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Parametric Analysis of Phenol Adsorption onto Polymeric Adsorbents

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Distribution equilibria have been investigated for the adsorption of phenol on two macroporous adsorbent resins (Amberlite XAD-4 and Lewatit OC-1064). The experiments were done using a batch technique. The effects of solution pH, temperature, and ionic strength were studied. Results with a new commercial resin (Lewatit OC 1064) have been compared with measurements on the well-established adsorbent resin Amberlite XAD-4. The Langmuir equation was selected to analyze the effect of pH, temperature, and ionic strength on the equilibrium. The influence of the pH has been interpreted in terms of the phenol dissociation. The fraction of free water has been considered in evaluating the influence of the sodium sulfate concentration.

Introduction

In water pollution control, treatment methodologies such as biological degradation, adsorption, ion exchange, chemical oxidation, membrane separations, incineration, and stream stripping, have been applied for the removal of organic contaminant, but the use of polymeric adsorbent resins is an interesting option for recovery where the economics of solvents or chemical regeneration of the adsorbent are favorable (*1−3*).

Only a few of the synthetic adsorbents developed, whose preparation Kunin (*4*) describes, have been employed for adsorption of organic compounds, i.e., Amberlite XAD-2, XAD-4, and XAD-8, Lewatit OC-1064, Duolite S 37, and ES 863 (*5*).

Van Vliet and Weber have studied phenol uptake on some of these commercial adsorbents. In order to compare their behavior, we have calculated the distribution coefficient and the uptake for a phenol concentration of 1 g/L from the phenol load versus concentration plots reported by these authors (*6*). These results are summarized in Table 1 which shows different retentions for the same range of solution concentration. Nevertheless, the resin from Rohm and Haas, Amberlite XAD-4, clearly shows a better behavior than the others.

The values calculated by the authors for XAD-4 resin ($\Delta q/\Delta c$ = 0.240 L·kg^{-1} dry resin, q = 1,002 mol·kg^{-1} dry resin) are similar to those found in the literature. In this work, the authors have extended the experimental condition range (pH, salinity), where no data have been found, and an equilibrium parameter analysis has been done on the basis of the Langmuir equation.

Amberlite XAD-4 and Lewatit OC-1064 have been selected for this study. Amberlite XAD-4 is the best-known polymeric adsorbent for phenol, and measurements on its properties and its application can be found in the literature (*8−11*). Lewatit OC-1064 is a new commercial resin from Bayer AG which appears to be a good adsorbent for phenol. Both are aromatic-based adsorbents which are particularly effective for adsorbing a nonpolar organic from aqueous solutions (*7*).

Experimental Methods

Table 1. Comparative Uptake of Phenol onto Different Polymeric Adsorbents

resin	av distrib coef ($\Delta q/\Delta C$) (L·g^{-1} resin)	q at C = 10.62 mmol·kg^{-1} (mol of phenol·kg^{-1} of resin)
Amberlite XAD-2	0.0787	0.425
Duolite S 37	0.0989	0.425
Duolite ES 863	0.1379	0.637
Amberlite XAD-8	0.2172	0.744
Amberlite XAD-4 (*6*)	0.2979	1.062
Amberlite XAD-4^a	0.270	1.002

^{a} This work.

Table 2. Physical Properties of Amberlite XAD-4 and Lewatit OC-1064

properties	Lewatit OC-1064	Amberlite XAD-4
matrix	polystyrene−divinylbenzene	polystyrene−divinylbenzene
av pore size, Å	50	50
porosity, %	50−55	51
specific surface, m^2·g^{-1}	744^a	750^a
true wet density, g·cm^{-3}	1.010^a	1.020^a
skeletal density, g·cm^{-3}	1.09	1.085
moisture, %	66.51^a	53.84^a
thermal stability, K	274−393	274−393

^{a} Experimental.

The present study reports the adsorptive performance of two synthetic adsorbents, Lewatit OC-1064 and Amberlite XAD-4. Both resins are macroporous, and are based on a matrix structure of cross-linked polystyrene. The averaged and experimental characteristics of both resins are summarized in Table 2 (*6*, *8−11*).

