$$\log K = 6.7369 - (770.5/T) - 0.01986T \quad (11)$$

which correlates the data of Campbell and Maass (1930), Morgan and Maass (1931), and Wang and Himmelblau (1964) very well.

The predicted values of the physical properties for the sulfur dioxide-water system are listed in Table 1.

Comparison of Experimental Results with Theory

The experimental results are shown in Figures 1 and 2 as the plots of the absorption rate N_A of sulfur dioxide against the reciprocal square root of the exposure time $1/\sqrt{t}$. The solid lines in these figures represent the theoretical lines for absorption with an instantaneous reversible reaction, calculated from Equations (8) and (10) with $A_0 = 0$ using the physical properties predicted by the above-mentioned methods, while the dashed lines show the theoretical lines for physical absorption, calculated from the Higbie equation, that is, Equation (8) with $A_0 = 0$ and $\beta = 1$. As can be seen in these figures, the measured values of the absorption rate are in good agreement with the theoretical lines.

NOTATION

A = concentration of sulfur dioxide in solution, g-mole/l
A_i = interfacial concentration or physical solubility of sulfur dioxide in solution, g-mole/l
A_0 = concentration of sulfur dioxide in bulk of solution, g-mole/l
D_A = liquid-phase diffusivity of sulfur dioxide in solution, cm^2/s
D_E, D_F = effective diffusivities of H^+ and HSO_3^- ions in solution, cm^2/s
E, F = concentrations of H^+ and HSO_3^- ions in solution, g-mole/l
E_0, F_0 = concentrations of H^+ and HSO_3^- ions in bulk of solution, g-mole/l
K = equilibrium constant of reaction (1), g-mole/l
N_A = average rate of absorption of sulfur dioxide, g-mole/(cm^2)(s)
T = absolute temperature of solution, °K
t = exposure time of liquid to gas, s
x = distance from interface into liquid, cm
β = reaction factor

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Kinetic Behavior of Weak Base Anion Exchangers

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In this work, twelve different weak base anion exchangers were studied as to their decrease of breakthrough capacity with increasing specific loading. From the curves, the rate coefficients were calculated and correlated with the values characterizing the ion exchanger. It was found that the sorption rate of acid increases with decreasing average molality of functional groups in the ion exchanger.

SCOPE

The present tendency in the demineralization of water by means of ion exchange resins is to operate at a high specific loading of the resin. For this purpose, only ion exchangers with good mechanical-osmotic stability and with the best kinetic properties can be used. The strong electrolyte resins exhibit an ion exchange rate sufficiently high so that their breakthrough capacity decreases relatively slowly with increasing specific loading. On the other hand, the slower kinetics of weak electrolyte resins results in a considerable drop of breakthrough capacity at

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higher flow rates. For this reason, the weak-base anion exchangers, which usually represent the kinetically weakest point in a demineralization plant, will be discussed in the present study.

The purpose of this work was to compare various types of weak base resins with regard to their applicability for higher specific loading. The determination of breakthrough capacity dependence on the specific loading is rather lengthy, whereas the exact measurement of acid sorption rate requires the skill and experience. For this reason, we tried to establish the criterion for estimating whether a resin is suitable for higher specific loading or not.

CONCLUSIONS AND SIGNIFICANCE

We found that the sorption rate of acid increases with decreasing internal molality of functional groups. This value can be easily calculated from the values of total water content and total weight capacity of the ion exchanger. In this way we can estimate the kinetic properties of an unknown or a new resin.

With styrene-DVB weak base resins, the sorption rate considerably increases with increasing porosity because a higher porosity leads to a higher water content and thus to a higher molality of functional groups. In the case of acrylic resins, the molality of functional groups may be high enough even for gel type of resins, and so the increasing porosity does not necessarily mean the higher sorption rate.

The dependence of the sorption rate on the internal molality of functional groups does not apply to those resins where the sorption rate is influenced by the chemical reaction of the functional group and not only by the diffusion rate of acid in the resin particle. This may be the case of gel type of weak base resins at a low acid concentration, and hence the simple relation between the sorption rate and the molality of functional groups does not hold. These resins, however, are not much in use any longer.

