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A Model for Adsorption Capacities of Charcoal Beds: II. Challenge Concentration Effects

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Equilibrium and kinetic capacities of charcoal beds for adsorbed toxic gases and vapors depend on the concentrations of those gases and vapors. Water vapor also affects these capacities. The assumption of competitive (vapor *vs.* water vapor) equilibria for adsorption led to a mathematical model that quite successfully described relative humidity effects on adsorption capacities of water-immiscible vapors. This same model now has been demonstrated to describe published and new data on vapor concentration effects equally well. Parameters obtained from fits of experimental data with the model can be used for comparisons of charcoal characteristics, adsorbate-charcoal interactions and water-charcoal interactions. Correlations by this model are useful for extrapolating data to untested vapor concentrations and relative humidity conditions.

Introduction

Beds of charcoal in the forms of sampling or indicator tubes, respirator canisters, and air cleaning adsorber banks commonly are used to remove vapors of organic chemicals from air. It has been observed that breakthrough times (times to reach defined effluent concentrations) and capacities (amounts held at breakthrough) of such charcoal beds for a given chemical vapor or gas are functions of the concentration of that vapor or gas in air.(1-6)

Measured breakthrough times (t_b) for adsorbate removal from flowing air by activated charcoal have been fit to the modified Wheeler equation,(7,8)

$$t_b = \frac{W_e W}{C_o Q} + \frac{W_e \rho_B}{k_v C_o} \ln\left(\frac{C_b}{C_o}\right) \quad (1)$$

the Mecklenburg equation,(1,2)

$$t_b = \frac{w_s \rho_e A n}{C_o Q}\left[Z + \frac{1}{a_e \rho_e}\left(\frac{dG}{\eta}\right)^{0.41}\left(\frac{\eta}{\rho_a D_{20}}\right)^{0.67} \ln\left(\frac{C_b}{C_o}\right)\right] \quad (2)$$

and the Yoon/Nelson equation,(9,10)

$$t_b = \frac{W_e}{C_o Q} + \frac{W_e}{k\, C_o Q} \ln\left(\frac{C_b}{C_o - C_b}\right) \quad (3)$$

where C_o is the constant challenge concentration and C_b is the effluent concentration at airflow rate, Q. Other parameters are defined in the references given. In each of these models breakthrough time is directly proportional to a bed capacity parameter. Respectively,

W_e = kinetic adsorption capacity;(7)
w_s = weight adsorbed at total saturation;(1) and
W_e = total saturation capacity.(9)

Algebraic expressions for adsorption isotherms have been used for fitting breakthrough time and capacity data, including the Freundlich,(1,10)

$$W_e = k\ C^m \qquad (m \leq 1) \quad (4)$$

and the Dubinin,(2,7)

$$W_e = \rho W_o \exp\left(\frac{-B\ T^2}{\beta^2}[\ln(p_s/p)]^2\right) \quad (5)$$

Both of these have disadvantages that limit their usefulness (see Discussion). In the previous paper in this series a mathematical model was derived to describe effects of relative humidity on adsorption capacities of charcoal beds for a fixed challenge concentration of a water-immiscible vapor.(11) It had the form of

$$W_e = \frac{k_1 C^m}{1 + k_3 C_w^n + k_2 C^m} \quad (6)$$

At fixed water vapor concentrations, C_w, this reduces to a combination Langmuir/Freundlich isotherm. In this paper, this isotherm model is used to correlate published and unpublished data, interpret the parameters obtained, and use them to extrapolate to other challenge concentrations and relative humidities.

Review and Reformulation of the Model

The assumptions that led to the mathematical form of the model and that provide the physical interpretation of its parameters were as follows:(11)

1. Only adsorbed (or condensed) water, W, and adsorbate, A, affect the capacity, X_A, for the water-immiscible adsorbate.
2. The rate of adsorption is not affected significantly by water present.
3. Water and adsorbate equilibria exist between gas and solid phases.
4. There is a fixed concentration of homogeneously distributed micropores, $[P]_o$, which can contain either n molecules of water or m molecules of adsorbate.

