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Effect of Relative Humidity on the Adsorption of Selected Water-Miscible Organic Vapors by Activated Carbon

Khaldoun H. Kawar[a]
Dwight W. Underhill[b]*

[a]Tammer Sciences, Inc.,
Naperville, IL, 60564;
[b]School of Public Health,
University of South Carolina,
Columbia, SC 29208

The adsorptive capacity of activated charcoal was determined experimentally for the vapors of 2-ethoxyethanol, pyridine, acetic acid, and piperidine from dry air and from air saturated with water vapor. Vapor concentrations ranged from 100 mg/m^3 to at least 1000 mg/m^3; the temperature was kept constant at 25°C. The reduction in the adsorptive capacity of the activated charcoal by the relative humidity over the entire range of experimental conditions was accounted for by the Hansen-Fackler modification of the Dubinin-Radushkevich equation. This procedure allows the use of the activity coefficients, which are basic thermodynamic factors often available in the literature, to estimate the effect of adsorbed moisture on the adsorption of these organic compounds from a humidified atmosphere.

Keywords: adsorption, activated carbon, isotherm, relative humidity, moisture, organic compounds

Without question, adsorption is often the preferred method to remove small concentrations of contaminants from airstreams, and many such applications, as in respirator cartridges, are critical to human health. Optimizing the performance of adsorption systems requires combining theory with experiment; but there are so many very different theoretical approaches that the industrial hygienist may feel bewildered in trying to apply theory to experiment. For example, the Langmuir equation, the Brunauer, Emmett, and Taylor (BET) equation, the restricted BET equation, the two dimensional van der Waals' equation, and the Dubinin-Radushkevich equation are just a few of the equations that may be applied to the same data for the adsorption of a single component on activated carbon. The number of models available becomes even more complex when one tries to modify these equations or combinations of them to account for the coadsorption of mixtures on activated carbon. Because water vapor is almost always present in the ambient air in the range of tens of thousands of parts per million or more, the industrial hygienist must nearly always consider coadsorption with water vapor as an important confounding factor.

There are several reasons that the potential theory first given by Polanyi,(1) modified by Dubinin and Radushkevich,(2) and later by Manes and his co-workers,(3,4) provides an excellent framework on which to model the coadsorption of compounds on activated carbon. These include:

- The Dubinin-Radushkevich isotherm has been found to give an excellent description of solvent vapors on dry activated carbon.
- The Dubinin-Radushkevich theory can predict accurately the coadsorption of compounds from water solutions,(3,4) proving that this theory applies to coadsorption in the presence of water.
- Potential theory is simple to grasp, and the resulting equations do not have to become so complex that they are not useful.
- The extension of potential theory to the adsorption of mixtures (at least as applied here) uses as an important part of its input the thermodynamic properties of these compounds and their solutions in water. Thus, what we know about the activity coefficients and solubility coefficients of these compounds (and a surprising amount of such information is available in the literature) can be applied directly in these adsorption equations,

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* Author to whom correspondence should be addressed.