The uptake of strong acids by weak base anion exchangers can be described by the equation

$$\overline{R_3N} + H_3O^+ + Cl^- \rightleftarrows \overline{R_3NH^+} + \overline{Cl^-} + H_2O$$

in which the resin phase is indicated by barred symbols.

The mechanism of acid neutralization by weak base anion exchangers is completely different from that by strong base anion exchangers. With strong base anion exchangers, the functional groups are completely ionized, the concentration of H^+ ion in the particle is negligible in the range of low acid concentrations, and consequently the rate controlling step is either the diffusion of acid across the liquid film surrounding the particle or the interdiffusion of OH^- and Cl^- ions in the particle with subsequent neutralization of the OH^- by the H^+ ion at the resin particle surface. With weak base anion exchangers, the content of strong base groups is usually low so that the interdiffusion of OH^- and Cl^- ions would be very slow. The conversion of weak base resin into the salt form thus requires the protonization of functional groups by the acid from the solution. The uptake of acids by weak base anion exchangers is consequently sorption rather than ion exchange.

Helfferich (1965) proposed the mechanism for the ionization of undissociated functional groups by the reaction with coion, which can be applied to weak base resins. Following that mechanism, the sorption rate depends on the extent of acid invasion of the resin shell in the salt form by the acid from the solution; when having crossed the shell, the H^+ ion protonizes the functional group and the anion of the acid balances the newly formed positive charge. The boundary between the shell and inner core remains sharp and retreats toward the particle center. The sorption rate is proportional to the diffusion rate of the H^+ ion in the resin and increases greatly with increasing solution concentration. For this mechanism, the following rate equation was derived:

$$\frac{dF}{dt} = \frac{3\overline{D}_H\bar{c}_H^{\,o}}{X \cdot r_o^2[(1-F)^{-1/3} - 1]} \qquad (1)$$

The sorption rate of acid is thus proportional to the concentration of the H^+ ion within the resin at the particle surface and inversely proportional to the molarity of functional groups and to the square of particle diameter. The swelling changes during sorption are neglected.

Adams et al. (1969) discussed in detail the uptake of acids by weak base anion exchangers. They proved the sorption rate of weak base gel type of anion exchangers with a low cross-linking independent of the particle diameter. It means that the rate controlling step is the chemical reaction of the functional group. They confirmed Helferich's mechanism for macroporous resins above 0.001 N acid concentration and for gel type of weak base anion exchangers with low cross-linking at high acid concentration (above 0.1 N). Bolto and Warner (1970) confirmed the validity of Helferich's mechanism for sixteen different weak base resins.

Modern weak base resins are produced mostly with macroporous styrene-DVB matrix mainly for the reason of reversible sorption and desorption of organic matter from treated water. The weak base anion exchangers with acrylic skeleton, either gel type or macroporous, have been produced, also exhibiting high efficiency of desorption of organic matter. With all these resins, the influence of the chemical reaction of the functional groups on the sorption rate can be neglected.

From Equation (1) it results that the acid concentration $\bar{c}_H^{\,o}$ influences the sorption rate as much as the diffusion properties of the resin. Its ratio to the acid solution concentration $\bar{c}_H^{\,o}/c_H$ is determined by Donnan equilibrium. When a weak base resin is in free base form, the invasion of the resin particle by the acid from the solution takes place, and the acid concentration within the resin will be the same as that in the solution. But as soon as the resin shell in the salt form is formed, the acid will be partially excluded from the resin particle, and the acid concentration within the resin at the particle surface will be lower than that in the solution. The influence of Donnan potential increases with decreasing acid solution concentration. Glueckauf (1962) found

that the value of acid concentration within the resin at the particle surface can be depicted by the empirical relation $\bar{c}_H^o/c_H = k \cdot c_H^{1-z_G}$, where k and z_G are empirical constants with z_G between zero and unity. The extent of acid invasion is thus characterized by this ratio after the shell has been formed.

The acid invasion increases with decreasing crosslinking in the case of gel type of styrene-DVB weak base anion exchangers. In the case of macroporous, weak base resins, the acid invasion is very low in the densely crosslinked areas with high charge density, whereas in the pores the influence of Donnan potential is reduced. The average value of acid invasion over the whole volume of resin particle thus increases as the macroporosity increases.