TABLE I
Capacities of Two Charcoals for Chloroform[A,B]

Charcoal	Bed Depths (cm)	Relative Humidity (%)	Average Chloroform Conc. (mg/m^3)	Adsorption Capacities (g/g$_c$)[C] Experimental	Calculated	% Difference
Base	1,3	50	200	0.0424	0.0410	-3.3
	1,2,3,4	50	570	0.0681	0.0664	-2.5
	2,4	50	770	0.0759	0.0728	-4.1
	Interpolated[D]	50	450	0.0600	0.0610	+1.6
	1,2,3,4	80	450	0.0158	0.0177	+12.1
	2,4	95	450	0.0089	0.0096	+7.6
				Average Absolute Difference		=5.2
ASC	1,3	50	200	0.0662	0.0637	-3.8
	1,2,3	50	570	0.0964	0.0952	-1.2
	2	50	770	0.1085	0.1023	-5.7
	Interpolated[D]	50	450	0.0890	0.0889	-0.1
	1,2,3,4	80	450	0.0208	0.0243	+17.0
	2,4	95	450	0.0114	0.0125	+10.1
				Average Absolute Difference		=6.3

[A] Reference 11.

[B] Conditions: bed diameter = 3.1 cm; flow velocity = 8.8 cm/sec; temperature = 23° C; atmospheric pressure = 0.77 atm.

[C] At 50% breakthrough.

[D] Interpolated from the preceding data.

A fifth assumption, that the challenge concentration of adsorbate, [A], is kept constant, was only valid for the analysis of relative humidity effects alone. The first four assumptions led to the Langmuir/Freundlich-type of capacity expression with an added term for adsorbed water:

$$X_A = \frac{mbK_A[A]^m}{1 + K_W[W]^n + K_A[A]^m} \qquad (7)$$

where n = average number of water molecules filling a charcoal micropore;

m = average number of adsorbate molecules filling a charcoal micropore;

K_A = coefficient for adsorbate equilibrium between the vapor phase and condensed phase (charcoal);

K_W = coefficient for water equilibrium between the vapor phase and the condensed phase; and

[ ] = molar concentrations.

The units of K_A and K_W are L/mole to the mth and nth powers, respectively. The constant b (units of capacity) is related to the concentration (pores/g$_c$) of micropores in the charcoal, $[P]_o$, Avogadro's number, A_o, and the molecular weight of the adsorbate, MW_A, by:

$$b = [P]_o(MW_A)/A_o \qquad (8)$$

At a constant water vapor concentration (relative humidity) the inverse of Equation 7 can be expressed as:

$$\frac{1}{X_A} = \frac{1}{mb} + \left(\frac{1 + K_W[W]^n}{mbK_A}\right)[A]^{-m} \qquad (9)$$

This equation predicts that a value of m can be selected that will result in a linear relationship between the reciprocal of the capacity ($1/X_A$) and the reciprocal of the challenge vapor concentration to the mth power. The boundary condition is $m \geq 1$, or at least one molecule of A in a filled micropore or site.

Application of the Model with No RH Effect Data

Equation 9 above can be applied to concentration effect data by selecting values of $m \geq 1$ that give the best fit to the linear relationship between $1/X_A$ and $[A]^{-m}$, as indicated by the highest correlation coefficient, R. Many programmable calculators and computers are available that have programs to perform such fits of data and calculate R. More rigorous curve fitting techniques are available and could be used.[12] For this purpose, however, the simpler approach, which assumes that the error in [A] is negligible, is adequate.

This approach results in the best values of m, the intercept 1/mb, and the slope, $(1 + K_W[W]^n)/mbK_A$. The values of b and $[P]_o$ can be calculated easily and considered characteristics of the charcoal. The parameters n, K_W and K_A can be obtained from the slope values if enough relative humidity effect data are also available.

TABLE II
Parameters Derived from Concentration Effect Data for Chloroform at 50% RH[A]

Charcoal	m	R	Intercept	Slope	b (g/g$_c$)	$[P]_o \times 10^{-20}$ (pores/g$_c$)
Base	1.0	0.9998	9.67	2786	0.10	5.2
ASC	1.0	0.9973	7.43	1542	0.13	6.8

[A] See Table I.