Adsorption isotherms were measured in a batch jacketed contactor of 500 mL capacity (Figure 1). The vessel was glass and contained a temperature controller which acted on the heater of the jacket liquid, and allowed a temperature control ±0.5 K. The contactor was also provided with a pH indicator, of precision 0.001 pH unit, a condenser, and a system for the addition and removal of samples.

The effects of phenol concentration, solution pH, salinity, and temperature were investigated. Experiments were carried out by modifying only one of these variables in each run. The remaining variables were kept constant.

Each subsequent stage involved a modification from the previous state (generally, by the addition of a small amount of solute), and then a new equilibrium state was reached which was characterized by sampling and analyzing the

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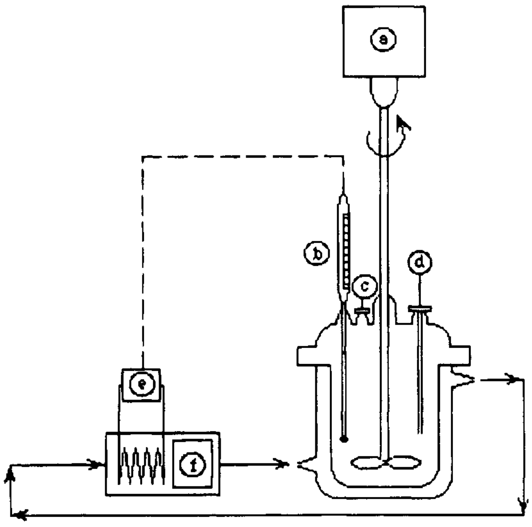

Figure 1. Scheme of the experimental system of adsorption: (a) stirrer; (b) thermometer; (c) inlet/outlet port; (d) pH meter; (e) temperature controller; (f) pump.

phenol concentration (HPLC). The amount of phenol in the resin was calculated by mass balance.

The adsorption isotherms were measured in the absence of solutes other than phenol using the staggered procedure. In this case, 20 min between additions was sufficient for equilibrium. Phenol was added as a concentrated aqueous solution (40 g·L^{-1}).

The solution pH was changed by the addition of 0.1 M sodium hydroxide, and the effect of the ionic strength was measured by the addition of known masses of sodium sulfate. Three isotherms were studied.

Comparative Analysis of Phenol Adsorption Isotherms

Results. Figure 2 shows the adsorption isotherms of phenol on OC-1064 and XAD-4 resins. Experimental results were fitted to the Langmuir equation (eq 1), where

$$q = \frac{KQc}{1 + Kc} \qquad (1)$$

c is the molality of phenol (mol·kg^{-1} water), K is the Langmuir constant (kg·mol^{-1}), Q is the maximum adsorption capacity (mol·kg^{-1} dry resin), and q is the amount absorbed in the solid phase (mol·kg^{-1} dry resin). Possible pH changes, as a result of the high adsorption of phenol on these sorbents, were avoided by using high ratios of solution volume to adsorbent. Within the range of concentration studied it appears that the resin XAD-4 shows that higher amounts of phenol are retained on its surface.

Measurements of phenol adsorption were made at three temperatures. Experimental results are given in Table 3. The repeatability of the measurement data are also given in the table.

These results have been compared with those found in the literature for the same experimental conditions (*3*, *6*, *8*). All the authors report a Langmuir shape isotherm for phenol adsorption on XAD-4. In all cases, even ours, the results are very close: retention ranges around 1 mol·kg^{-1} dry resin for a phenol concentration of 1 g·L^{-1}, e.g., Van Vliet et al. (*6*) report exactly a retention of 1.06 mol·kg^{-1} dry resin. These results are summarized in Table 1.