The absolute value of $\bar{D}_i$ is very difficult to find, and so it is usually related to the diffusion coefficient of the species i in the solution. Some empirical relations have been derived, for example, the relation $\bar{D}_i = D_i\epsilon/2$ (Wheeler, 1951), where ϵ is the fractional pore volume. Therefore, it is possible to estimate the value of the diffusion coefficient in the particle.

When deducing the dependance of the breakthrough capacity of the resin bed on the specific loading, we started from the material balance and the rate equation depicting the kinetics of the process. We considered an infinitesimal layer dz in the boundary in the column where the degree of exhaustion F changes by dF (Figure 1). If we take the boundary in the column as the frame of reference, then ion exchange moves upward at a rate $v_B = \dfrac{v}{\dfrac{\bar{X}}{c_H} + \beta}$, and the solution downward at a rate $v - v_B$.

From the material balance

$$q \cdot v_B (F_{z+dz} - F_z) = q \cdot dz \cdot \left(\frac{\partial F}{\partial t}\right)_z \tag{2}$$

Substituting $(\partial F/\partial t)_z$ by Equation (1) and for $\bar{c}_H^o = k \cdot c_H^{2-z_G}$, we obtain after the integration for boundary conditions $z = 0$, $F = 0$:

$$z = \frac{v_B \cdot \bar{X} \cdot r_o^2}{3\bar{D}_H \cdot k \cdot c_H^{2-z_G}} \left[\frac{3}{2} - F - \frac{3}{2}(1-F)^{2/3}\right] \tag{3}$$

In Figure 1 the area P is proportional to the amount of nonexhausted functional groups when the breakthrough of acid into the effluent occurs:

$$P = \int_0^1 z dF = \frac{v_B \cdot \bar{X} \cdot r_o^2}{30\bar{D}_H \cdot k \cdot c_H^{2-z_G}} \tag{4}$$

and

$$\frac{\bar{X}_s}{\bar{X}} = \frac{Z - P}{Z} \tag{5}$$

For dilute solutions $c_H << \bar{X}$

$$\bar{X}_s = \bar{X} - \frac{r_o^2 \cdot \bar{X}}{30\bar{D}_H \cdot k \cdot c_H^{1-z_G}} \cdot s \tag{6}$$

When $X_s = C_s/1 - \beta$ and $X = C_o/1 - \beta$

$$C_s = C_o - \frac{r_o^2 \cdot C_o}{30\bar{K}_D} \cdot s \tag{7}$$

where $\bar{K}_D = \bar{D}_H \cdot k c_H^{1-z_G}$

EXPERIMENTAL

We tested twelve different weak base resins. The tests were performed in laboratory columns 23 diam containing 100 ml of the resin in free base form and of standard particle size. The influent solution was 0.02 N HCl at specific loading 20, 40, 60, and 90 bed volumes/hr, the regeneration level was 1.6 eq/l sodium hydroxide resp. Na_2CO_3. This level was found to be sufficient for good quality of treated water, except for Amberlite IR 45 where it was necessary to use a level 2.0 eq/l. The cycles were performed beyond breakthrough point to assure approximately the same exhaustion of the bed. The breakthrough capacities relate to 1% breakthrough point.

