

CHAPTER 6

ADSORPTION

THE THEORY OF ADSORPTION

Adsorption involves accumulation of substances at an interface, which can either be liquid-liquid, gas-liquid, gas-solid or liquid-solid. The material being adsorbed is termed the adsorbate and the adsorbing phase the adsorbent.

The expression sorption including both adsorption and absorption is generally used for a process where the components move from one phase to another, but particularly it is used when the second phase is solid.

A solid surface in contact with a solution has the tendency to accumulate a surface layer of solute molecules, because of the imbalance of surface forces - an adsorption takes place. The adsorption results in the formation of a molecular layer of the adsorbate on the surface. Often an equilibrium concentration is rapidly formed at the surface and is generally followed by a slow diffusion onto the particles of the adsorbent.

The rate of adsorption is generally controlled by the rate of diffusion of the solute molecules. The rate varies reciprocally with the square of the diameter of the particles and increases with increasing temperature (Weber et al., 1963). For practical application either Freundlich's isotherm or Langmuir's isotherm provide a satisfactory relationship between the concentration of the solute and the amount of adsorbed material. The Freundlich isotherm is expressed in the following equation:

$$a = k \cdot C^n \quad (6.1)$$

where

k and n = constants

a = the amount of solute adsorbed per unit weight

C = the equilibrium concentration of the solute in the liquid phase

The values of k and n are given for several organic compounds in Table 6.1 (Rizzo et al., 1977).

TABLE 6.1

Freundlich's constant for adsorption of some organic compounds on activated carbon

Compound	k(mg/g)	n
Aniline	25	0.322
Benzene sulphonic acid	7	0.169
Benzoic acid	7	0.237
Butanol	4.4	0.445
Butyraldehyde	3.3	0.570
Butyric acid	3.1	0.533
Chlorobenzene	40	0.406
Ethylacetate	0.6	0.833
Methyl ethyl ketone	24	0.183
Nitrobenzene	82	0.237
Phenol	24	0.271
TNT	270	0.111
Toluene	30	0.729
Vinyl chloride	0.37	1.088

Langmuir's adsorption isotherm is based on the following expression:

$$a = \frac{A_o \cdot C}{1 + b \cdot C} \quad (6.2)$$

where

a and C = as defined above

b and A_o = constants.

As can be seen, $a = A_o/b$ when $C \rightarrow \infty$

TABLE 6.2

Langmuir's constant for adsorption of some organic compounds on activated carbon

Compound	A_o (g/g)	b (l/mg)
Phenol	0.118	1.15
p-Nitrochloro-benzene	0.286	0.714
Dodecylbenzene sulphonate	1.83	13.2

The Langmuir constant for several organic compounds which can be adsorbed on activated carbon has been found by Weber et al., (1964). Most types of waste water contain several substances which will be adsorbed and in this case a direct application of Langmuir's adsorption isotherm is not possible.

Weber et al. (1965) have developed an equation, (6.3) and (6.4), for competitive adsorption of two substances (A and B). In other words competitive adsorption can be described in the same way as a competitive enzymatic reaction:

$$a_A = \frac{A_{Ao} \cdot C_A}{1 + b_A \cdot C_A + b_B \cdot C_B} \quad (6.3)$$

$$a_B = \frac{A_{Bo} \cdot C_B}{1 + b_A \cdot C_A + b_B \cdot C_B} \quad (6.4)$$

Langmuir's constants for some organic compounds generally found in waste water are shown in Table 6.2.

When it is necessary to find whether Freundlich's adsorption isotherm or Langmuir's adsorption isotherm gives the best fit to a set of data, the two plots shown in Figs. 6.1 and 6.2 should be used. When a linear relation between $\log a$ and $\log C$ is obtained, Freundlich's adsorption isotherm is a good description of a set of data as:

$$\log a = \log k + n \log C \quad (6.5)$$

On the other hand, when the reciprocal values of a and C give a linear equation (see Fig. 6.2) Langmuir's adsorption isotherm gives a good description of the set of data, since

$$\frac{1}{a} = \frac{1 + b \cdot C}{A_o \cdot C}$$

or

$$\frac{1}{a} = \frac{1}{A_o} \cdot \frac{1}{C} + \frac{b}{A_o} \quad (6.6)$$

It is possible to a certain extent, to predict the adsorption ability of a considered component. The solubility of the dissolved substance is by far the most significant factor in determining the intensity of the driving forces. The greater the affinity of a substance for the solvent, the less likely it is to move towards an interphase to be adsorbed. For an aqueous solution this means that the more hydrophylic the substance is the less likely it is to be adsorbed. Conversely hydrophobic substances will be easily adsorbed from aqueous solutions.

