed in Fig. 2. The experimental equilibrium concentrations in the adsorbed phase were calculated by the material balance:

$$q_{i,\exp}^* = V(C_{oi} - C_i^*)/m \tag{2}$$

where *V* is the liquid volume in each bottle and *m* is the activated carbon load expressed in dry weight. The bisolute isotherms were predicted by using the equations of Butler and Ockrent (1936) and the IAS theory (Radke and Prausnitz, 1972; Do, 1998) with single solute equilibrium parameters.

The Butler-Ockrent binary equations are:

$$q_j^* = \frac{Qb_jC_j^*}{1 + b_1C_1^* + b_2C_2^*} \quad j = 1, 2 \tag{3}$$

where *Q* is the maximum adsorbed concentration (monolayer capacity) for the two solutes.

The main assumption in the IAS theory is that the adsorption of the solute species occurs simultaneously at constant temperature and constant spreading pressure, $\pi$. At equilibrium the spreading pressure for all species is equal to the spreading pressure of the mixture, $\pi_{\text{mixture}}$. For a bisolute system, the IAS equations are given below:

$$C_j^* = x_jC_j^0(\pi) \quad j = 1, 2 \tag{4}$$

$$\sum_{j=1}^{2} x_j = 1 \tag{5}$$

$$Z = \frac{A\pi}{R_gT} = \int_0^{C_1^0} \frac{q_1^*}{C_1^*}dC_1^* = \int_0^{C_2^0} \frac{q_2^*}{C_2^*}dC_2^* \tag{6}$$

where $C_j^o(\pi)$ is the hypothetical concentration of the pure component *j* that gives the same spreading pressure, $\pi$, as that of the mixture and *Z* is the reduced spreading pressure.

Solving numerically Eqs. (4) to (6) will give the solutions for the unknowns: the hypothetical concentrations of the pure compounds, $C_j^o$, the adsorbed phase mole fractions, $x_j$, and the reduced spreading pressure, Z. Knowing the values of $x_j$ and $C_j^o$, the total amount adsorbed, $q_T$, can be calculated from the equation:

$$\frac{1}{q_T} = \sum_{j=1}^{2} \frac{x_j}{q_j^o} \tag{7}$$

where $q_j^o$ is the adsorbed amount of pure component *j* at the hypothetical concentration $C_j^o$ calculated by the appropriate isotherm.

The amount adsorbed contributed by the component *j* in the mixture at equilibrium is given by:

$$q_j^* = x_jq_T \tag{8}$$

For Langmuir, Freundlich and Sips isotherms the spreading pressure Eq. (6) can be integrated analytically and the hypothetical concentration, $C_j^o$, versus reduced spreading pressure, *Z*, is available in analytical form as shown in Table 2. Then Eqs. (4) and (5) can be combined together to yield the following equation:

$$F(Z) = \sum_{j=1}^{2} \frac{C_j^*}{C_j^0(Z)} - 1 = 0 \tag{9}$$

To solve Eq. (9) for *Z* we use the Newton-Ralphson method:

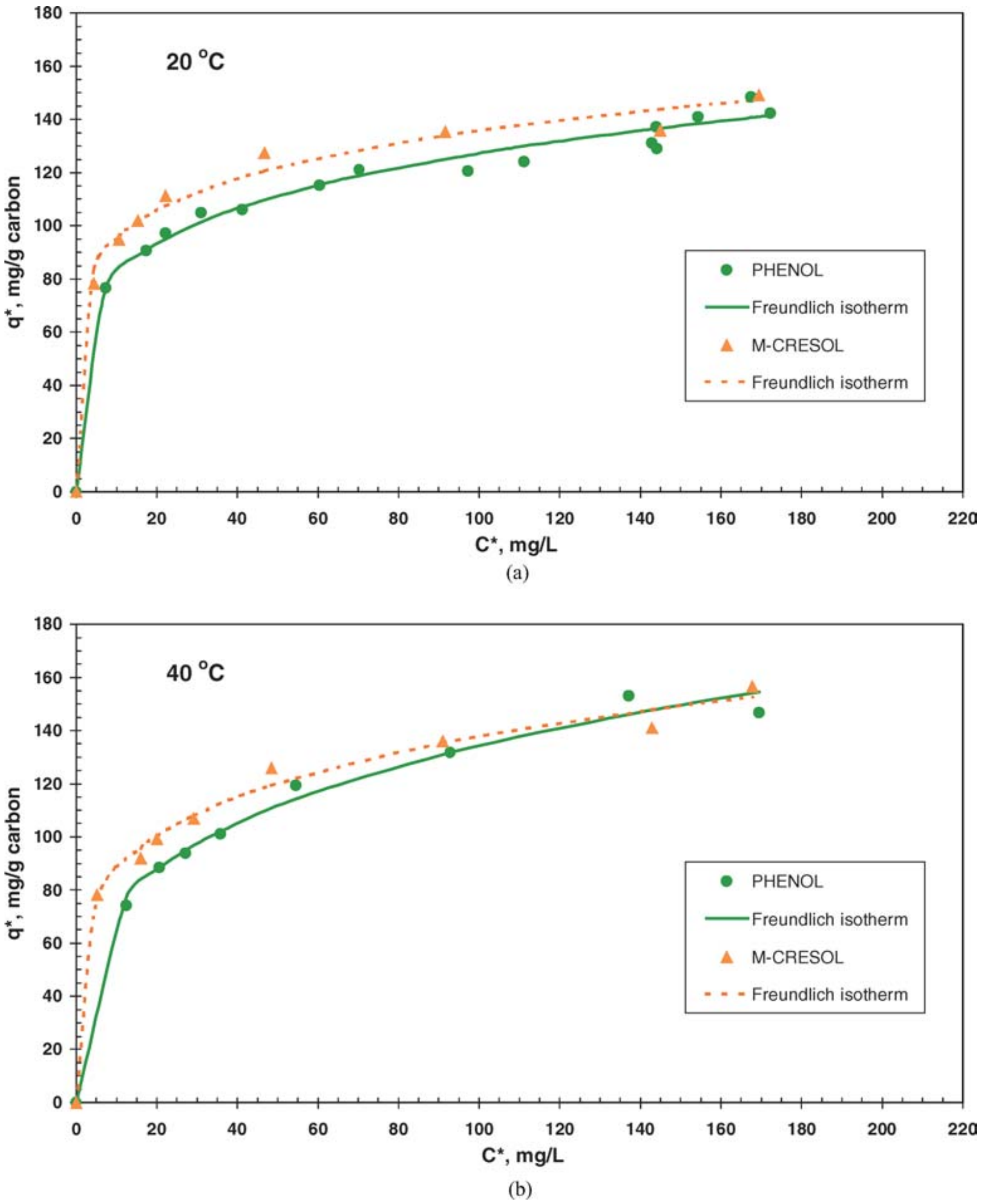
$$Z^{K+1} = Z^K - \frac{F(Z^K)}{F'(Z^K)} \tag{10}$$