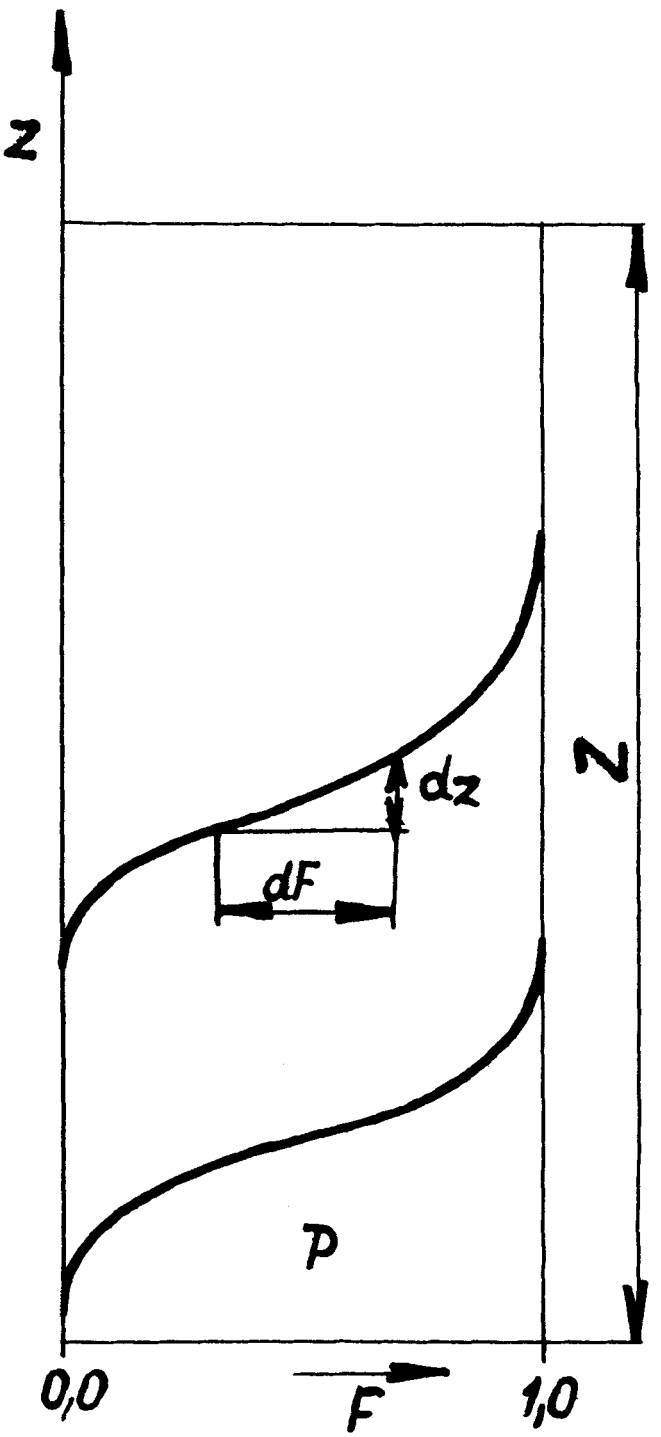

Fig. 1. Transient boundary in the column.

With Amberlite IRA 93 we also tested the effect of SO_4^{2-} ions in the influent.

The water content values and the weight capacities of the resins were determined by standard methods (Helfferich, 1962). For r_o we took weight average particle diameter (Millar et al., 1962).

DISCUSSION

Weak base anion exchangers are usually regenerated by the lowest regeneration level possible, dictated by the regenerant economy, sufficient to achieve good quality of treated water. At these regeneration levels, the weak base resin is not completely converted to the free base form. The extent of regeneration depends at a given regeneration level on the total volume capacity and on the amount of strong base groups. The regeneration level in this work was chosen to correspond to the regeneration level used in practice.

Equation (7) has been derived on the assumption that the ion exchanger is in the free base form only. In the case of weak base resins not fully regenerated, and if we suppose that the remaining functional groups in the salt form do not affect the sorption, we can use for C_o the value of capacity extrapolated to the specific loading $s = 0$ bed volumes/hr which corresponds to the amount of regenerated functional groups.

For this reason it was necessary to achieve the same extent of exhaustion of the bed with increasing specific loading. This was no problem with the weak base anion exchangers with sharp boundary in the column. In the case of ion exchangers with a less sharp boundary (Lewatit MP 62, Wofatit AD 41), the same extent of exhaustion of the bed may not have been achieved. For this reason, the coefficients $\overline{K}_D$ were calculated from the region of approximately linear dependance between the specific loading $s = 60$ and 20 bed volumes/hr for these resins and between $s = 90$ and 20 bed volumes/hr for other resins.

The values of $\overline{K}_D$ calculated from the curves in Figure 2 are in accordance with the results obtained by kinetic measurement. Bolto and Warner (1970) found the value 61.10^{-7} cm²/s for Amberlite IRA 93 and 93.10^{-7}cm²/s for Amberlite IRA 68 (in our case $57.4 \cdot 10^{-7}$cm²/s and $84.5 \cdot 10^{-7}$ cm²/s, respectively).