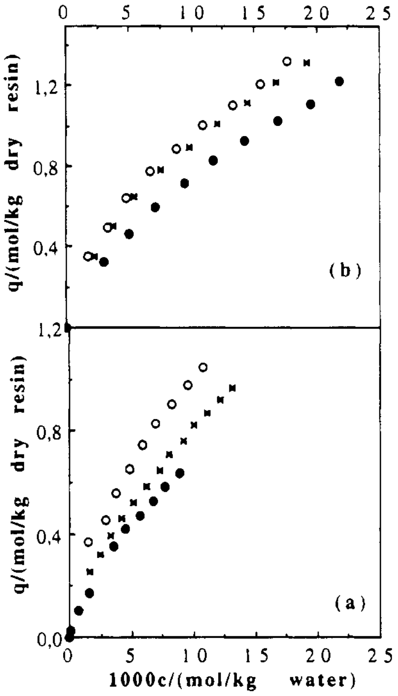

Figure 2. Adsorption isotherms of phenol on the (a) OC-1064 resin at pH 5.73 (○, 293 K; *, 303 K; ●, 323 K) and (b) XAD-4 resin at pH 5.73 (○, 307 K; *, 327 K; ●, 341 K). Solid lines represent the best fit of eq 1, and symbols represent experimental results.

Table 3. Adsorption Isotherms of Phenol on the OC-1064 and Resins at pH 5.73

a. OC-1064

$T = 307$ K			$T = 327$ K			$T = 341$ K		
C	q_{exp}	q_{calc}	C	q_{exp}	q_{calc}	C	q_{exp}	q_{calc}
0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.538	0.367	0.289	1.626	0.251	0.207	0.106	0.025	0.014
2.848	0.455	0.471	2.497	0.321	0.301	0.723	0.101	0.089
3.751	0.557	0.573	3.273	0.393	0.376	1.562	0.171	0.179
4.782	0.651	0.672	4.197	0.459	0.456	3.496	0.354	0.345
5.812	0.743	0.756	5.154	0.523	0.531	4.516	0.417	0.415
6.907	0.830	0.832	6.131	0.584	0.600	5.632	0.472	0.481
8.150	0.907	0.906	7.226	0.648	0.670	6.694	0.527	0.537
9.446	0.978	0.973	7.980	0.708	0.714	7.651	0.585	0.581
10.73	1.046	1.029	9.106	0.761	0.773	8.788	0.634	0.627

b. XAD-4

$T = 298$ K			$T = 303$ K			$T = 323$ K		
C	q_{exp}	q_{calc}	C	q_{exp}	q_{calc}	C	q_{exp}	q_{calc}
0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.721	0.350	0.287	2.200	0.348	0.312	2.985	0.323	0.290
3.220	0.499	0.477	3.719	0.504	0.479	4.909	0.464	0.440
4.771	0.643	0.634	5.419	0.650	0.633	7.044	0.593	0.583
6.652	0.772	0.786	7.438	0.781	0.781	9.361	0.712	0.714
8.788	0.888	0.923	9.776	0.894	0.919	11.68	0.827	0.827
10.91	1.003	1.031	12.02	1.008	1.028	14.27	0.928	0.934
13.26	1.106	1.128	14.41	1.114	1.124	16.90	1.022	1.029
15.51	1.210	1.205	16.77	1.218	1.203	19.55	1.113	1.111
17.63	1.320	1.265	19.22	1.315	1.272	21.78	1.220	1.172

Parameters of the Langmuir Equation. The results were treated in terms of the Langmuir equation. which was found to provide a good description of the results for both adsorbents. Phenol retention, q, is given as an explicit function of its solution concentration, c. Other equations that have been used include the Freundlich, BET, and Van Vliet equations (*6*). However, the Langmuir equation was selected because its parameters, equilibrium constant and adsorption capacity, could be evaluated from each measurement and later be modified to take into account the effect of pH and ionic strength. In Figure 2, the fitting of the model to the experimental data is shown in solid lines, and the quality of the fitting is indicated in Table 4 by the

Table 4. Langmuir Equation Parameters

	T/K	K (kg·mol^{-1})	$E_1{}^a$/%	$E_2{}^b$/(mol·g^{-1})
Lewatit OC-1064	293	0.267	1.132	0.008 63
(Q = 2.1 mol·kg^{-1})	303	0.143	1.805	0.005 10
	323	0.133	5.277	0.002 56
Amberlite XAD-4	307	0.159	0.996	0.010 11
(Q = 2.5 mol·kg^{-1})	327	0.105	0.998	0.007 35
	341	0.068	0.998	0.006 43

[a] $E_1 = (1/n)\Sigma_n^1|q_{exp} - q_{calc}|/q_{exp} \times 100$. [b] $E_2 = [(\Sigma_1^n q_{exp} - q_{calc})^2]^{1/2}/n$.