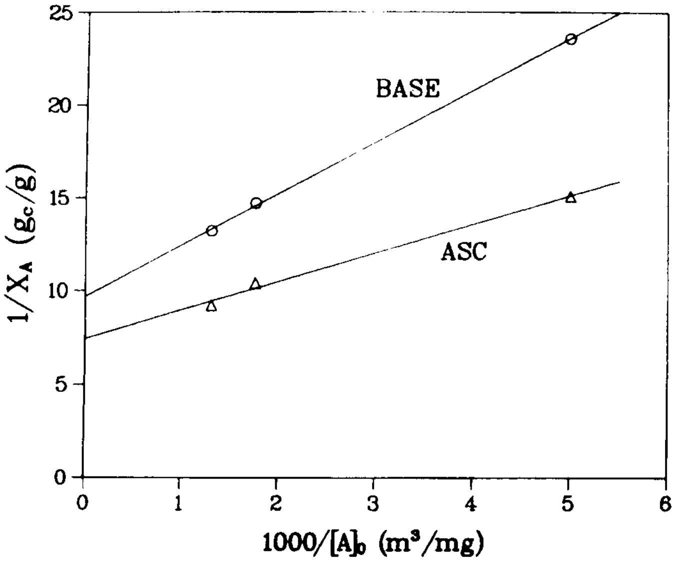

Figure 1 — Plots of Wood's (Table I) chloroform capacity data for two charcoals according to the proposed model.

The first example of using only concentration effect data with this model is with chloroform breakthrough data generated in the author's laboratory for two charcoals (BASE and ASC-impregnated) preconditioned and run at 50% RH (Table I). The resulting best fit parameters and the values calculated from them are given in Table II. Values of $m > 1$ gave poorer fits of the data to Equation 9 than m = 1. Figure 1 shows the data plotted according to Equation 9 and the solid lines calculated from the best fit parameters. Values of b (and thus $[P]_0$) for the two charcoals were similar, in agreement with physical measurements by Quantachrome Corporation that showed these two charcoals to be very similar in pore characteristics.

The most extensive studies of vapor concentration effects on respirator cartridge capacities have been done by Nelson *et al.*[1–3] The data from Table IV of Reference 1 for eleven solvent vapors and three cartridge types preconditioned and run at 50% RH to 10% breakthrough are reproduced in Table III. They were used with the proposed model, as described in the preceding section, to derive the parameters also given in Table III. Expect for isopropanol (m = 1.5) the minimum value of m (= 1.0) gave the best linear correlations according to Equation 9. Correlation coefficients were all greater than 0.97. Figure 2 shows the data plots for acetone and two different cartridges.

Values of b from intercepts and m were used with Equation 8 to calculate active pore concentrations, $[P]_0$, which ranged from 1.7×10^{21} to 3.7×10^{21} pores/g of charcoal (Table III). The narrow range (estimated standard deviation = 0.7×10^{21}) of this parameter for widely different solvents confirms that the average, $[P]_0 = 2.6 \times 10^{21}$, can be considered a parameter of the charcoals contained in the cartridges.

In contrast, the slopes of the linear fits to Equation 9 vary more than two orders of magnitude with solvent. If it is assumed that the $K_W[W]^n$ term is constant at constant 50% RH, relative values of K_A can be calculated from these slopes and intercepts (Table III). It would be interesting to try to relate relative K_A values to properties of the solvents, such as polarity, volatility, solubility in water, or molecular size; however, that is beyond the scope of this paper. The type of charcoal in the cartridges also can affect K_A, even though b's are similar (see hexane in Table III).