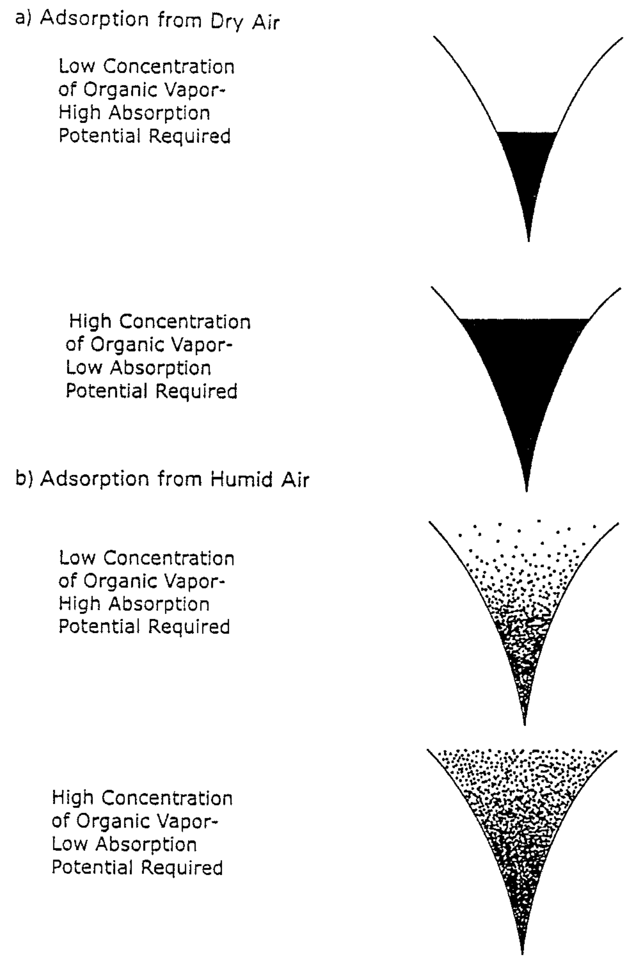

FIGURE 1. Potential well in a micropore

drastically reducing the number of unknown constants that appear in these equations. This in turn has the great benefit of reducing the amount of data that must be collected to define the parameters of the system.

An earlier article used a modification of the Dubinin-Radushkevich theory to determine the affect of relative humidity on the adsorption of water immiscible compounds in the presence of water vapor.(5) The present article extends this theory to the adsorption of water miscible compounds from a humid atmosphere.

Development of the Theory

Here the two important examples of the application of potential theory—the adsorption of a single component, and the coadsorption of an immiscible solvent with water—are reviewed; then this theory is extended to the coadsorption of water miscible compounds from humid atmospheres.

Adsorption of a Single Component

Figure 1 shows the adsorption of a single component in a "potential well" according to the Polanyi potential theory. The concentration of the adsorbate in this potential well will increase until the free energy gained through compression matches the loss in free energy through "falling" into this potential well. Where the potential energy is equal or greater than that required to compress the vapor to a concentration greater than the vapor pressure of the pure liquid, then condensation occurs. This energy of compression is

$$\epsilon = RT \ln\left(\frac{P_0}{P}\right) \tag{1}$$

and the corresponding volume of the adsorption space is:

$$\phi = n\bar{V} \tag{2}$$

where:

ϵ = adsorption potential, calories/mole

ϕ = volume of adsorption space for which the adsorption potential is $\geq \epsilon$, mL/kg

n = moles of adsorbed vapor, moles

P = pressure at which n moles of vapor are adsorbed, bars

P_0 = saturated vapor pressure, bars

R = ideal gas constant, 1.9872 calories/mole/K

T = absolute temperature, K

$\bar{V}$ = molar volume, mL/mole

According to the generalized Dubinin-Radushkevich equation,(6,7) the pore volume corresponding to a particular potential energy is

$$\phi = V_0 \exp(-k\epsilon^b) \tag{3}$$

where:

V_0 = micropore volume, mL/kg

k = the first structural constant, moles^b calories(1/$^{-b}$)

b = the second structural constant, dimensionless

Regarding the factor "b" in Equation 3, note that b=2 gives the standard Dubinin-Radushkevich equation and that b = 1 gives the Freundlich isotherm. In practice, values of "b" differing from b=2 have been found for some commercial activated carbons.(8)

Adsorption of a Water-Miscible Compound from a Humid Atmosphere

Polanyi(9) also applied his theory of adsorption to adsorption of a single component from solution. Hansen and Fackler(10) modified this theory to nonideal solutions of complete miscibility; their reason being "Polanyi's theory as applied to adsorption from solution (1920) cannot be general, for it is not symmetric with respect to solute and solvent." The required symmetrical equation, developed by Hansen and Fackler, is

$$\frac{a_{1\phi}}{a_{2\phi}{}^{\alpha}} = \frac{a_{1B}}{a_{2B}{}^{\alpha}} \exp\left(\frac{\epsilon_1(\phi) - \alpha\epsilon_2(\phi)}{RT}\right) \tag{4}$$

where the a are the activities of the components; the subscripts 1 and 2 refer to the organic component and water, respectively; and the subscripts ϕ and B refer to the adsorbed and bulk phases. The factor, α, is the ratio of the partial molar volumes of the organic component and of water, respectively.