where the upperscript denotes for the iteration number and *F*' = *dF*/*dZ* is given by:

$$F'(Z^K) = \left[-\sum_{j=1}^{2} \frac{C_j^*}{C_j^0(Z)q_j^0}\right]_{Z=Z^K} \tag{11}$$

*Table 2*. Hypothetical concentration ($C_j^o$) vs reduced spreading pressure ($Z$) for different isotherms.

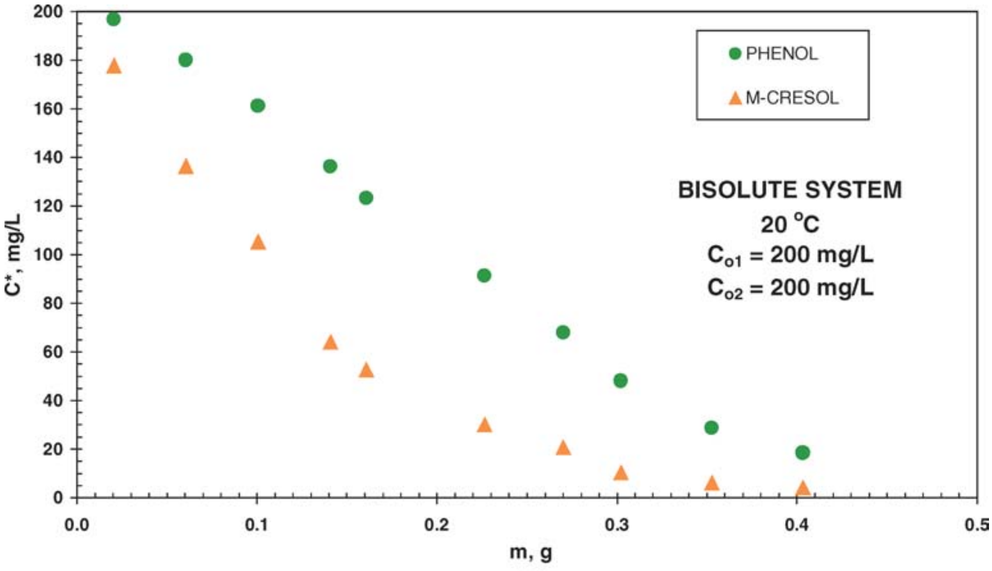
| Isotherm | Adsorbed amount of pure component $j$ | Reduced spreading pressure | Hypothetical concentration of pure component $j$ |
|---|---|---|---|
| Langmuir | $q_j^* = \frac{Q_j b_j C_j^*}{1+b_j C_j^*}$ | $Z = Q_j \ln\left(1 + b_j C_j^o\right)$ | $C_j^o = \frac{1}{b_j}\left(\exp\left(\frac{Z}{Q_j}\right) - 1\right)$ |
| Freundlich | $q_j^* = K_j C_j^{*\,1/n_j}$ | $Z = n_j K_j \left(C_j^o\right)^{1/n_j}$ | $C_j^o = \left(\frac{Z}{n_j K_j}\right)^{n_j}$ |
| Sips | $q_j^* = \frac{Q_j (b_j C_j^*)^{1/n_j}}{1+(b_j C_j^*)^{1/n_j}}$ | $Z = n_j Q_j \ln\left(1 + \left(b_j C_j^o\right)^{1/n_j}\right)$ | $C_j^o = \frac{1}{b_j}\left(\exp\left(\frac{Z}{n_j Q_j}\right) - 1\right)^{n_j}$ |



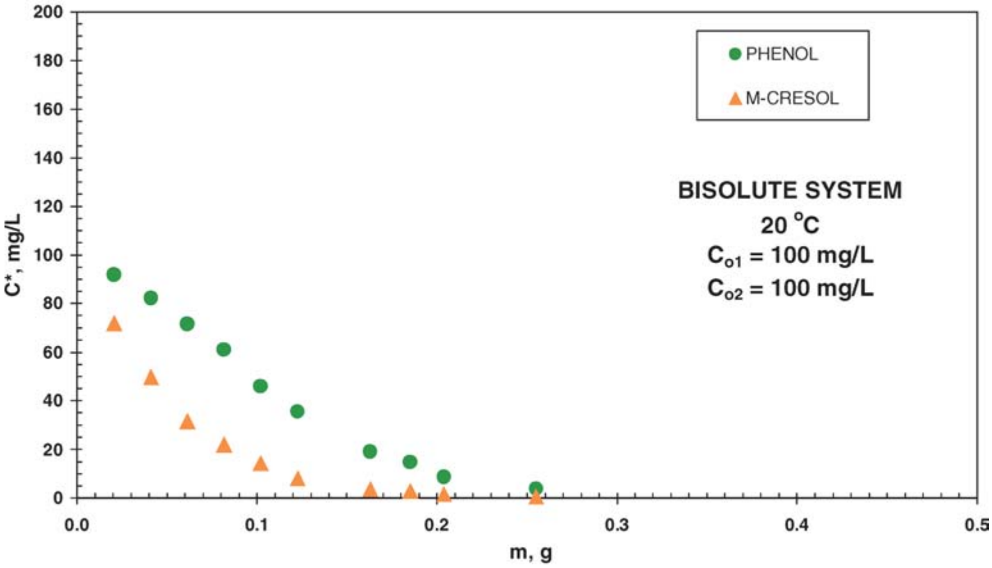
(a)