The third example of applying this model was with data published by Werner[4] for trichloroethylene at five relative humidities (5% to 85%) and four challenge concentrations

TABLE III
Parameters Derived from Concentration Effect Data of Nelson and Harder[A]

Solvent	Cartridge Type	m	R	Intercept	Slope	b (g/g_c)	$[P]_0 \times 10^{20}$ ($pores/g_c$)	Relative K_A
Acetone	2	1.0	0.9793	6.31	1411	0.16	17	42
Acetone	3	1.0	0.9873	4.34	907	0.23	24	45
Benzene	1	1.0	0.9747	2.82	406	0.35	27	65
Carbon tetrachloride	1	1.0	0.9983	1.05	499	0.95	37	20
Dichloromethane	1	1.0	0.9953	3.77	4271	0.27	19	8
Diethylamine	1	1.0	0.9987	2.25	1484	0.45	37	14
Hexane	1	1.0	0.9712	3.01	299	0.33	23	94
Hexane	3	1.0	0.9849	2.82	134	0.35	24	196
Isopropanol	1	1.5	1.0000	2.55	23720	0.26	26	1
		1.0	0.9968	2.13	1257	0.46	46	16
Methyl acetate	1	1.0	0.9791	4.62	1186	0.22	18	36
Methyl chloroform	1	1.0	0.9885	1.54	313	0.65	29	46

[A] Reference 1, Table IV.

(300 to 1300 mg/m^3). The test beds were initially dry, rather than pre-equilibrated at the test air humidity as in the first two cases above and as assumed by the model. The best fit parameters obtained by Equation 9 and calculated are given in Table IV. At 50% RH the best fit (R = 1.0000) was for m = 1.4, rather than for m = 1.0 as with the other RHs. The curve fit (R = 0.9979) of the 50% RH data for m = 1.0 was not much worse, but gave a significantly different slope, as would be expected. At 85% RH the slope for m = 1.0 was negative, but for m = 1.03 was slightly positive.

These inconsistencies are due mostly to uncertainties in the data and deviations from model assumptions. At the higher relative humidities dry beds are heated significantly by the adsorption of water vapor,(13) which affects the capacity and other physiochemical characteristics of the system. The fits (Figure 3), however, still give very good empirical correlations that can be used for extrapolations and interpolations of data. Except for the highest humidity case, where the heating effect is most extreme, the active pore concentration values calculated were quite consistent, 1.4 - 2.4 × 10^{21} pores/g$_c$.

Application of the Model with Dry and Higher RH Condition Data

When adsorption capacity *vs.* challenge concentration data are available for dry conditions as well as for higher humidities, an alternative use of Equation 9 can be made. If the humidity term, $K_W[W]^n$, is very small ($\ll 1$), K_A can be calculated from the intercept/slope ratio. For Werner's data(4) at 5% RH this gives $K_A = 0.00456$ for m = 1.0.

Humidity variation data for the same system at a fixed challenge concentration can be handled as in Reference 11, ($1/X_A$ *vs.* $[W]^n$), giving the best value of n and slope/intercept = $K_W/(1 + K_A[A]^m)$. Folding in the values of K_A and m gives a value for K_W. Thus, all the parameters needed to define humidity and challenge concentration effects using Equation 9 are derived and available for calculating bed capacities (and breakthrough times) at untested conditions.

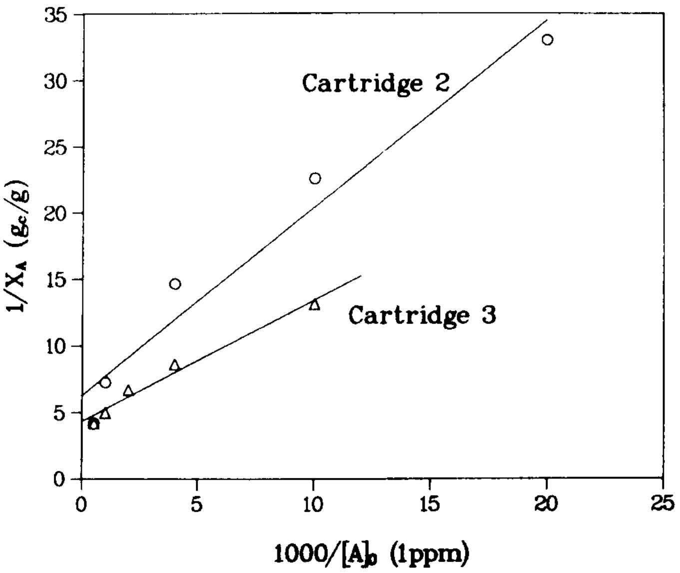

Figure 2 — Plots of Nelson's(1) acetone capacity data for two cartridges according to the proposed model.