But from the extended Dubinin-Radushkevich equation, at the same site in the adsorption space, that is, $\phi(\epsilon_1) = \phi(\epsilon_2)$, then from Equation 3:

$$V_0 \exp(-k_1\epsilon_1^b) = V_0 \exp(-k_2\epsilon_2^b) \tag{5}$$

or:

$$\epsilon_1 = \left(\frac{k_2}{k_1}\right)^{\frac{1}{b}} \epsilon_2. \tag{6}$$

Combining Equations 4 and 6 gives the following result for the

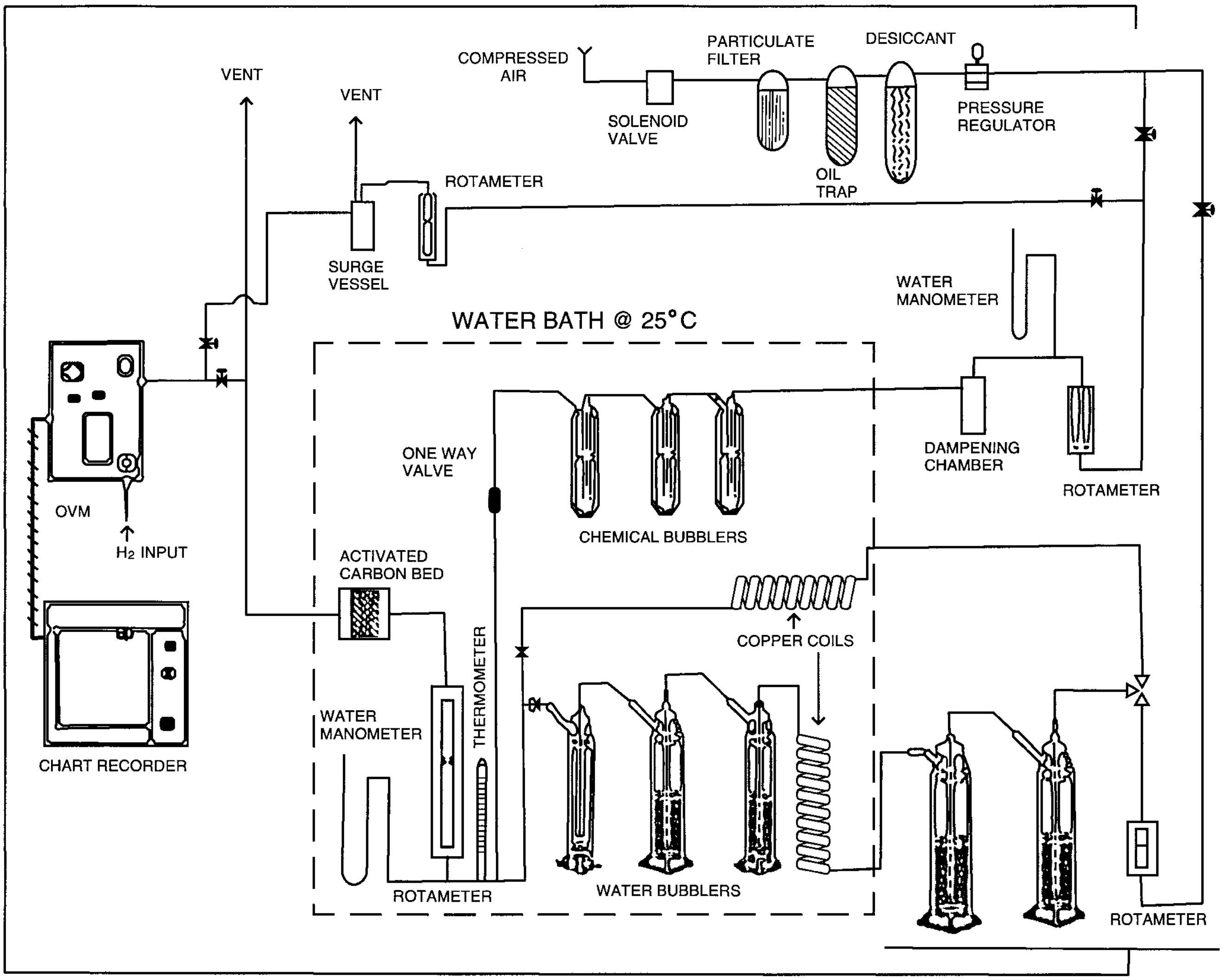

FIGURE 2. Diagram of the test system

activity of a water miscible organic compound in aqueous solution:

$$\epsilon_1 = \frac{RT\left(\ln\left(\frac{a_{1i}}{a_{1B}}\right) - \alpha \ln\left(\frac{a_{2i}}{a_{2B}}\right)\right)}{Z} \qquad (7)$$

where Z = a function of k_1, k_2, and b, that is,

$$z = 1 \pm \left(\frac{k_1}{k_2}\right)^{1/b}.$$

But here the concern is with the adsorption of an organic vapor from a water-saturated atmosphere. Although there is no bulk liquid phase, there is an equivalent gas phase, and the last equation can be used just as generally if the subscript b, instead of referring to the bulk phase, now applies to the aqueous surface in contact with the gaseous phase.

The moles of the organic compound that were adsorbed can be calculated from the integral:

$$m = \int_0^\infty \frac{x_1\, d\phi}{V} \qquad (8)$$

which for the generalized Dubinin-Radushkevich equation is:

$$m = -bk_1V_0 \int_0^\infty \frac{x_1 \epsilon^{b-1} \exp(-k_1\epsilon^b)\, d\epsilon}{V} \qquad (9)$$

where:

x_1 = mole fraction of the organic component and $\bar{V}$ = molar volume of mixture, L/mole.

Test System

Here the adsorption coefficients of five water miscible compounds were determined from both dry air and air saturated with water vapor, and it is shown that the equations developed here can be used to correlate such data effectively. By eliminating lower relative humidities as a variable, a simple single condition can be used as a baseline for estimating the maximum effect of the relative humidity. This approach can prove to be extremely useful to the practicing industrial hygienist, especially when evaluating the performance of respirator cartridges.