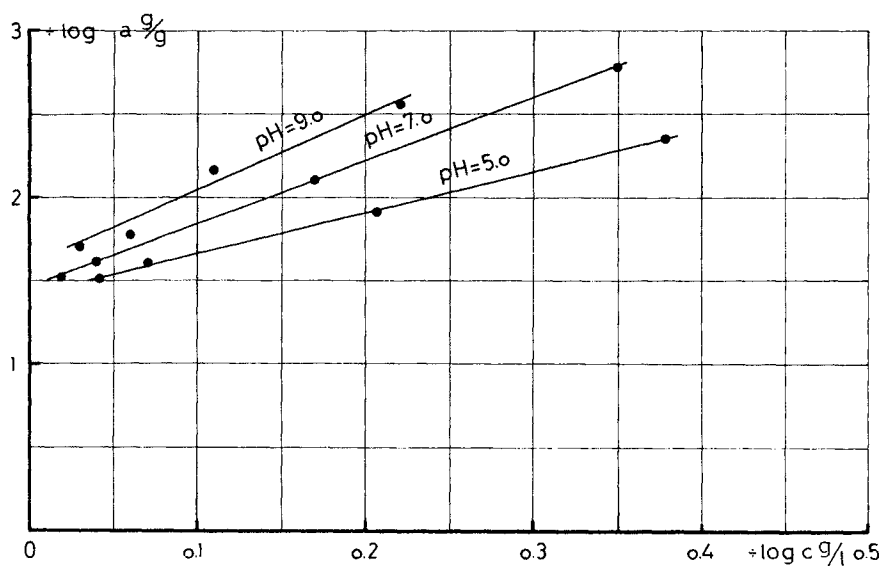


Fig. 6.1. Adsorption of lysine on a cellulose cation exchanger at three different pH-values. (Jørgensen, 1976a)

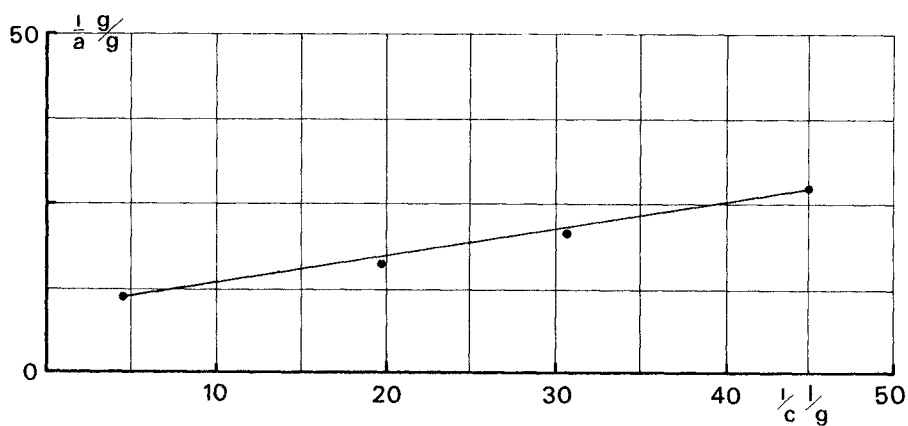


Fig. 6.2. Adsorption of uric acid on activated carbon (pH = 9.0) (Jørgensen, 1976b)

Many organic components, e.g. sulphonated alicyclic benzenes, have a molecular structure consisting of both hydrophylic and hydrophobic groups. The hydrophobic parts will be adsorbed at the surface and the hydrophylic parts will tend to stay in the water phase.

The sequential adsorption operation

The sequential operation is frequently called contact filtration, as the typical application includes treatment in a mixing tank followed by filtration, but more frequently settling is used for removal of the used adsorbents in industrial waste water engineering. The sequential adsorption operation is limited to treatment of solutions where the solute to be removed is adsorbed relatively strongly when compared with the remainder of the solution. This is often the case, when colloidal substances are removed from aqueous solutions using carbon as in the production of process water.