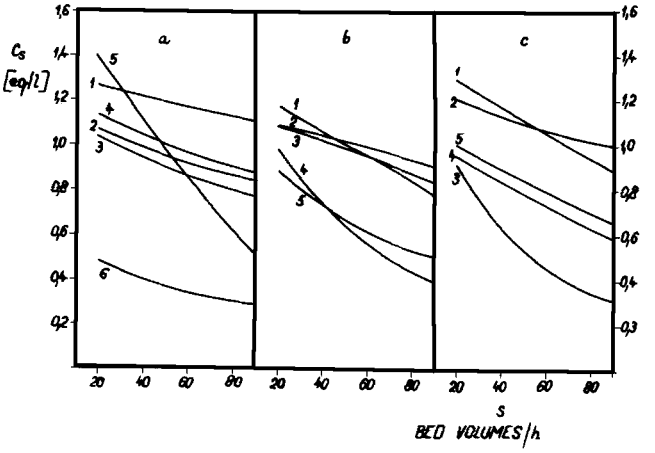

Fig. 2. Breakthrough capacity as a function of specific loading.

- a 1 Amberlite IRA 68
- 2 Amberlite IRA 94S
- 3 Amberlite IRA 93
- 4 Amberlite IRA 93 (50% SO_4^{2-})
- 5 Amberlite IR 45
- 6 Zerolit HIP
- b 1 Lewatit HL 9247
- 2 Lewatit CA 9222
- 3 Lewatit MP 64
- 4 Lewatit MP 62
- 5 Lewatit CA 9249
- c 1 Wofatit Y 17 reg. NaOH co-current
- 2 Wofatit Y 17 reg. Na_2CO_3 countercurrent
- 3 Wofatit AD-41 reg. NaOH cocurrent
- 4 Wofatit AD 41 reg. Na_2CO_3 cocurrent
- 5 Wofatit AD 41 reg. Na_2CO_3 countercurrent

From Tables 1 and 2 it can be seen that the rate of acid sorption for weak base anion exchangers increases approximately with increasing water content. This value is not sufficient to characterize the kinetic properties of the resin (Lewatit MP 64 and Amberlite IRA 93 exhibit about the same rate, whereas their water content values are different).

When plotting the values of $\overline{K}_D$ coefficients vs. those of the molality of functional groups of ion exchanger in free base form in milliequivalents per gram water ($m = \frac{100 - W}{W} \cdot C_W$), we obtain the curve in Figure 3.

TABLE 1

Resin mark	Type	W (wt %) f.b.	W (wt %) Cl^-	C_W (meq/g) f.b.	C_W (meq/g) Cl^-	m (meg/g) f.b.	m (meg/g) Cl^{-1}
Amberlite							
IRA 93	Macroporous	54.5	60.3	4.70	4.03	3.92	2.64
IRA 94S	Macroporous	56.0	59.5	4.60	3.92	3.61	2.67
IRA 68	Acryl. gel	60.0	62.0	5.30	4.44	3.53	2.73
IR 45	Gel	43.0	44.0	5.60	4.65	7.42	5.90
Lewatit							
MP 62	Macroporous	45.5	52.5	5.10	4.30	6.11	3.89
MP 64	Macroporous	60.0	64.5	5.75	4.75	3.83	2.62
CA 9247	Macroporous	57.0	63.5	5.60	4.65	4.22	2.67
CA 9249	Acryl. macrop.	56.0	60.0	5.12	4.31	4.02	2.87
CA 9222	Acryl. macrop.						
Wofatit							
AD 41	Macroporous	47.5	53.5	4.80	4.08	5.30	3.55
Y 17	Acrylic gel	57.0	58.2	5.70	4.72	4.30	3.37
Zerolit HIP	Isoporous	52.0	52.0	4.70	4.00	4.34	3.68