A schematic diagram of the test system is shown in Figure 2, and a detailed description is as follows. Laboratory compressed air at 40 psi passed through sequentially a pressure regulator, a particulate filter, an oil trap, and an adsorbent bed containing a mixture of a drying agent and activated charcoal. This inflowing air was then split into three streams: (1) a main stream, (2) a generating stream, and (3) a dilution stream. The main airstream passed first through a calibrated rotameter, then through a two-way valve to direct the air either through the water bubblers for the humid tests or bypassing the bubblers for the dry tests. For the latter, to bring the air to the desired temperature the main airstream was directed through a copper tubing coil submerged in the temperature-controlled water bath. To perform the runs under humid conditions, the main airstream was directed through, in series, four bubblers half filled with double distilled water, and a fifth bubbler kept empty to trap entrained water droplets. The first two bubblers were outside the water bath.

The air in the generating stream was saturated with the vapor of a test analyte by bubbling it through three impingers in series. The five analytes (acetic acid, allyl alcohol, pyridine, piperidine, and 2-ethoxyethanol) are widely used water miscible compounds. Although a rotameter helped maintain a constant input of analyte, the actual generation rate was determined gravimetrically. A dampening chamber between the rotameter and the impingers eliminated the pulsating of the rotameter from the air bubbling through the impingers.