The method for dealing with the spent adsorbent depends upon the system under consideration. If the adsorbate is valuable material, it might be desorbed by contact with a solvent other than water. If the adsorbate is volatile, it may be desorbed by reduction of the partial pressure of the adsorbate over the solid by passing steam or air over the solid. In the case of most sequential adsorption operations in the context of waste water treatment, the adsorbate is of no value and it is not easily desorbed. The adsorbent may then be regenerated by burning off the adsorbate, followed by reactivation.

A mathematical treatment of the sequential operation distinguishes between single-stage operations, multi-stage concurrent operations and multi-stage countercurrent operations.

A schematic flowchart for a single-stage operation is shown in Fig. 6.3.

As the amount of adsorbent is usually very small compared with the amount of solution treated and since the solute to be removed is adsorbed much more strongly than the other components present, the adsorption of the latter may be ignored. Furthermore, the adsorbent is generally insoluble in the solution. If the water (see Fig. 6.3) to be treated contains S kgs of unadsorbed substance (water) then the adsorbable solute concentration is reduced from Y_0 to Y_1 kgs of solute per kg of solvent.

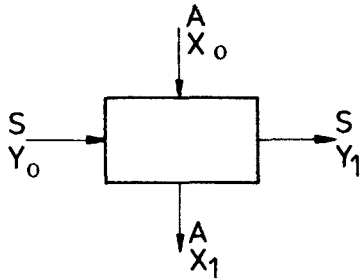


Fig. 6.3. Flowsheet for the single-stage operation.

If the adsorbent added is A kgs, then the solid adsorbate content increases from Y_0 to X_1 kgs of solid per kg of adsorbent. In most cases fresh adsorbent is used so that $X_0 = 0$. The mass balance of the solid removed is given by the following equation:

$$S(Y_0 - Y_1) = A(X_1 - X_0) \quad (6.7)$$

This equation is called the operating line and is shown in Fig. 6.4 together with the equilibrium curve. This could be either Freundlich's or Langmuir's isotherm.

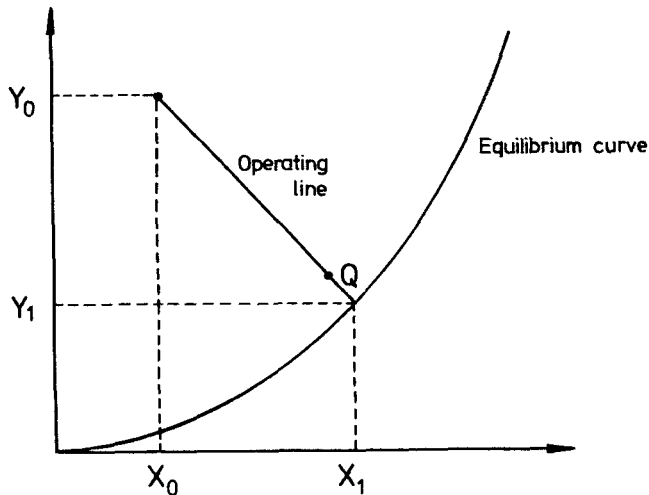


Fig. 6.4. Operating line and equilibrium curve for a single-stage operation.

If sufficient time of contact is allowed, so that equilibrium is almost reached, the final liquid and solid concentration will correspond to a point (see Q, Fig. 6.4), which is quite close to the equilibrium curve.

The mass balance assumes that the amount of liquid mechanically retained with the solid after filtration or settling is negligible. This is usually the case.

If Freundlich's adsorption isotherm can be used, we can at the final equilibrium condition set up the following equation:

$$Y_1 = k \cdot X_1^n \quad (6.8)$$

Since the adsorbent used ordinarily contains no initial adsorbate, that is $X_0 = 0$, then the two equations yield:

$$\frac{A}{S} = \frac{Y_0 - Y_1}{(Y_1/k)^{1/n}} \quad (6.9)$$

As can be seen, this permits analytical calculation of the adsorbent solution ratio for a given change in solution concentration, provided that the constants in the equation system are known.