TABLE IV
Parameters Derived from Concentration Effect Data of Werner for Trichloroethylene[A]

Relative Humidity (%)	m	R	Intercept	Slope	b (g/g$_c$)	$[P]_o \times 10^{-20}$ (pores/g$_c$)
5	1.00	0.9728	2.06	454	0.48	22
25	1.00	0.9823	1.95	592	0.52	24
50	1.40	1.0000	2.33	9179	0.31	14
	1.00	0.9979	1.83	1081	0.55	25
65	1.00	0.9885	2.74	1825	0.36	17
85	1.00	0.9995	−0.41	10995	−2.3	-
	1.03	0.9995	0.13	12860	7.4	340

[A]Reference 4.

In accordance with Werner's data,(4) such a treatment of the RH variation for the 293 - 303 mg/m data set yields $K_W([W]^n_{sat}) = 59.3$ for n = 6.2 (R = 0.9989). The saturated water vapor concentration, $[W]_{sat}$ = 22 000 mg/m^3 at 23°C, comes from using fractional relative humidities instead of absolute water vapor concentrations, according to the equation:

$$K_W[W]^n = K_W[W]^n_{sat}(RH)^n \qquad \textbf{(10)}$$

The calculated parameters (m, b, K_A, n, and K_W) then were used to calculate bed capacities at all the experimental relative humidities and challenge concentrations. Table V shows a comparison of calculated capacities and experimental ones. The average absolute difference was 5.8%.

A similar comparison using Werner's data(4) at 1304 - 1356 mg/m^3 to give $K_W([W]^n_{sat}) = 44.8$ for n = 5.9 (R = 0.9988), resulted in an average absolute difference of 6.8%.

Application of the Model with No Dry Condition Data

When bed capacity or breakthrough time data for a range of challenge concentrations are available only at higher relative humidities (≥ 50%), these two effects still can be correlated by the proposed model. An iterative calculation process can be used as follows:

1. Obtain the parameters m, b, slope (S_1), and intercept (I_1) from the best linear fit of $1/X_A$ *vs.* $[A]^m$ at one selected RH$_j$.
2. As a first approximation assume $K_A = I_1/S_1$ (*i.e.*, $K_W[W]^n \ll 1$) and calculate K_A.
3. For one concentration, $[A]_i$, with data at three or more RHs estimate the dry bed capacity by:

$$(X_A)_{Dry} = \frac{mbK_A[A]_i^m}{1 + K_A[A]_i^m} \qquad \textbf{(11)}$$

4. Calculate the ratios $(X_A)_{Dry}/X_A$ at each RH and find the values of slope (S_2) and n that give the best linear fit with an intercept of 1.00 for the equation:

$$\frac{(X_A)_{Dry}}{X_A} = 1 + \left(\frac{K_W[W]^n_{sat}}{1 + K_A[A]_i^m}\right)(RH)^n \qquad \textbf{(12)}$$

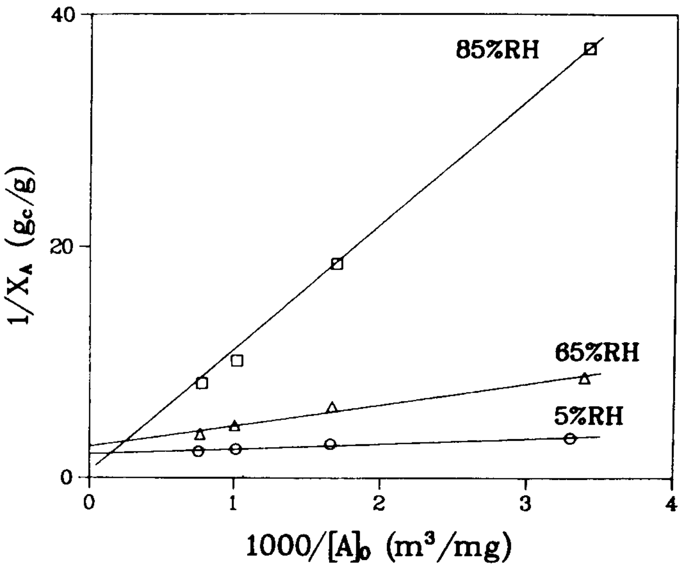

Figure 3 — Plots of Werner's(4) trichloroethylene capacity data at three relative humidities according to the proposed model.