The main stream was then reunited with the generating airstream to give a combined flow rate of 20 L/min. The concentrations in the combined stream ranged from as high as five times the current threshold limit value to as low as possible without losing accuracy. An in-line thermometer and a water manometer monitored the temperature and pressure of the combined airstream. The combined airstream then passed through a calibrated rotameter to the bed of activated carbon, also completely submerged in the water bath.

The dilution stream used to dilute the sampling airstream, first passed through a rotameter then into a bottle vented on the side. The bottle was kept under constant positive pressure by keeping the airflow at a preset rotameter reading.

The adsorbent was 8/16 mesh, Barnebey-Cheney grade #177, activated carbon, having a CCl_4 activity of 60%, as measured by ASTM method D-3467.(11) Before use it was dried for 24 hours at 140–150°C, cooled, and stored in a desiccator. In each test, 4 g of activated carbon were placed in a Teflon® bed, 2.5 cm in diameter; the depth of the activated carbon in the bed was approximately 2.5 cm.

A flame ionization detector (Century System Corp., Model 118 Organic Vapor Analyzer) continuously monitored the organic content in a 0.6 L/min side-stream from the effluent air. Due to the corrosive nature of some compounds tested, and to prevent water condensation in the detector, the sampling stream was mixed with 0.4 L/min of dilution air. The dilution air did not effect the zero reading of the detector because it was the same air used to zero the system.

Determination of the Adsorption Coefficient

The amount of adsorbed organic vapor was calculated from the equilibrium breakthrough curves by performing a mass balance around the activated carbon bed over the time it took the effluent concentration to equal the influent concentration.(12) The equations used were:

$$M = \dot{q} \int_0^{\infty} (C_0 - C)\, dt \tag{10}$$

where:

$\dot{q}$ = flow of air through the adsorption bed, m^3/sec

C = concentration of analyte in the effluent air from the adsorption bed, kg/m^3

C_0 = concentration of analyte in air entering the adsorption bed, kg/m^3

M = mass of adsorbed vapor at equilibrium, kg

t = time, sec

and:

$$K = \frac{M}{\rho W} \tag{11}$$

where:

K = adsorption coefficient, L/kg

W = dry weight of adsorbent, kg

ρ = density of pure organic liquid, kg/L

In using Equations 10 and 11 to determine the uptake of analyte, the integration is stopped at the point where the input and effluent concentrations of analyte are indistinguishable. The adsorption coefficients so determined are given in Table I and by the symbols in Figures 3 and 4.

DATA ANALYSIS

Adsorption on Dry Activated Carbon

Because the standard Dubinin-Radushkevich equation (b=2) gave an excellent fit to the data obtained at 0% RH, this equation was used here and throughout the remainder of this article. Table I and the lines in Figure 3 give the adsorption coefficients calculated from the Dubinin-Radushkevich equation (Equation 3) for adsorption from dry air. The V_0 and k_1 used in these calculations are given in Table II. The thermodynamic properties of the adsorbates used in these calculations are tabulated in Table III. In Figure 3 the match between the experimentally determined results (the symbols) and the estimates derived from the Dubinin-Radushkevich equation (the lines) appear so close that a casual look at this figure might suggest that the lines merely represent the best-fitting curves drawn through the experimental results; whereas actually the lines represent the Dubinin-Radushkevich equation as applied to these data.