However, removal of a given amount of solute may be accomplished by the use of less adsorbent, if the solution is treated with separate small batches of adsorbent rather than a single large batch. This method is the multi-stage concurrent operation. The savings are greater the larger the number of batches, but the expense of equipment and even handling costs will increase with the number of stages. It is therefore rarely economical to use more than two or three stages. A schematic flowchart and operating diagram for two ideal stages of concurrent adsorption are shown in Fig. 6.5. As seen, the same quantity is treated in each stage, but by two different amounts of adsorbent A_1 and A_2 . The material balances are given by the following equations:

$$S(Y_0 - Y_1) = A_1(X_1 - X_0) \quad (6.10)$$

$$S(Y_1 - Y_2) = A_2(X_2 - X_0) \quad (6.11)$$

These two equations provide the operation lines as shown in Fig. 6.6.

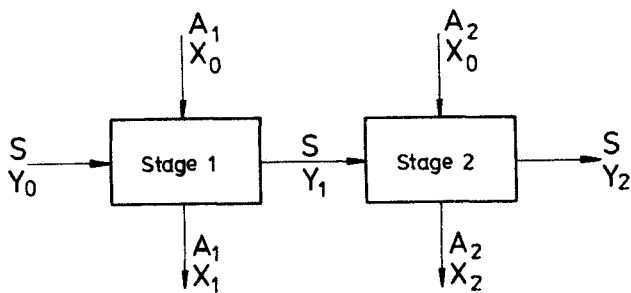


Fig. 6.5. Flowchart for a two stages concurrent operation.

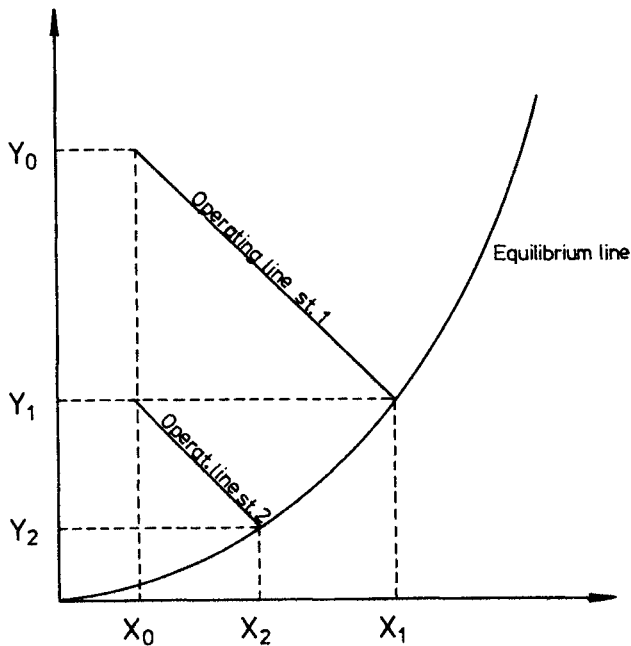


Fig. 6.6. Operating diagram for two stages concurrent adsorption.

When Freundlich's expression is used as a description of the adsorption isotherm and fresh adsorbent is used in each stage, $X_0 = 0$, the two-stage system can be computed directly:

$$\frac{A_1}{S} = \frac{Y_0 - Y_1}{(Y_1/k)^{1/n}} \quad (6.12)$$

$$\frac{A_2}{S} = \frac{Y_1 - Y_2}{(Y_2/k)^{1/n}} \quad (6.13)$$

or

$$\frac{A_1 + A_2}{S} = k^{1/n} \left(\frac{Y_0 - Y_1}{Y_1^{1/n}} + \frac{Y_1 - Y_2}{Y_2^{1/n}} \right) \quad (6.14)$$

The minimum total adsorbent is found by setting $\frac{d(A_1 + A_2)}{dY_1} = 0$. This reduces to:

$$\left(\frac{Y_1}{Y_2} \right)^{1/n} - \frac{1}{n} \cdot \left(\frac{Y_0}{Y_1} \right) = 1 - \frac{1}{n} \quad (6.15)$$

Equation (6.15) can be solved for Y_1 , and the adsorbed quantity can be found by equations (6.12) and (6.13).