(b)

*Figure 1*. Comparison of experimental and calculated isotherms for single solute adsorption of phenol and *m*-cresol in aqueous solutions (a) $T = 20°\mathrm{C}$ and (b) $T = 40°\mathrm{C}$.



(a)



(b)

*Figure 2*. Measured equilibrium solute concentrations in the liquid phase as a function of the mass of carbon added to the bisolute system (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

Figures 3–6 show the measured and predicted isotherms for phenol and $m$-cresol in the mixture. The average relative error between experimental and calculated values of the solid equilibrium concentrations is presented in Table 3 for the models used herein. We can observe that all predictions based on the Langmuir isotherm for pure compounds give poor results with considerable systematic deviation between theory and experiment. But, the use of the Freundlich or Sips isotherm, instead of the Langmuir isotherm, for estimating the spreading pressure in the IAS theory leads to a better agreement of the predictions with the experimental loading values. The best agreement is seen in Fig. 6, where the IAS theory based on Sips isotherm for pure compounds is used. For the higher initial composition (Fig. 6(a)), this model approaches

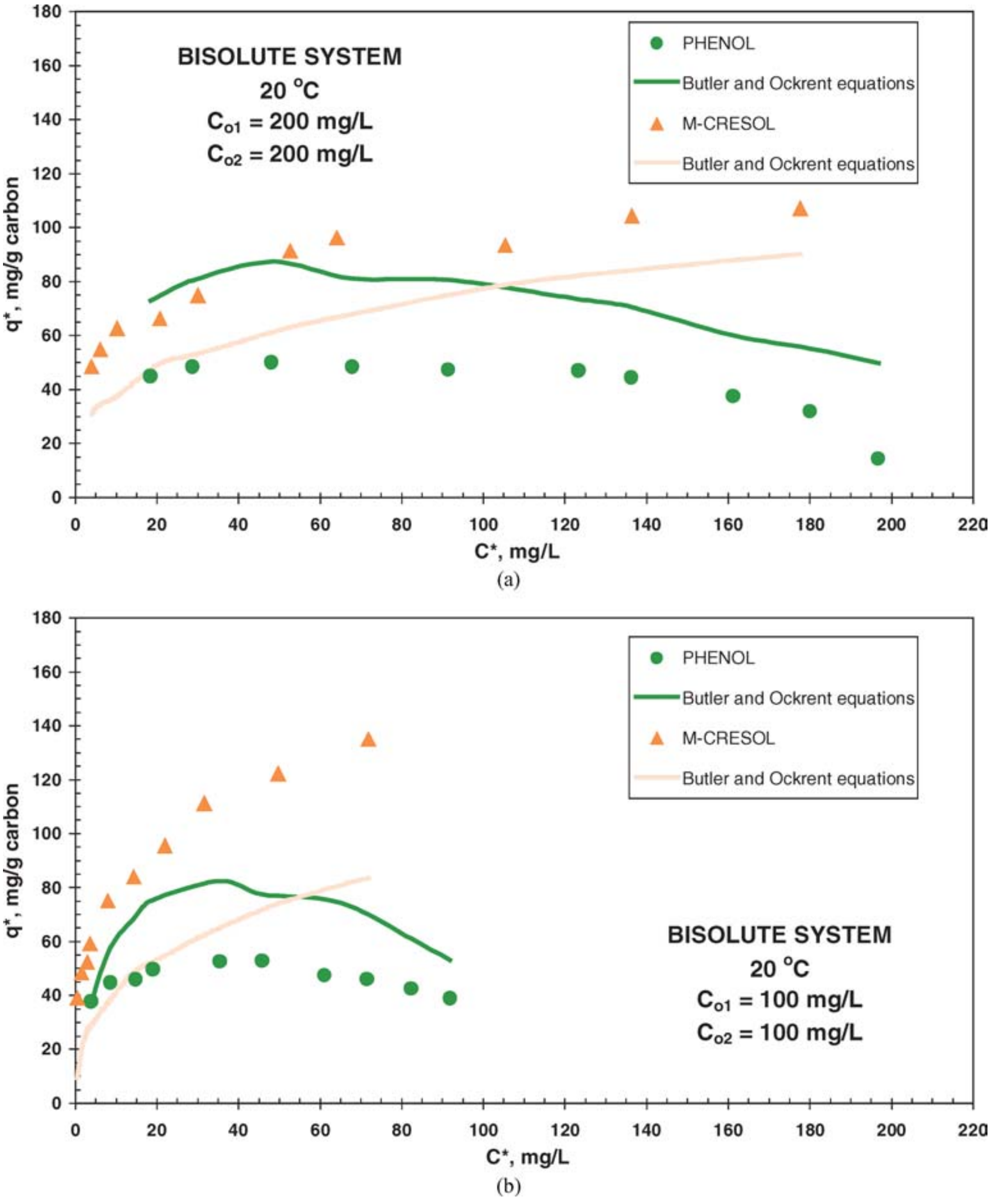
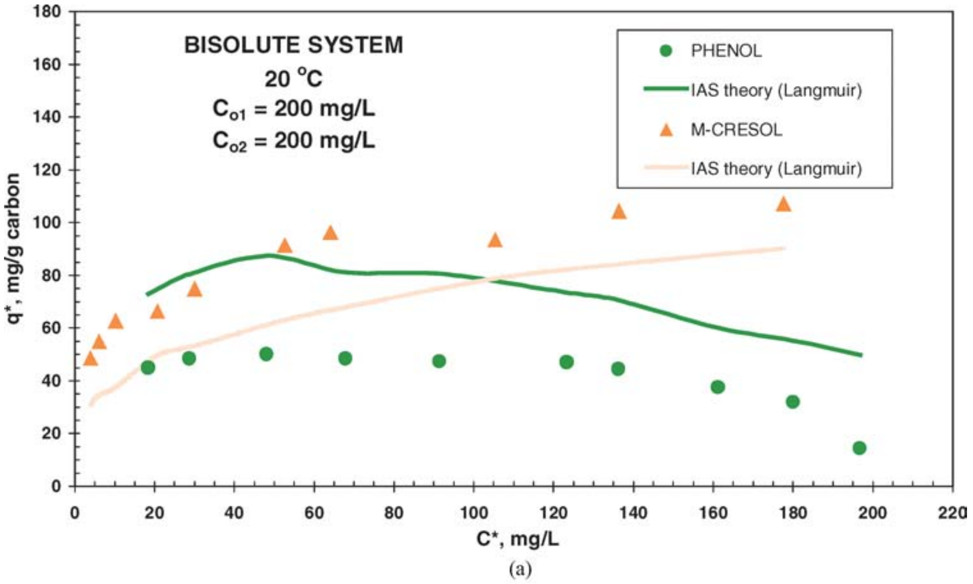