Adsorption at 100% RH

Before Equations 7 and 9 can be used, the relationship between the activity coefficients and the mole fraction of the organic compound in solution must be established. This relationship is a basic thermodynamic property that has an important place in the literature of physical chemistry. Here the van Laar equations(13) were used:

$$\ln \gamma_1 = A_{12}\left(\frac{A_{21}x_2}{A_{12}x_1 + A_{21}x_2}\right)^2; \tag{12a}$$

$$\ln \gamma_2 = A_{21}\left(\frac{A_{12}x_1}{A_{12}x_1 + A_{21}x_2}\right)^2 \tag{12b}$$

where γ_1 and γ_2 are the activity coefficients of the organic compound and of water, respectively, and A_{12} and A_{21} are the van Laar coefficients for the binary solution (the organic compound dissolved in water).

The activity of each component is its mole fraction multiplied by its activity coefficient.

The van Laar coefficients are often available for solutions of organic compounds, and the values of the van Laar coefficients,

TABLE I. Measured and Calculated Adsorption Coefficients

Compound	Concentration (ppm)	Uptake at 0% RH (mL/kg)		Uptake at 100% RH (mL/kg)	
		Measured	Calculated	Measured	Calculated
Acetic acid	83	293.4	310.9	202.7	188.3
	92	314.9	318.7	204.3	195.2
	183	399.4	371.1	230.7	244.5
	293	423.9	407.2	267.6	280.9
	495	456.6	446.6	296.8	323.2
	678	464.6	469.4	332.6	349.1
	880	484.0	487.7	433.1	370.6
Allyl alcohol	46	126.9	108.9	37.8	40.0
	117	163.6	187.3	68.1	71.5
	190	222.8	223.7	99.6	94.8
	235	241.1	245.5	104.2	106.2
	379	289.1	298.6	144.6	136.2
	516	329.5	335.3	167.0	158.5
	728	375.2	378.0	194.3	185.9
Ethoxyethanol	30	358.2	370.2	248.0	234.7
	57	431.0	413.0	262.5	264.9
	121	461.0	461.4	296.0	301.8
	212	486.7	495.2	302.1	329.9
	317	533.4	517.6	377.0	351.6
Piperidine	75	347.4	337.3	255.1	251.8
	149	366.5	383.5	290.3	292.7
	287	418.8	427.5	337.0	333.4
	411	462.1	451.2	348.9	356.0
	682	496.2	483.4	387.4	387.9
Pyridine	54	267.6	257.2	147.9	151.9
	109	301.1	313.1	188.2	183.9
	208	362.5	367.5	220.0	216.1
	286	389.6	394.8	228.1	232.7
	546	459.4	449.8	275.8	270.0

along with their source, are given in Table III. Were these coefficients unavailable, they can be estimated from vapor-liquid equilibria data, for which Hine and Mookerjee have tabulated many of the then available data.(14,15) For compounds for which no data are available, they give an additive scheme to estimate the required vapor-liquid equilibria.

Here the van Laar coefficients for piperidine-water were estimated from gas-liquid equilibria at reduced pressures.(16) An independent measurement of the activities in this same system was made earlier, but apparently the results were never published in detail.(17)

The overall computation procedure was as follows:

- First determine the concentration of organic component in equilibrium with the ambient, humid air. At the gas liquid interface (which is at the top of the potential well), the adsorption potential should be very close to zero. Thus, for this purpose, one can use the available vapor-liquid equilibrium data to establish the organic concentration.
- Then, from Equation 7, calculate the mole fraction of organic compound as a function of potential energy. Next calculate from Equation 8 the total uptake of the organic component. Because the equilibrium factor, Z, is not known, it is determined through trial and error.

Finding the values of the unknown parameters, k_1, V_0, and Z, was helped appreciably by the availability of a comprehensive set of tested subroutines for, among many other tasks, locating roots and finding minima of equations.(18) The computer program developed here is available on request from the authors.