Even greater economy in the use of adsorbent can be achieved by a countercurrent operation. Fig. 6.7 shows a diagram of this operation and Fig. 6.8 shows the operation line and equilibrium curve for this case. The operating line can be set up as follows:

$$S(Y_0 - Y_2) = A(X_0 - X_1) \quad (6.16)$$

and if Freundlich's adsorption isotherm can be used and $X_0 = 0$, then a combination of this equation and (6.16), provides the following expression:

$$\frac{S}{A} = \frac{Y_0 - Y_2}{(Y_1/k)^{1/n}} \quad (6.17)$$

An equation for calculating Y_1 can be found eliminating $\frac{S}{A}$:

$$\frac{Y_0}{Y_2} - 1 = \left(\frac{Y_1}{Y_2} \right)^{1/n} \cdot \left(\frac{Y_1}{Y_2} - 1 \right) \quad (6.18)$$

It is then possible to calculate $\frac{S}{A}$ directly from (6.17).

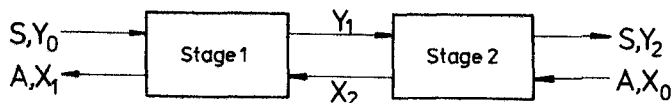


Fig. 6.7. Flowsheet for a two stages countercurrent adsorption.

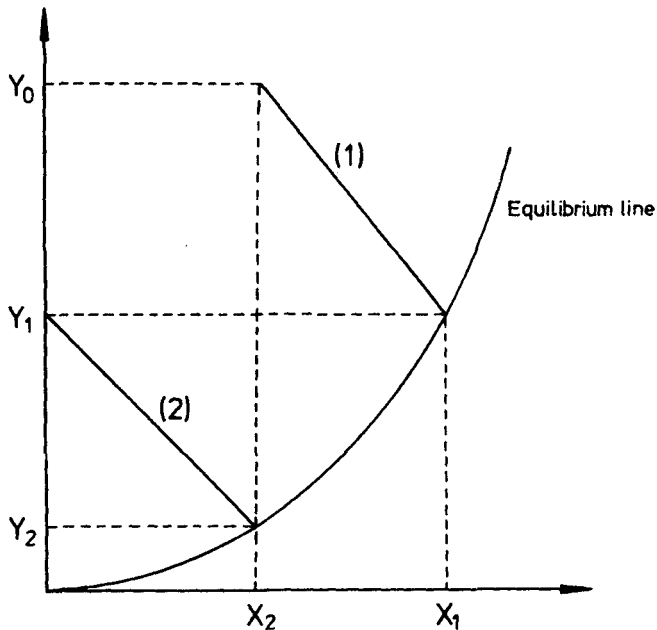


Fig. 6.8. Operating diagram for two stages countercurrent adsorption.

If Freundlich's adsorption isotherm cannot be used, it is of course possible to use the diagram for the necessary calculation as shown in Fig. 6.8.

The continuous operation

In this operation the water and the adsorbent are in contact throughout the entire process without a periodic separation of the two phases. The operation can either be carried out in strictly continuous steady-state fashion by movement of the solid as well as the fluid or in a semicontinuous fashion characterized by moving fluid but stationary solid, the so-called fixed bed adsorption.

Due to the inconvenience and relatively high cost of continuously transporting solid particles, it is generally found more economical to use a stationary bed of adsorbent for waste water treatments. Only this case will therefore be treated mathematically.

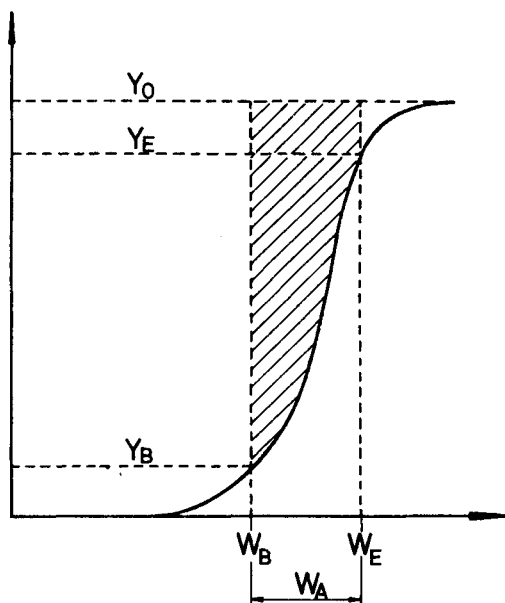


Fig. 6.9. Idealized break-through curve.