*Figure 3*. Comparison of bisolute experimental and predicted isotherms by the Butler and Ockrent equations (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

reasonably the experimental loading values of *m*-cresol and phenol, but the behaviour of the displaced phenol in the region of higher concentrations is not satisfactorily represented. For the lower initial composition (Fig. 6(b)), the behaviour of the experimental isotherms is represented satisfactorily, although a small systematic deviation between theory and experiment is still observed.

Seidel et al. (1986) and Seidel and Gelbin (1988) in their work with organic solutes and activated carbon found considerable deviations between measured and predicted isotherms by using the IAS theory. These authors concluded that chemical interactions between solutes in the adsorbed phase could be responsible for those deviations and that the IAS theory is not suitable in these cases.



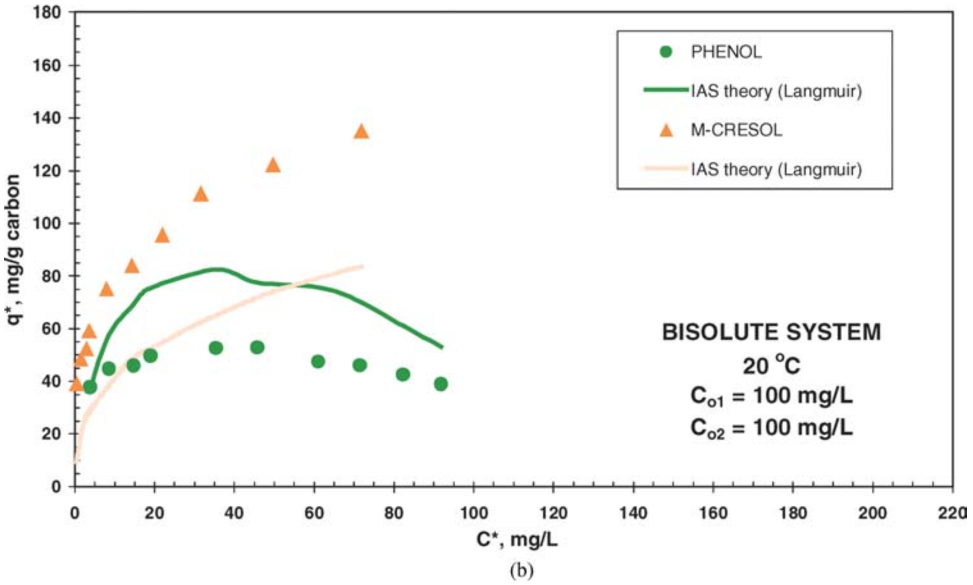
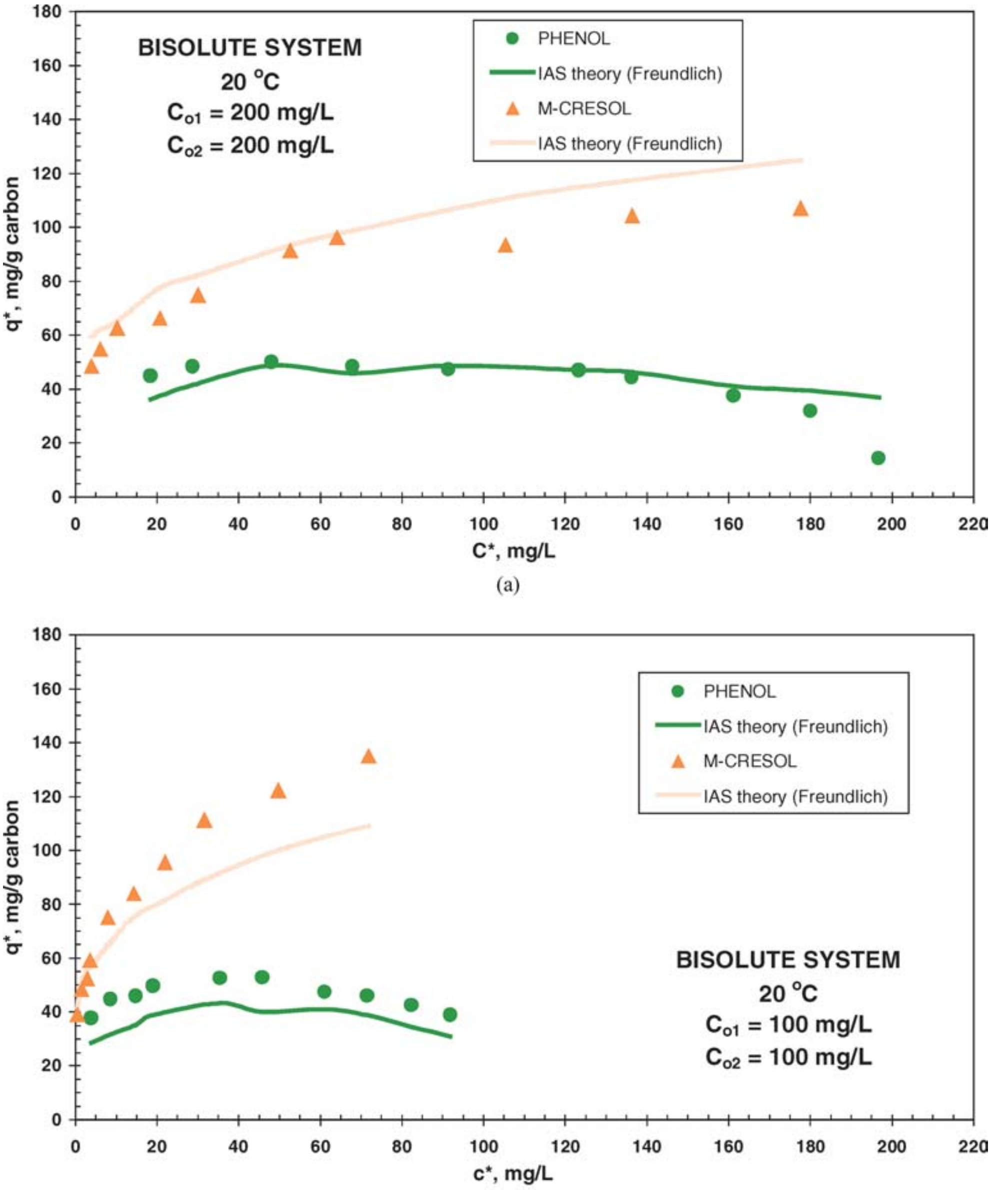