Table 5. Evaluation of the Phenol Uptake with Temperature onto OC-1064 (c = 3.02; L/S = 27.92 L·kg^{-1}; E_1 = 1.557; E_2 = 0.0066) and XAD-4 (c = 2.33; L/S = 19.95 L·kg^{-1}; E_1 = 3.842; E_2 = 0.0055) Resins

OC-1064				XAD-4			
T/K	c	q_{exp}	q_{calc}	T/K	c	q_{exp}	q_{calc}
295.00	2.507	0.546	0.521	298.00	2.231	0.416	0.423
297.00	2.507	0.547	0.502	303.00	2.369	0.409	0.393
303.00	3.092	0.543	0.532	308.00	2.571	0.399	0.374
308.00	3.336	0.528	0.519	313.00	2.890	0.383	0.368
313.00	3.559	0.513	0.503	318.00	3.230	0.367	0.362
318.00	3.750	0.519	0.484	323.00	3.315	0.363	0.331
323.00	3.963	0.488	0.467	328.00	3.740	0.342	0.330
328.00	4.303	0.460	0.462	333.00	4.037	0.327	0.318
333.00	4.356	0.464	0.433				
338.00	4.760	0.438	0.433				
343.00	5.153	0.413	0.430				
348.00	5.366	0.397	0.415				
353.00	5.780	0.375	0.412				

magnitude of the Euclidean norm of the deviation (E_1) and the average relative deviation (E_2).

The standard deviations between experimental and calculated retention, determined as

$$\delta_i = 100\left[\frac{1}{n}\sum_1^n\left(\frac{q_{calc} - q_{exp}}{q_{exp}}\right)^2\right]^{1/2} \qquad (2)$$

are satisfactory. For all runs, the standard deviations between the measured and calculated values are less than 7%.

The equilibrium constants decrease with temperature, and the adsorption enthalpies derived from the Arrhenius relation are −19.6 and −17.1 kJ·mol^{-1} for XAD-4 and OC-1064, respectively. Results have been fitted using an average value of capacities, Q, calculated for each resin from the low concentration results. The value of adsorption capacity, Q, reported in Table 4 can be larger, which is due to the fact that phenol gives rise to nonrestricted multilayer adsorption at high solution concentration.

The effects of temperature are given in Table 5. The results of phenol adsorption as a function of temperature (Table 5) were fitted to eq 3, which can be derived from

$$\ln\left[\frac{q}{(Q - q)c}\right] = \ln K_o - \frac{\Delta H}{RT} \qquad (3)$$

the Langmuir equation by taking into account the Arrhenius relation. The adsorption enthalpies, ΔH, are 19.6 kJ·mol^{-1} for OC-1064 and 21.2 kJ·mol^{-1} for XAD-4, and the preexponencial constants, K_o, calculated from the y-axis intercept are 5.8 × 10^{-5} kg·mol^{-1} for OC-1064 and 1.26 × 10^{-5} kg·mol^{-1} for XAD-4. The standard deviation is 3.9% for the OC-1064 resin and 4.3% for XAD-4.

Table 6. Influence of pH on Phenol Adsorption onto the Resins OC-1064 (c = 3.08; L/S = 27.81 L·kg^{-1}; T = 295 K; E_1 = 6.0255; E_2 = 0.0093) and XAD-4 (c = 2.5; L/S = 20.78 L·kg^{-1}; T = 295 K; E_1 = −7.659; E_2 = 0.0104)