The design of a fixed bed adsorber and the prediction of the length of the adsorption cycle requires knowledge of the percentage approach to saturation at the break point. Fig. 6.9 shows an idealized break-through curve.

Let us consider a case where the flow of water through an adsorbent bed is $S \text{ kg/h m}^2$ - entering with an initial solute concentration of $Y_0 \text{ kg solute/kg solvent}$. The total, solute free, effluent after a given time is called $W \text{ kg/m}^2$ (see Fig. 6.9). The break-through curve should be steep and the solute concentration in the effluent rises rapidly from close to zero to that of the incoming water. Some low value Y_B is arbitrarily chosen as the break-point concentration and the column is considered exhausted when the effluent concentration has risen to some other arbitrarily chosen concentration of value Y_E , close to Y_0 . The critical values are the quantity of effluent W_B at the break-point and the shape of the curve between W_B and W_E (see Fig. 6.9).

The effluent accumulated during the occurrence of the break-through curve is:

$$W_A = W_E - W_B \quad (6.19)$$

The adsorption zone, that part of the bed in which the concentration changes from Y_B to Y_E , is considered to have a constant height of Z_A m. If we call the time required for the adsorption zone to move its own height down the column after the zone has been established for T_A , then:

$$T_A = \frac{W_A}{S} \quad (6.20)$$

Correspondingly we call the time required for the adsorption zone to establish itself and move out the bed, T_E , which will be possible to calculate from:

$$T_E = \frac{W_E}{S} \quad (6.21)$$

If we call the height of the entire adsorbent bed for, Z (m), and, T_F , the time required for formation of the adsorption zone, we get:

$$Z_A = Z \frac{T_A}{T_E - T_F} \quad (6.22)$$

The quantity of solid removed from the water in the adsorption zone from the break-point to exhaustion is called U kg solid/m².

This area is shown in Fig. 6.9.

If all the adsorbent in the zone was saturated with solute, it would contain $Y_O \cdot W_A$ kg solute/m².

Consequently at the break-point, the zone is still within the column. The fractional ability, f , of the adsorbent in the zone still to adsorb is:

$$f = \frac{U}{Y_O \cdot W_A} = \frac{\int_{W_B}^{W_E} (Y_O - Y) dW}{Y_O \cdot W_A} \quad (6.23)$$

If $f = 0$ it means that the adsorbent in the zone is saturated, and the time of formation of the zone at the top of the bed, T_F , should be the same as the time required for the zone to travel a distance equal to its zone height, T_A . On the other hand, if $f = 1.0$ so that the solid in the zone contains essentially no adsorbate, the zone formation should be very short.

These limiting conditions are described by:

$$T_F = (1 - f)T_a \quad (6.24)$$

Equations (6.22) and (6.24) provide:

$$Z_a = Z \frac{T_A}{T_E - (1 - f)T_A} = Z \frac{W_A}{W_E - (1 - f)W_A} \quad (6.25)$$

The adsorption column is Z m tall of unit cross sectional area and contains $Z \cdot Q$ kg adsorbent, where Q is the apparent packed density of the solid in the bed. If the column was in complete equilibrium and saturated at an adsorbate concentration of X_T kg adsorbate/kg solid, the adsorbate weight would be $Z \cdot Q \cdot X_T$ kg. At the break-point the adsorption zone of height, Z_a , is at the bottom of the column, but the rest of the column, $Z - Z_a$ m, is substantially saturated. At the break-point therefore, the adsorbed solid is:

$$(Z - Z_a) Q \cdot X_T + Z_a \cdot Q \cdot f \cdot X_T \quad (6.26)$$

The fractional saturation of the column at the break-point is:

$$\frac{(Z - Z_a) \cdot Q \cdot X_T + Z_a \cdot Q \cdot f \cdot X_T}{Z \cdot Q \cdot X_T} = \frac{Z - (1 - f) \cdot Z_a}{Z} \quad (6.27)$$

In the fixed bed of adsorbent, the adsorption zone moves downwards through the solid as we have seen.