*Figure 4*. Comparison of bisolute experimental and predicted isotherms by the IAS theory based on Langmuir equation for pure compounds (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

In order to understand the role of chemical interactions in the adsorbed phase on the calculated solid equilibrium concentrations, a modified Butler-Ockrent model (Ho and McKay, 1999) was derived from the previous Eq. (3) by introducing interaction coefficients, $\eta_1$ and $\eta_2$, that are functions of the adsorbed phase concentrations:

$$q_j^* = \frac{Qb_jC_j^*/\eta_j}{1 + b_1C_1^*/\eta_1 + b_2C_2^*/\eta_2} \quad j = 1, 2 \quad (12)$$

The coefficients $\eta_1$ and $\eta_2$ were experimentally evaluated by fitting the above equations to each couple of measured loading values of phenol and *m*-cresol. The calculated isotherms in Fig. 7(a) (broken lines) show an excellent agreement with the measured ones. The fitted interaction coefficients $\eta_1$ and $\eta_2$ were correlated with the adsorbed phase mole fractions $q_1^*/q_T$ and $q_2^*/q_T$, where $q_T = q_1^* + q_2^*$. The correlations are shown in Fig. 8. These correlations for $\eta_1$ and $\eta_2$ were also used in Eqs. (12) to calculate the adsorbed phase



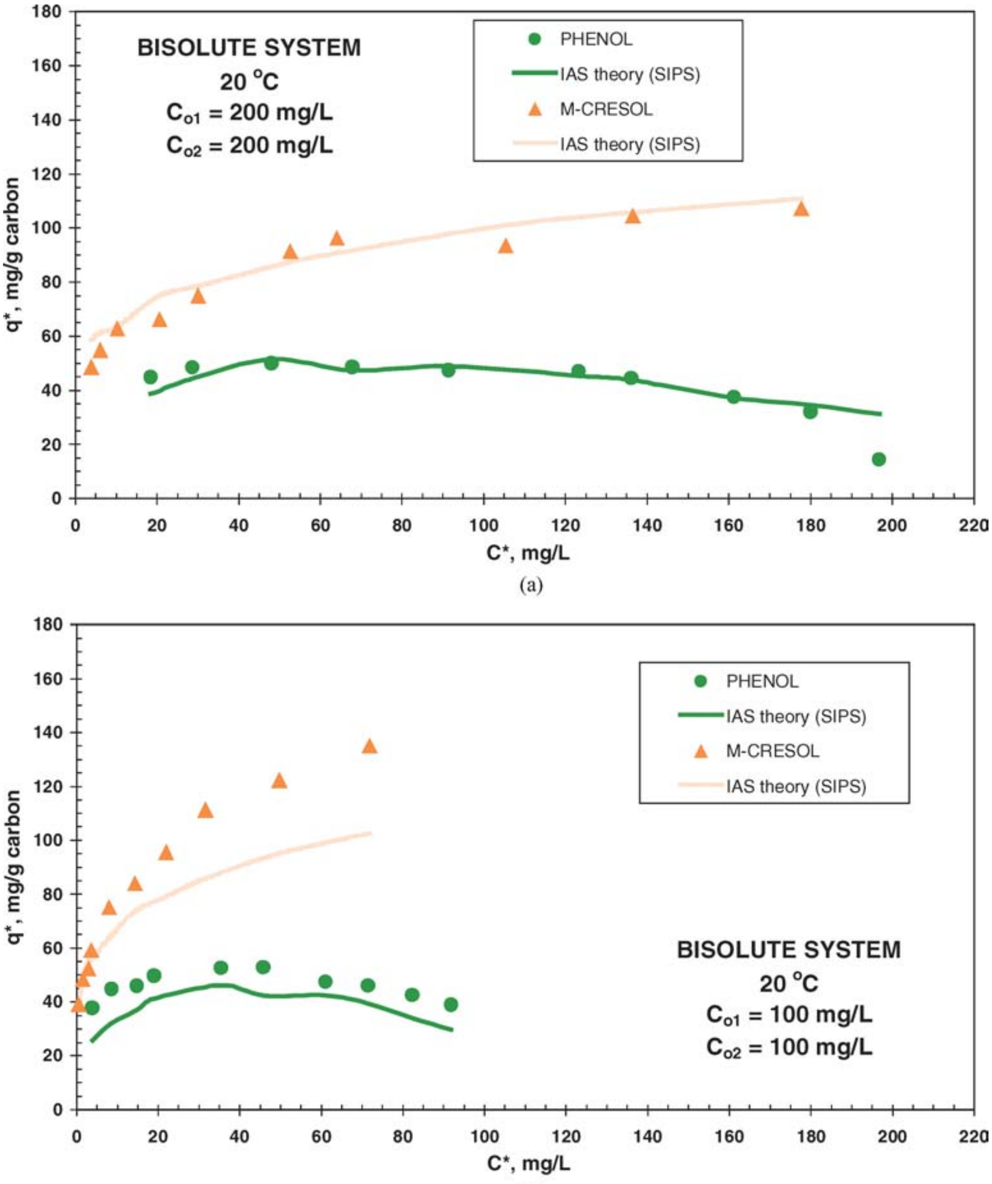
(a)