OC-1064				XAD-4			
pH	c	q_{exp}	q_{calc}	pH	c	q_{exp}	q_{calc}
5.900	2.837	0.434	0.462	7.030	2.1030	0.426	0.425
6.200	2.837	0.434	0.462	8.500	2.1250	0.425	0.418
6.700	2.837	0.434	0.462	8.950	2.2840	0.417	0.424
7.230	2.847	0.434	0.463	9.340	2.4970	0.406	0.414
7.500	2.847	0.434	0.462	9.620	2.7620	0.393	0.396
8.050	2.868	0.432	0.462	9.700	3.1870	0.373	0.426
8.120	2.762	0.433	0.447	9.960	3.7430	0.345	0.396
8.350	2.815	0.436	0.451	10.150	4.3460	0.321	0.367
8.450	2.943	0.433	0.465	10.430	5.2490	0.270	0.296
8.640	2.953	0.435	0.461	10.730	6.3430	0.216	0.212
8.850	3.187	0.423	0.480	11.210	7.5970	0.153	0.097
9.100	3.432	0.408	0.489	11.520	8.8620	0.087	0.058
9.560	3.995	0.393	0.470	11.920	8.8620	0.052	0.023
9.910	4.579	0.362	0.416	12.140	9.5100	0.051	0.020
10.120	5.759	0.328	0.410				
10.400	6.641	0.261	0.314				
10.730	7.374	0.211	0.208				
10.940	8.171	0.169	0.154				
11.260	8.978	0.122	0.088				
11.630	9.244	0.076	0.040				
11.900	9.403	0.061	0.022				
12.260	9.488	0.050	0.010				
12.300	9.487	0.046	0.009				

Solution pH. Phenolic compounds are weak acids, and their nonionic and dissociated forms show different adsorption behavior. The pH of the solution can affect both the phenol speciation and also the resin structure. pH affects weak ion exchange resins, improving or depleting the ion uptake and changing the functional groups and the resin structure (*12*). For adsorbent resins, there is no detailed description of the effect on surface area, pore size distribution, or surface polarity. From the results summarized in Table 6 adsorption is high in acid solutions of phenol, where the nonionic species predominates, and low in basic solutions. Hence, if adsorption is conducted at several pH units below the pK_a value of the compound to be retained, to achieve good efficiencies separation processes can be achieved.

The effect of the solution pH on phenol adsorption is independent of the type and characteristics of the adsorbent. The adsorption curves for XAD-4 and OC-1064 can be overlaid, and both curves are nearly coincident.

The concentration of undissociated phenol c can be calculated from the concentration of phenol measured analytically by HPLC, and $c°$ (mol·kg^{-1} of water), by eq 4,

$$c = \alpha c° \qquad (4)$$

where α is a function of hydrogen ion concentration, c_{H^-}, and the dissociation constant of phenol, K_a:

$$\alpha = \left(\frac{K_a}{c_{H'}} + 1\right)^{-1} \qquad (5)$$

Introducing this factor into eq 1, an apparent equilibrium constant, K', can be derived

$$K' = \alpha K = \frac{K}{(1 + K_a/c_{H'})} \qquad (6)$$

so

$$\log(K/K' - 1) = \mathrm{pH} - \mathrm{p}K_a \qquad (7)$$

with

$$K' = \frac{q}{(Q - q)c} \qquad (8)$$

Then, $\log(K/K' - 1)$ can be plotted against pH as shown

Table 7. Effect of the Ionic Strength on Phenol Adsorption of OC-1064 (c = 7.00; L/S = 11.37 L·kg^{-1}; T = 295 K; E_1 = 9.183; E_2 = 0.0791 and XAD-4 (c = 2.13; L/S = 20.00 L·kg^{-1}; T = 295 K; E_1 = −0.2437; E_2 = 0.0126) Resins