Table I and the lines in Figure 4 give these calculated adsorption coefficients. Table II gives, along with the V_0 and k_1 cited earlier, the values of the equilibrium coefficient, Z, used to calculate these adsorption coefficients, along with the root mean

TABLE II. Calculated Dubinin-Radushkevich and Hansen-Fackler Constants

Compound	Dubinin-Radushkevich Factor, V_0 (mL/kg)	Dubinin-Radushkevich Factor, $10^8{*}k_1$ (mole/cal)$^{(2)}$	Equilibrium Coefficient, Z (Dimensionless)	Percentage Root Mean Square Difference
Acetic acid	617.0	6.013	0.798	6.3
Allyl alcohol	727.7	12.11	1.023	5.7
Ethoxyethanol	609.2	4.680	1.277	4.5
Piperidine	627.3	4.498	0.976	2.3
Pyridine	647.6	6.801	1.314	2.5

TABLE III. Physical Characteristics of the Adsorbates

Compound	Vapor Pressure at 25°C (mmHg)(20)	Specific Gravity(21)	Molecular Weight (g/mole)	van Laar Coefficients(15, 22) A_{12}	A_{21}	Factor "α" (see Eq. 4)
Acetic acid	18.86	1.049	60.05	1.0594	0.7471	3.18
Allyl alcohol	28.11	0.854	58.08	1.987	1.2042	3.78
Ethoxyethanol	5.63	0.930	90.12	1.8987	0.708	5.38
Piperidine	30.19	0.861	85.15	1.700	1.22	5.49
Pyridine	20.68	0.978	79.10	2.3105	0.625	4.49
Water	23.756	0.997	18.02	NA	NA	NA

square percentage difference between the calculated and the experimental values. These root mean square percentage differences ranged between 2 and 6%, in the range of the experimental error of the measurements.

DISCUSSION AND ANALYSIS

A fundamental goal of this study was to provide a link between adsorption from air and adsorption from aqueous solution. The Hansen-Fackler modification of the Polanyi potential theory as it is used in this investigation provided such a link. As in the Polanyi theory, if one assumes that adsorption from the vapor phase is enhanced liquefaction, one can imagine the total adsorption space of activated carbon to be analogous to an irregularly shaped well containing a powerful force field. These forces are generated by the walls of this imaginary well and are analogous to the adsorption potential, as described by Polanyi. Therefore, at equilibrium and 100% relative humidity, this irregularly shaped well is filled with the organic-water solution. The concentration of the organic in this solution increases as the distance from the surface of the well increases and the surface layer is at equilibrium with its respective vapor mixture.

The individual potential energy wells may have an irregular shape, but their average shape as a function of potential energy may be described by the extended Dubinin-Radushkevich equation. With this concept one can use the Hansen-Fackler equation to describe the composition of the adsorbate solution, given the adsorption potential of both the water and organic at each equipotential plane, in addition to the bulk concentration of the reference solution.

In this analysis, there are three values (V_0, k_1, and Z) that must be determined experimentally. In theory this can be accomplished by three measurements: two tests at different organic vapor concentrations using a dry air carrier to find V_0 and k_1, and a third test using humidified air to determine the value of Z. This determination of two curves from three values fulfills the goal of developing a procedure in which minimal amounts of data are required to define the system parameters.

As expected, the effect of 100% relative humidity on the adsorption by activated carbon of five water-miscible organic compounds from the vapor phase depended on the adsorbate and its concentration. Over the concentrations studied, the adsorptive capacity of the activated carbon was reduced the greatest for allyl alcohol and the least for piperidine.

The use of this theory to give a semiempirical estimation of the use time of respirator cartridges also was investigated. In the Mecklenberg equation,(19) commonly used to evaluate the performance of respirator cartridges, the effect of changes in the static adsorption coefficient is more important to performance life than similar changes in the rate dependent variables. As it is the change in the static adsorption coefficient as a function of relative humidity that is determined here, it may be possible to use this to estimate the lifetime of respirator cartridges. The procedure used here was to direct the carrier gas (air, either dry or 100% humidified) through a bed of 40 grams of activated carbon in the shape of a respirator cartridge. The time it took in each case for 10% breakthrough of the organic component was measured. Then a rough calculation was made of the time for 10% breakthrough using the equation:

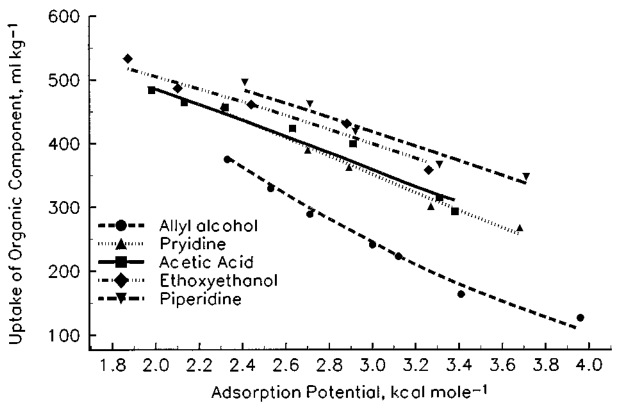

FIGURE 3. Adsorption of organic vapor from dry air versus adsorption potential

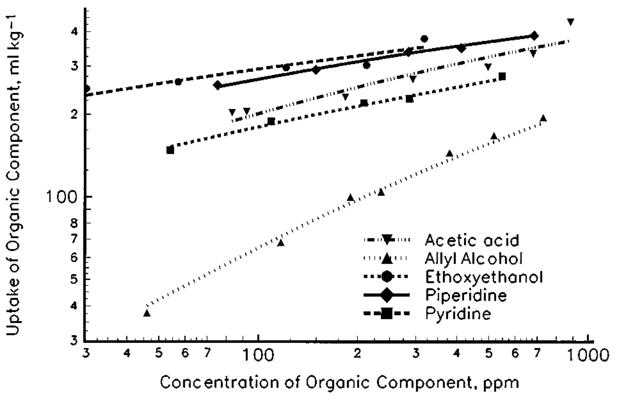

FIGURE 4. Adsorption of organic vapor from air saturated with water vapor versus organic vapor concentration

TABLE IV. Summary of the 10% Breakthrough Times for the Selected Water-Miscible Organic Compounds at 100% Relative Humidity (RH)

		10% Breakthrough Time		
Compound	Concentration (ppm)	Measured at 0% RH	Measured at 100% RH	Predicted at 100% RH
Acetic acid	83	48	38	29
	880	5.6	4.3	4.2
Allyl alcohol	46	20	6.5	7.7
	516	5.5	2.8	2.6
Ethoxyethanol	30	95	47	58
	317	7	5.2	4.7
Piperidine	75	28	24	21
	682	4.4	3.4	3.5
Pyridine	54	40.5	19.5	32
	546	5.5	3.3	3.3

$$t_{10\%\ \mathrm{wet}} = t_{10\%\ \mathrm{dry}}\left(\frac{k_{\mathrm{wet}}}{k_{\mathrm{dry}}}\right) \qquad \textbf{(13)}$$

where the $t_{10\%\ \mathrm{wet}}$ and the $t_{10\%\ \mathrm{dry}}$ are the times required for 10% breakthrough with humidified and dry carrier gas streams, respectively, and the k_{wet} and k_{dry} are the calculated adsorption coefficients at these same conditions, as given in Table I.

Table IV compares the measured 10% breakthrough times with the values estimated using Equation 13. There are some cases in which the values do not coincide, but many of the estimates are surprisingly close. Such estimates could be improved still further if the kinetic component of the Mecklenberg equation could be better defined, but that is beyond the scope of this research.

CONCLUSIONS

The presence of water vapor at saturation in air reduced the adsorptive capacity of the carbon to some extent for all the selected water-miscible compounds.

■ Quantitative estimates based on the Hansen-Fackler equations can be made on the extent of the effect of 100% relative humidity on the vapor phase adsorption of water-miscible organics by activated carbon.

■ The results of this study can be directly applied to the evaluation of respirator cartridge performance under 100% relative humidity.

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