(b)

*Figure 5.* Comparison of bisolute experimental and predicted isotherms by the IAS theory based on Freundlich equation for pure compounds (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

concentrations by a numerical procedure. Fig. 7(a) displays these concentrations (solid lines). Eqs. (12), with the previous correlations found for $\eta_1$ and $\eta_2$, were also tested to predict the equilibrium data obtained from a bisolute solution with other initial composition. Fig. 7(b) shows that the agreement between experimental and predicted isotherms is not so satisfactory as the shown in Fig. 7(a). This poor agreement is due to the finding of experimental interaction coefficients different from those estimated from the previous correlations.

Some empirical models, namely those of Fritz and Schluender (1974), Sheindrof et al. (1982) and Khan et al. (1996, 1997), were also used in this investigation to calculate the solid equilibrium concentrations. The adjustable parameters of these equations

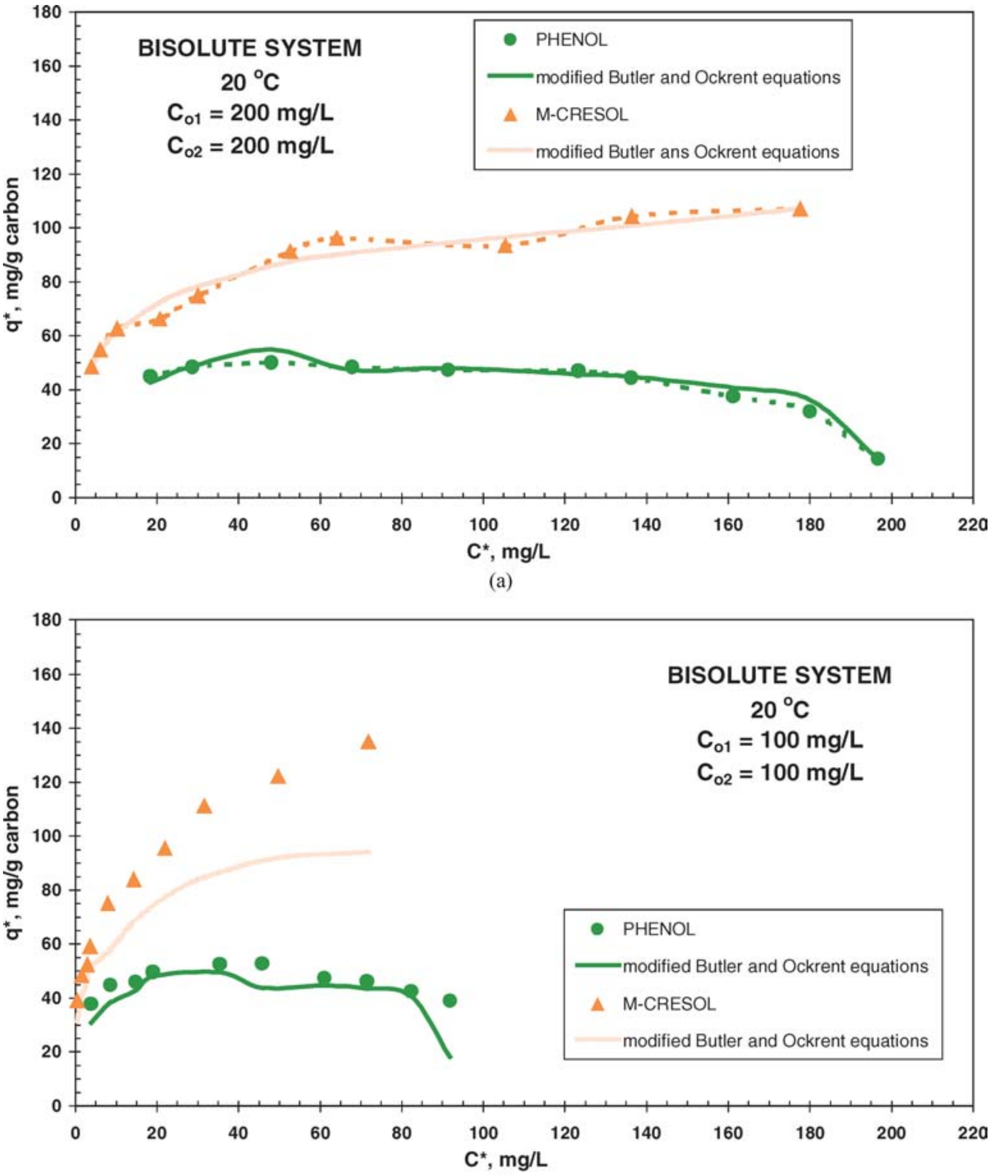


(a)