5. Estimate $K_W[W]^n_{Sat} = S_2(1 + K_A[A]^m_i)$.
6. Reestimate $K_A = (I_1/S_1)(1 + K_W[W]^n_{sat}(RH)^n_i)$.
7. Repeat Steps 3 to 6 until the same values of n and S_2 are obtained after two or more successive iterations.
8. If K_A and $K_W[W]^n_{sat}$ have not converged to repeatable values, they can be obtained by solving the simultaneous equations:

$$K_A = (I_1/S_1)(1 + K_W[W]^n_{sat}(RH_j)^n) \tag{13}$$

$$K_W[W]^n_{sat} = (S_2)(1 + K_A[A]^m_i). \tag{14}$$

The data in Table I for two charcoals were treated by this iterative procedure. Since only two measurements were done at 450 mg/m^3 chloroform (80% and 95% RH), a third data point was obtained for 450 mg/m^3 by interpolating the 50% RH data at the three other concentrations using Equation 5. In addition to the parameters in Table II this process yielded:

Base Charcoal: $n = 4.1$, $S_2 = 12.24$ at 450 mg/m^3;
$R = 0.9993$;
$K_A = -0.0520$; and
$K_W[W]^n_{sat} = -274$.

ASC Charcoal: $n = 4.4$, $S_2 = 13.12$ at 450 mg/m^3;
$R = 0.9986$;
$K_A = -0.0225$; and
$K_W[W]^n_{sat} = -120$.

Negative values of K_A and $K_W[W]^n_{sat}$ were due to extrapolation from high humidity (≥ 50%) conditions to dry. These negative values have no physical significance, but are still useful for describing the bed capacities in the experimental range (≥ 50% RH). Table I also shows calculated chloroform adsorption capacities and deviations from experimental values in which these derived parameters are used. The average absolute deviations were 5.2% for the Base charcoal and 6.3% for the ASC.

Discussion

One goal of correlating experimental data and defining relative humidity and challenge concentration effects is to provide the ultimate user with information to predict performance of the bed (canister, sampler, adsorber) at actual use conditions. The development of a descriptive model and the extraction of parameters for the model are the first steps. Sets of parameters for each bed/vapor combination and actual relative humidity and vapor concentration data can be inserted into a simple calculator or computer program to estimate bed capacity and service life.

With more bed/vapor combinations and relative humidity and challenge concentration data, it may be possible to estimate the parameters for untested cases. Is m = 1 usually the best value? Is there a consistent value of b that can be used? Can K_A be correlated to some property of the vapor species?

In compiling listings of useful parameters the question arises: what is the minimum number of laboratory tests that will define these parameters? Resources always are limited and must be balanced against the desirability of as much data as possible. In the case of the model presented here, the minimum number is five determinations of bed capacity.

TABLE V
Comparisons of Werner's[A] Experimental Capacities with Those Calculated Using the Proposed Model

Relative Humidity (%)	Vapor Conc. (mg/m^3)	Adsorption Capacities (g/g_c) Experimental	Calculated	% Difference
5	303	0.286	0.281	+1.7
25	295	0.257	0.277	-7.7
50	293	0.180	0.206	-14.5
65	295	0.114	0.101	+11.3
85	293	0.027	0.027	0.0
5	602	0.334	0.355	-6.3
25	605	0.320	0.355	-10.8
50	597	0.284	0.291	-2.6
65	599	0.160	0.169	-5.6
85	593	0.054	0.052	+4.3
5	987	0.399	0.397	+0.6
25	995	0.403	0.396	+1.7
50	978	0.342	0.345	-0.8
65	996	0.218	0.228	-4.7
85	986	0.098	0.080	+18.1
5	1331	0.434	0.416	+4.1
25	1306	0.431	0.414	+3.9
50	1356	0.370	0.375	-1.4
65	1322	0.262	0.262	0.0
85	1304	0.121	0.101	+16.7
			Average Absolute Difference	=5.8

[A] Reference 4, Table I.