TABLE 2

Resin mark	r_0 (mm)	C_0 (meq/cm³)	$\overline{K}_D \cdot 10^7$ (cm²/s)	
Amberlite				
IRA 93	0.45	1.18	57.4	
IRA 94S	0.42	1.20	62.4	
IRA 68	0.40	1.30	84.5	
IR 45	0.27	1.70	9.9	2,0 eq/l NaOH
IRA 93	0.45	1.26	61.2	50% SO_4^{2-}
Lewatit				
MP 62	0.36	1.25	13.05	
MP 64	0.44	1.16	58.2	
CA 9247	0.42	1.28	36.6	
CA 9249	0.47	0.97	37.5	
CA 9222	0.39	1.16	49.7	
Wofatit				
AD 41	0.40	1.32	16.3	
		1.13	31.7	Na_2CO_3 cocurrent
		1.17	32.8	Na_2CO_3 countercurrent
Y 17	0.40	1.41	35.7	
		1.31	56.6	Na_2CO_3 countercurrent
Zerolit HIP	0.42	0.58	36.8	

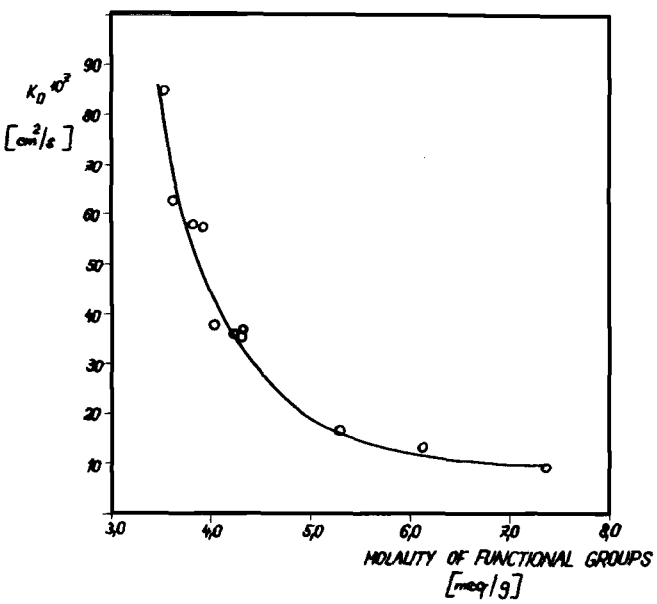

Fig. 3. Dependence of $\overline{K}_D$ coefficients on the molality of functional groups in the ion exchanger.

The molality of functional groups in Cl^- form cannot be used as Equation (1) has been derived, and so coefficients $\overline{K}_D$ were calculated neglecting swelling changes during sorption of acid. The acid invasion increases with decreasing molality of functional groups. The diffusion coefficient increases with decreasing weight capacity (at the same water content), decreasing electrostatic retardation and at the same weight capacity with increasing water content, consequently with decreasing molality of functional groups.

Millar (1968) found the acid invasion for isoporous resins independent of the molality of functional groups, whereas for gel type of anion exchangers the invasion increases greatly with decreasing molality of functional groups. It would mean that for different isoporous resins the sorption rate changes with the value of diffusion coefficient with about the same acid invasion.

We have been able to find only one value of Glueckauf parameter z_G given for isoporous weak base resin z_G = 0.77 (Adams et al., 1969). At the acid concentration 0.02 N and considering $k = 1.0$, the ratio c_H^0/c_H calculated from Glueckauf expression will be 0.406. From the value of total water content and from true density of the resin (1.1 g/cm³), we calculate the fractional pore volume of Zerolit HIP 0.572. From Wheeler's relation $\overline{D}_H = D_H \cdot \epsilon/2$, we can calculate the value of diffusion coefficient $0.858 \cdot 10^{-5}$ cm²/s (for $D_H = 3 \cdot 10^{-5}$ cm²/s). Multiplying both values we obtain the value of $\overline{K}_D$ equal to $34.8 \cdot 10^{-7}$ cm²/s, which is in accordance with the value obtained from the curve given in Figure 2 ($36.8 \cdot 10^{-7}$ cm²/s).

In the case of resins, it is not possible to decrease the molality of functional groups markedly, because the total volume capacity would be simultaneously reduced. At the same weight capacity, the increasing water content leads to the decrease of molality of functional groups, but also to the decrease of total volume capacity. Thus, it is necessary to choose a compromise between the total capacity and the kinetic properties; it means to influence the water content and the weight capacity so that the molality of functional groups will be as low as possible at the same time with sufficient total volume capacity. The example is Lewatit MP 64, which has high water content and high total volume capacity. Total volume capacity is higher than that of Amberlite IRA 93 with about the same kinetic properties. In the case of Lewatit MP 62 and Wofatit AD 41, the water content is substantially lower and consequently the molality of functional groups higher.