(b)

*Figure 6.* Comparison of bisolute experimental and predicted isotherms by the IAS theory based on Sips equation for pure compounds (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

were evaluated by computer programs based on a non-linear optimization technique using a least-squares method. Equilibrium measurements in the bisolute system with initial phenol and *m*-cresol concentrations of 200 mg/L were used in the evaluation of those parameters. Table 3 shows the average relative error between experimental and calculated values of the solid equilibrium concentrations. The predictions based on the IAS theory, with Sips isotherms for pure compounds, are globally poorer than the results obtained with the three tested empirical models. The Fritz and Schluender model, as shown in Fig. 9(a), leads to the best results similar to those obtained with the modified Butler and Ockrent equations. But, as can be observed in Fig. 9(b), the Fritz and Schluender model, like as shown for the modified Butler and Ockrent model, with adjustable



(a)

(b)

*Figure 7.* Comparison of bisolute experimental and calculated isotherms by the modified Butler and Ockrent equations (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

parameters determined on the basis of measurements in the system with a given initial composition in organics, is not able to produce adsorbed equilibrium concentrations with the same degree of accuracy when we start from another initial composition. Adsorption of organics from liquid solutions is complicated by the presence of several interactions between the solutes, the solutes-surface, the solvent-surface and the solutes-solvent (Yang, 2003). As some of these interactions can be dependent on the degree of the dilution of the system, we suspect that this can be the cause for the above described behaviour of the best models.

## Conclusions

A number of adsorption models were presented and used in this study to predict bisolute adsorption equilibrium data solely from single solute equilibria.

*Table 3.* Average relative error (%) between experimental and calculated values of the solid equilibrium concentrations for bisolute systems.

| | Phenol and *m*-cresol concentrations in the initial solution $C_{o1} = 200$ mg/L, $C_{o2} = 200$ mg/L | | Phenol and *m*-cresol concentrations in the initial solution $C_{o1} = 100$ mg/L, $C_{o2} = 100$ mg/L | |
|---|---|---|---|---|
| Model | Phenol | *m*-Cresol | Phenol | *m*-Cresol |
| Butler and Ockrent equations | 31.5 | 9.2 | 14.6 | 15.9 |
| IAS theory based on Langmuir isotherm for pure compounds | 31.5 | 9.2 | 14.6 | 15.9 |
| IAS theory based on Freundlich isotherm for pure compounds | 16.1 | 4.3 | 6.7 | 4.2 |
| IAS theory based on Sips isotherm for pure compounds | 12.0 | 3.0 | 6.5 | 4.8 |
| Modified Butler and Ockrent equations | 2.0 | 1.4 | 6.2 | 6.6 |
| Sheindrof et al. equations | 9.5 | 1.4 | 5.2 | 7.6 |
| Khan et al. equations | 9.0 | 1.5 | 5.6 | 9.6 |
| Fritz and Schluender equations | 1.5 | 1.4 | 4.4 | 7.9 |