Testing at three challenge concentrations at driest practical bed and air conditions will give the parameters m, b, and K_A. Testing at three relative humidities for a fixed challenge vapor concentration will give the parameters n and K_W. If planned right, however, the data from one test can be used for both data fits, reducing the total required data sets to five. The ranges of vapor concentration and relative humidity should span the values of practical interest.

Dry bed and dry air test data of Werner[(4)] was used to derive the parameter K_A by the assumption $K_W[W]^n \ll 1$. By using the values derived from this data for $K_W[W]^n_{sat}$ and n above, this translates to $RH \ll 0.5$. This means that tests at 50% RH are not dry enough to define K_A by this assumption.

Temperature effects[(13)] have not been considered yet in this model. Much more data are necessary to define them. It may turn out, however, that only the equilibrium coefficients K_A and K_W are affected significantly and they according to classical Arrhenius relationships.

The model worked well with both preconditioned bed data (Table I and Nelson[(1)]) and dry bed data (Werner),[(4)] even though the latter does not represent an equilibrium situation. It also worked with both 10% breakthrough capacities (Nelson[(1)]) and 50% (Table I and Werner[(4)]). This usefulness for situations not completely consistent with the original model assumptions is gratifying.

The value m = 1 for the best fit of concentration data (in most cases) was a consequence of the data, not the model. It was unexpected in light of the Dubinin theory of pore condensation. According to the model developed earlier, this is interpreted as only 1 molecule per pore. Perhaps, adsorption site would be more appropriate than pore. For water the range of n for different charcoals and studies was 3.4 to 8.4.

Comparisons of Adsorption Isotherm Models

The Langmuir isotherm (m = 1) fit of experimental data has three advantages: 1) it requires only vapor concentration and capacity data to be applied; 2) it has parameters that can be given physical meaning and possibly be determined by independent measurements or data; and 3) it extrapolates to theoretically acceptable limits at both high and low concentrations.

The Dubinin isotherm (Equation 5) usually is used by plotting ln W_e *vs.* $[\ln(p_s/p)]^2$ in the hope of getting a straight line which yields ρW_o from exp[intercept] and $-BT^2/\beta^2$ from the slope. For highly volatile or exotic vapors or gases the saturation vapor pressure, p_s, however, may not be available from references. The adsorbate liquid density, ρ, also may be difficult to obtain for estimating W_e from a known micropore volume, W_o. The Langmuir and Freundlich equations do not have such parameters, but can be used directly with experimental data.

The parameters of the Freundlich isotherm, Equation 4, have no physical interpretation and, therefore, no possibility of being calculated *a priori* for other vapors or charcoals for which no data exist. The Langmuir parameters, K_A and b in Equation 7, correspond to an equilibrium coefficient and an active pore (site) concentration, respectively. The Dubinin parameters, likewise, have physical interpretations.

All isotherm models adequately may describe adsorption capacities over a limited range of vapor concentrations. It may be necessary, however, to extrapolate data to higher or lower concentrations. For example, this may be necessary when 1) a better facepiece fit factor is allowed and, therefore, a larger challenge concentration can be tolerated; or 2) the acceptable exposure limit is lowered, thereby reducing the concentrations of interest. The Langmuir isotherm has the appropriate high and low concentration limits, which correspond to a finite number of possible adsorption sites and Henry's law (linear isotherm), respectively. The Freundlich isotherm does not approach a finite capacity limit at high concentrations. Neither the Freundlich nor the Dubinin isotherms approach linearity at low concentrations.

Conclusions

The model presented provides good correlations of effects of relative humidity and challenge vapor concentration on adsorption capacities of charcoal beds. Both dry charcoal bed and humidity preconditioned bed data can be fit by this model.

These correlations can be used to interpolate and even extrapolate laboratory test data to actual field conditions. At the sacrifice of some accuracy, as few as five measured capacities can provide the parameters for the model.

The model parameters have physical significance related to the adsorbent and adsorbate properties. Some trends in these parameters, observed with very limited data, should be examined further to discover ways of predicting these parameters for untested beds or adsorbates.

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