OC-1064					XAD-4				
$1000x_s$	c	q_{exp}	β	q_{calc}	$1000x_s$	c	q_{exp}	β	q_{calc}
0.000	7.080	0.782	160	0.818	2.110	1.562	0.424	77.50	0.409
0.401	6.558	0.794	119.3	0.804	3.980	1.413	0.431	56.05	0.409
0.774	6.513	0.792	98.04	0.812	5.760	1.275	0.437	40.33	0.438
1.150	6.269	0.795	84.04	0.816	7.560	1.285	0.451	33.55	0.437
1.490	6.237	0.792	75.02	0.818	9.720	1.168	0.457	32.44	0.427
1.820	6.110	0.791	68.35	0.820	12.20	1.062	0.462	31.38	0.420
2.120	5.884	0.803	63.51	0.773					
3.830	5.610	0.815	47.39	0.783					
7.290	5.408	0.823	35.12	0.834					
9.180	5.047	0.840	31.94	0.837					
10.90	4.887	0.846	29.93	0.838					
13.20	4.346	0.867	28.00	0.849					
15.50	4.037	0.879	26.60	0.853					
17.70	3.953	0.883	25.59	0.846					
19.93	3.825	0.889	24.78	0.846					

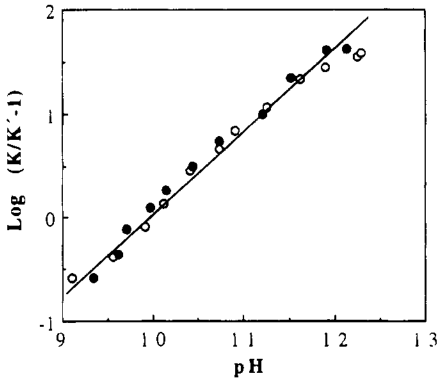

Figure 3. Linearized form of eq 6: ○, OC-1064; ●, XAD-4.

in Figure 3 to check this speciation approach on the Langmuir model (eq 7). The pK_a value was estimated from the y-axis intercept, resulting in pK_a = 9.95 which compares well with the literature value which is pK_a = 9.99 (*13*). Figure 3 shows that the pH does not have an influence on the resin structure or function, because the results fit the linear plot proposed by the acid speciation model. A comparison of the prediction from the model is given in Table 6. The comparison is based on E_1 and E_2. The standard deviation for the total amount of phenol adsorbed is 11% for the OC-1064 resin and 7% for XAD-4.

Sodium Sulfate Concentration. Although strong electrolytes are not adsorbed, and do not modify the adsorbent, the presence of an electrolyte such as sodium sulfate in an aqueous system enhances phenol adsorption (*3, 8, 14*). This is probably due to a "salting out" effect in the aqueous phase. The fraction of free water in solution decreases with an increase in salt content as a consequence of the ion solvation, causing an effective increase in the phenol concentration that can be estimated from eq 9.

$$c = \frac{c^\circ}{1 - \beta x_s} \tag{9}$$

Here c is the molality of the undissociated form of phenol. This equation has been derived by considering the solvation of water, using the term β, which is the amount of water bound to sodium sulfate.

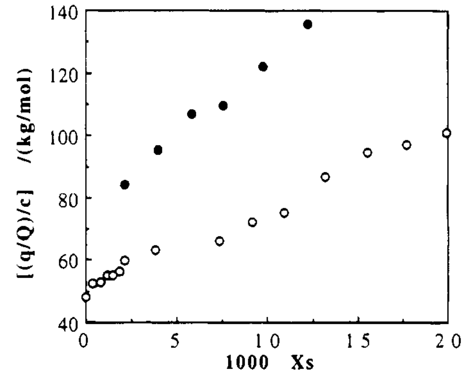

Figure 4. Effect of the ionic strength on phenol adsorption for the resins OC-1064 (c° = 7.00; liquid/solid = 11.37 L·kg^{-1}; T = 295 K) and XAD-4 (c° = 2.13; liquid/solid = 20.00 L·kg^{-1}; T = 295 K). The solid lines represent the correlation description of the data: ○, OC-1064; ●, XAD-4.

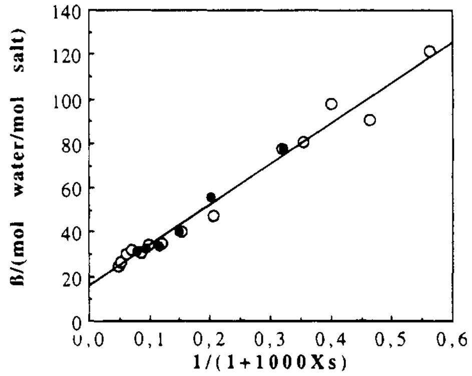

Figure 5. Solvation factor: ○, OC-1064; ●, XAD-4. The solid line represents the correlation of the data with eq 11.