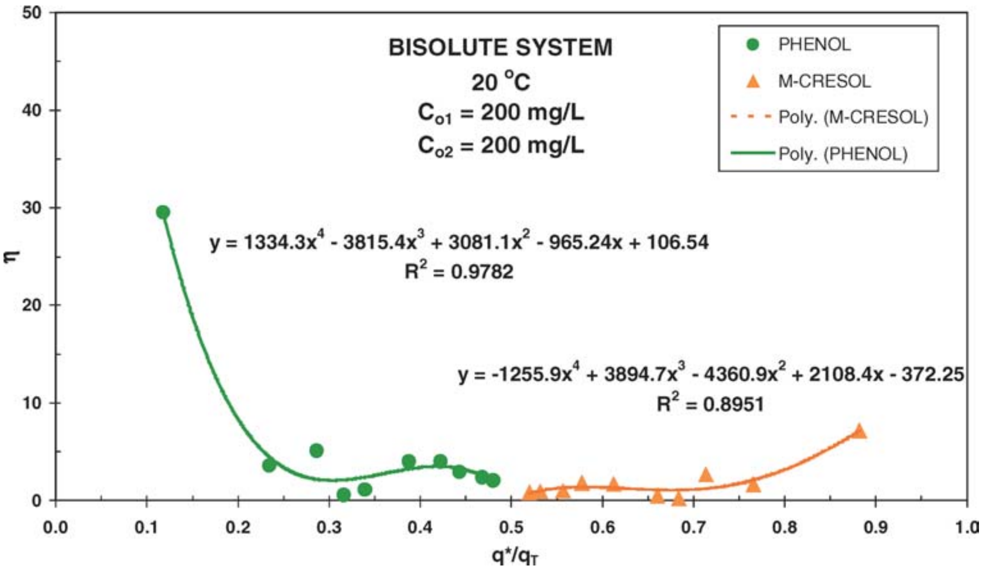


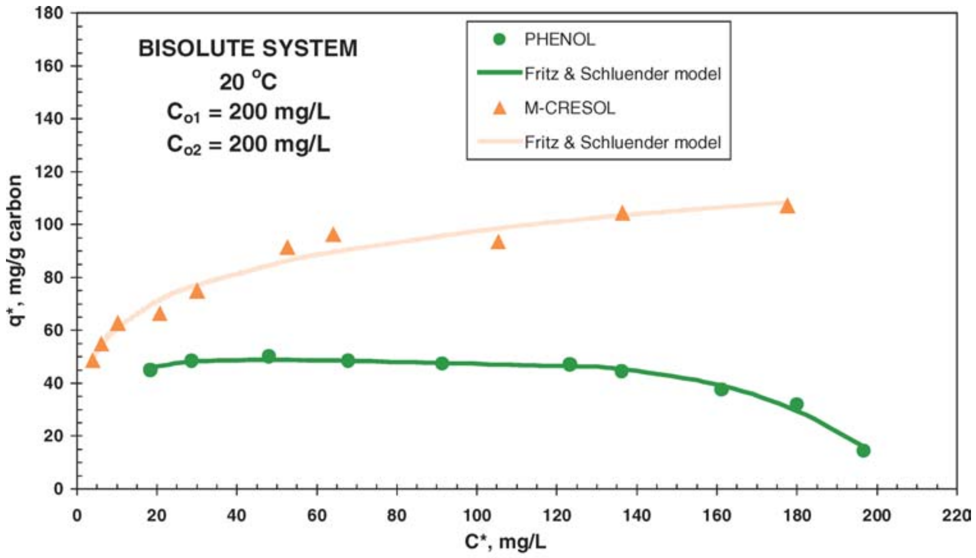
*Figure 8.* Correlations for the interaction coefficients experimentally found by fitting the modified Butler and Ockrent equations to measured loading values.

All predictions based on the Langmuir isotherm for pure compounds failed to represent adequately the experimental bisolute isotherms. The application of the IAS theory, based on the Sips isotherm for pure compounds, produced bisolute equilibrium data in better agreement with experimental results, but visible deviations between measured and predicted isotherms are still noted.

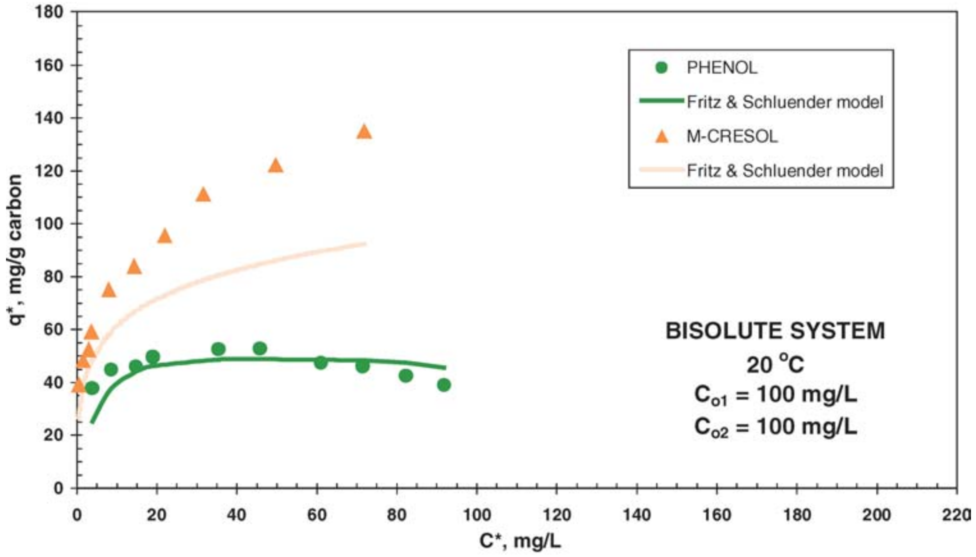
In regard to the heterogeneous nature of the activated carbon surface, the first conclusion from this study seems to be that the adequate representation of the single component isotherms is relevant for the successful application of the IAS theory in predicting multicomponent equilibrium data.

The second conclusion is that the conventional IAS theory, as presented in this study, is not able to predict with success the adsorption isotherms in binary mixtures when chemical interactions between the solutes in the adsorbed phase are present.

The results of several empirical models were compared with the predictions based on the IAS theory. The Fritz and Schluender model was able to represent accurately the experimental adsorption isotherms of phenol and *m*-cresol from a mixture with an initial composition



(a)



(b)

*Figure 9.* Comparison of bisolute experimental and calculated isotherms by the Fritz and Schluender equations (a) $C_{o1} = C_{o2} = 200$ mg/L and (b) $C_{o1} = C_{o2} = 100$ mg/L.

of 200 mg/L in both solutes. But, with the same parameter values it was not able to represent with the same accuracy the isotherms for another initial composition of the mixture.

## Nomenclature

| | |
|---|---|
| $A$ | surface area of adsorbent per unit mass |
| $b$ | equilibrium constant in the Langmuir and Sips equations, L/mg |
| $C^*$ | liquid equilibrium concentration, mg/L |
| $C_j^o$ | hypothetical concentration of the pure component $j$, mg/L |
| $K$ | Freundlich equilibrium constant, $L^{1/n}/(g\ mg^{1/n-1})$ |
| $m$ | activated carbon load, g |
| $N$ | number of experimental points |
| $n$ | constant in the Freundlich and Sips equations |
| $Q$ | maximum adsorbed concentration, mg/g |
| $q^*$ | solid equilibrium concentration, mg/g |

| | |
|---|---|
| $q_T$ | total amount adsorbed, mg/g |
| $R_g$ | gas constant |
| $T$ | temperature |
| $Z$ | reduced spreading pressure |

*Greek Words*

| | |
|---|---|
| $\eta$ | interaction coefficient |
| $\pi$ | spreading pressure |

*Subscripts*

| | |
|---|---|
| cal | cal calculated |
| exp | experimental |
| j | component |
| o | initial |
| 1 | phenol |
| 2 | $m$-cresol |

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