The operating line of the entire tower is:

$$S (Y_0 - 0) = A (X_T - 0) \quad (6.28)$$

or

$$\frac{S}{A} = \frac{Y_0}{X_T} \quad (6.29)$$

Since the operating line passes through (0.0) of Fig. 6.10, at any level in the column, the concentration of solute in the water, Y , and adsorbate upon the solid, X , are then related by the equation:

$$S \cdot Y = A \cdot X \quad (6.30)$$

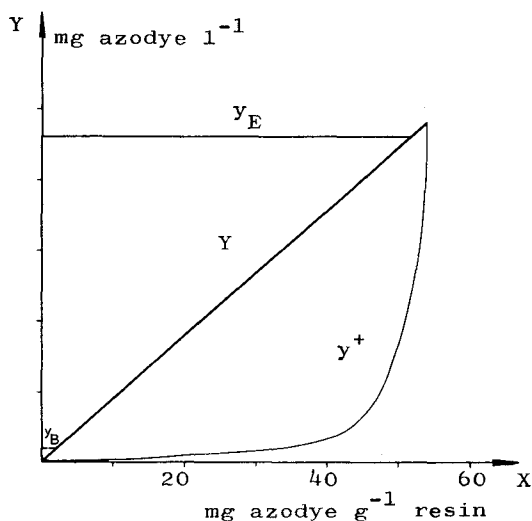


Fig. 6.10. Y is the operating line, y^+ the equilibrium curve, y_B is considered as break-point and at y_E the bed is saturated.

Over the differential height, dZ , the rate of adsorption is:

$$SdY = K_t \cdot a (Y - Y^+)dZ \quad (6.31)$$

where K_t = the overall transfer coefficient, a = the outside surface area of the solid particles and Y^+ = the equilibrium concentration.

For the entire adsorption zone:

$$N_t = \int_{Y_B}^{Y_E} \frac{dY}{Y - Y^+} = \frac{Z_a}{H_t} = \frac{Z_a}{S/K_t \cdot a} \quad (6.32)$$

where N_t = the overall number of transfer units in the adsorption zone.

The success of this analysis hinges upon the constancy of K_t or H_t for the concentration within the adsorption zone. This will of course depend upon the relative constancy of the resistance to mass transfer in the fluid and within the pores of the solid. In chapter 7 a parallel problem based on ion exchange will demonstrate the method of using this equation in a typical case (see example 7.1).

Rate of adsorption

The adsorption rate can be limited by external diffusion, internal diffusion or by a combination of external and internal diffusion. The external diffusion controls the transfer of solute from the water to the boundary layer of fluid immediately adjacent to the external surface of the adsorbent. The external diffusion is governed by molecular diffusion and in a turbulent flow by eddy diffusion.

The process can be described by the following equation:

$$V_a = k_e \cdot a (Y - Y^+) \quad (6.33)$$

where V_a = the rate of adsorption; Y = the concentration of the adsorbate in the fluid and Y^+ = the concentration of the adsorbate in the fluid in equilibrium with the existing concentration in the adsorbent. k_e = the external mass transfer coefficient.

Internal diffusion processes control the transfer of solid from the exterior of the adsorbent to the internal surface (Weber and Rumer, 1965). This condition is represented by the following equation:

$$V_a = k_i \cdot a \cdot \Sigma \cdot (X^x - X) \quad (6.34)$$

where Σ = the interparticle void ratio; X^x = the concentration of adsorbate in the solid phase that is assumed to be in equilibrium with the coexisting liquid phase at concentration, Y ; X = the actual concentration of adsorbate in the solid phase.

If the internal and the external diffusion occur in comparable rates the respective mass transfer coefficient, measured individually, may be added (King, 1965):

$$\frac{1}{K} = \frac{1}{k_e} + \frac{1}{k_i} \quad (6.35)$$

The diffusion coefficient as used in the design of a practical column must either be found in the literature or found by experimentation. The internal diffusion can be found by equilibrium experiments by use of equation (6.33).

Example 7.1 shows the application of the diffusion coefficient or rather H_t in column design.