The distribution coefficient of phenol between phases is shown in Figure 4. Experimental and predicted values are given in Table 7, for both resins, as a function of the sodium sulfate concentration. This improvement of the adsorption for both resins indicates a solution effect. So, the concentration of phenol, c, can be described as a function of the solvation factor β (mol of associated water·mol^{-1} of salt) and the molar ratio of sodium sulfate in the solution, x_s (mol of Na_2SO_4·mol^{-1} of water). Introducing eq 9 into eq 1, an alternative form of the Langmuir equation containing the apparent equilibrium constant K'' can be derived.

$$K'' = \frac{K}{1 - \beta x_s} \tag{10}$$

Figure 5 shows that the average number of water molecules associated to the electrolyte depends on the electrolyte concentration. As the electrolyte concentration increases, the average number of water molecules surrounding the electrolyte decreases. The relationship between the solvation factor, β, and the salt concentration, x_s, can be expressed by

$$\beta = \beta_\infty + \frac{\beta_0}{1 + x_s} \tag{11}$$

The solvation factor at high concentrations, β_∞, and the solvation factor at low concentrations, β_0, can be derived from the regression of the results. These values are β_∞ = 15.8 mol of water·mol^{-1} of salt and β_0 = 182 mol of water·mol^{-1} of salt, and are independent of the resin used. The standard deviations between experimental and calculated values are 11% for the OC-1064 resin and 7% for XAD-4.

General Equation for the Prediction of Phenol Adsorption

On the basis of these measurements a modified Langmuir equation (eq 12) is proposed to predict the equilibrium in systems where the pH, salinity, and temperature can change. Two of these variables are exclusively related to

$$q = \frac{K(T)Qc^\circ}{(1 - \beta x_s)/\alpha + K(T)c^\circ} \tag{12}$$

the sorbate concentration, and the Langmuir constant is linear in the temperature term. The basic Langmuir equation applies only to neutral or undissociated phenol. Equation 12 can be derived from eqs 4, 8, and 10.

In Figure 6, the effect of pH is shown. To maintain the same loading ratio, q/Q, for an increase of pH, a higher solution concentration will be required, as indicated by the upward displacement along the lines shown in Figure 6. Temperature and salinity effects are also shown in this figure for two loading ratios, 0.6 and 0.4, respectively, at three values of each variable. Basically, phenol adsorption can be conducted using these variables, so this equation could be a useful tool not only for predicting loading data during adsorption operation but also for calculating the phenol distribution in the stripping step.

Salinity also changes the amount of adsorbed phenol, but high concentrations are required. The addition of salt prior to an adsorption step is being used in industrial processes to improve phenol recovery from waste waters. Many others industrial wastes already contain high amounts of sodium sulfate as a consequence of the production process.

This analysis has been carried out with the Langmuir equation. Other equilibrium relations could have been considered. However, the former equation has a restricted applicability to low concentration where adsorption takes place, forming a monolayer. This restriction could be avoided by using the BET isotherm.

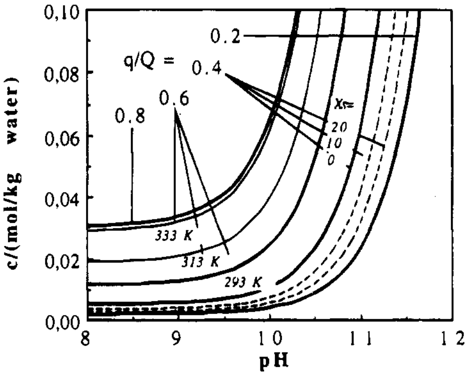

Figure 6. Prediction of phenol uptake.

Although the resins tested have basically the same composition, there are observed differences in the adsorption of phenol that are probably due to their internal microstructure. The values reported in the paper of the specific surface, Q and adsorption constant, K, indicate that the strength of the interaction between the bound sorbate and surface is clearly different in both resins. This difference gives rise to an interesting comparative behavior.

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