In the case of gel type of styrene —DVB resins, we can obtain a higher water content and thus a lower molality of functional groups by decreasing DVB content only. These resins would have a low mechanical osmotic stability and a low volume capacity. For this reason, isoporous resins are more suitable as the cross-linking is formed by $-CH_2$ bridges, and the spaced skeleton is formed with a good mechanical osmotic stability.

With acrylic weak base resins, higher water content can be obtained even with gel type of resins. The more hydrophilic acrylic skeleton swells to a higher extent, due to the use of a more polar compound instead of styrene. Moreover, the fractional void volume between resin beads for gel type of resin Amberlite IRA 68 is only 0.3, whereas for styrene-DVB resins it is usually 0.4. This enables the increased water content with simultaneously high volume capacity. Unfortunately, weak base resins of acrylic type have a higher consumption of rinse water after regeneration except for Lewatit CA 9249, probably due to a low amount of weakly acidic groups. This is confirmed by the fact that after regeneration with Na_2CO_3, the required rinse water is decreased approximately to the level of styrene-DVB weak base anion exchangers.

When SO_4^{2-} ions are in the influent, some of the strong base groups in the resin may remain in HSO_4^- form, and the equilibrium capacity will be slightly higher. In case the acid invasion is complete, the sorption rates for sulfuric acid will be the same as for hydrochloric acid. Adams et al (1969) confirmed about the same sorption rates for Amberlite IRA 93. For this resin we also found about the same coefficients $\overline{K}_D$ in the presence of sulfuric acid. In case that acid invasion is not complete, the

presence of SO_4^{2-} ions leads to the increasing acid concentration within the resin at the particle surface. The sorption rate will thus be higher and the decrease of breakthrough capacity with increasing specific loading less sharp.

In case of converting some of the functional groups into HCO_3^- form, the sorption rate substantially increases (Richter, 1962). This may be realized either by the presence of carbon dioxide in the influent or by the regeneration with Na_2CO_3 or $NaHCO_3$ (Figure 2). The resin is now more ionized and may now behave partly like strong base anion exchanger. The sorption rate will thus be higher. The effect of variable solution composition has not been sufficiently tested, so it is difficult to prove the validity of the presented theory for other acid composition than hydrochloric acid.

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NOTATION

c_H = concentration of acid in the solution, meq/cm³ solution
$\bar{c}_{H^o}$ = concentration of acid within the resin at the resin particle surface, meq/cm³ solution
C_o = total volume capacity of the bed, meq/cm³ resin bed
C_s = breakthrough capacity of the bed at specific loading s, meq/cm³ resin bed
C_W = weight capacity of ion exchanger, meq/g dry ion exchanger
D_i = diffusion coefficient of species i in the solution, cm²/s
$\bar{D}_i$ = diffusion coefficient of species i in the resin, cm²/s
F = degree of exhaustion
k = constant of proportionality
m = molality of functional groups in the ion exchanger, meq/g solution
q = internal cross section of column, cm²
r_o = particle radius of resin beads, cm
s = specific loading of the bed, bed volumes/hr
t = time, s
v = linear flow rate, cm/s
v_B = rate of motion of the boundary in the column, cm/s
W = total water content of the ion exchanger, wt %
$\bar{X}$ = concentration of functional groups in the ion exchanger, meq/cm³ resin bead
$\bar{X}_s$ = concentration of exhausted functional groups in the ion exchanger at specific loading s, meq/cm³ resin bead
z = axial space coordinate in columns
z_G = Glueckauf's parameter
Z = bed height in column operations, cm
β = fractional void volume
ϵ = fractional pore volume in the ion exchanger

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Characterizing the Effect of Feed Rate in Continuous Flow Vessels Having Turbine Agitators

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Continuous flow agitated vessels are often used for processing non-Newtonian fluids, particularly for fermentation and polymerization processes. For process control applications and for scale-up purposes, the effect of feed rate on the operation of the system should be understood. The mixing in such systems is one aspect of the operation which can be studied in the laboratory by measuring residence time distributions. The effect of

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