Factors influencing adsorption

The extent of adsorption is proportional to the surface area. To be able to compare different adsorbents a specific surface area, defined as that portion of the total surface area, that is available for adsorption per unit of adsorbent, is used. This means that the adsorption capacity of a nonporous adsorbent should vary inversely with the particle diameter, while in the case of highly porous adsorbents, the capacity should be almost independent of the particle diameter. However, for some porous material such as activated carbon, the breaking up of large particles to form smaller ones opens some tiny sealed channels in the column, which might then become available for adsorption (Weber and Morris, 1964).

The nature of the adsorbate also influences the adsorption. In general an inverse relationship can be anticipated between the extent of adsorption of a solute and its solubility in the solvent (water) from which adsorption occurs. This is the so-called Lundilius' rule, which may be used for semi-quantitative prediction of the effect of the chemical character of the solute on its uptake from solution (water) (Lundilius, 1920).

Ordinarily the solubility of any organic compound in water decreases with increasing chain length because the compound becomes more hydrophobic as the number of carbon atoms becomes greater. This is Traube's rule.

Adsorption from aqueous solution increases as homologous series are ascended, largely because the expulsion of increasingly large hydrophobic molecules from water permits an increasing number of water-water bonds to form. Fig. 6.11 shows the effect of molecular weight on the capacity for adsorption for several sulphonated alkylbenzenes. As seen, the figure illustrates very well the above mentioned Traube's rule. The molecular weight is also related to the rate of uptake of solutes by activated carbon, if the rate is controlled by intraparticle transport. Data are plotted in Fig. 6.12 for the rates of adsorption of a series of sulphonated alkylbenzenes of different molecular size. It can be seen that the molar rate of uptake decreases with increasing molecular weight.

It is also in accordance with Lundilius' rule that increasing ionization means decreasing adsorption (when water is the solvent). For instance, the adsorption of several substituted benzoic acids varies inversely with the dissociation constant (Langlois, 1955).

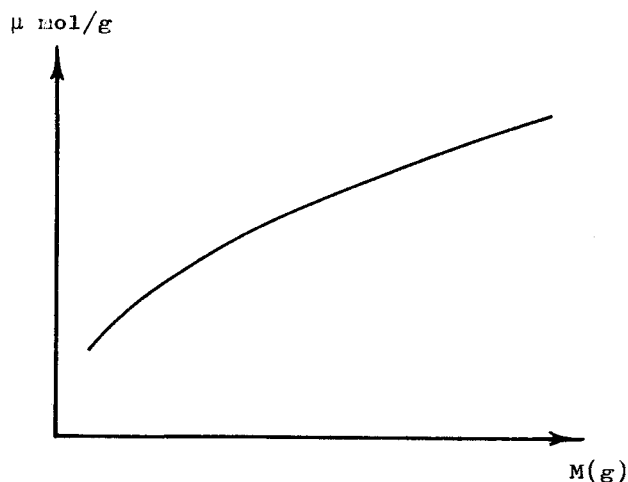


Fig. 6.11. Effect of molecular weight on capacity for adsorption for several sulphonated alkylbenzenes.

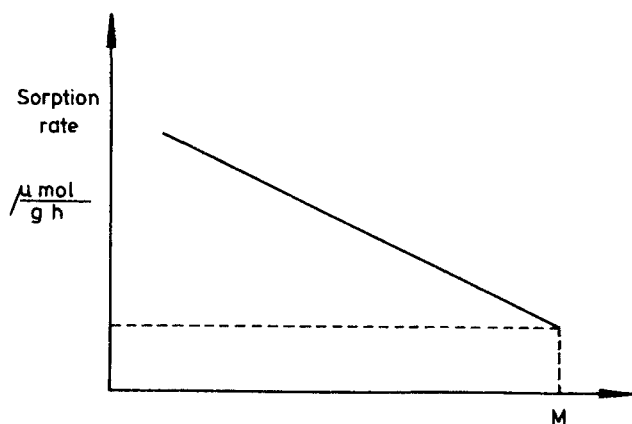


Fig. 6.12. Sorption rate plotted to the molecular weight.

Also compounds with an amphoteric nature have maximum adsorption at the isoelectric point.

pH strongly influences the adsorption as hydrogen and hydroxide ions are adsorbed, and the charge of the other ions are influenced by the pH of the water. For typical organic pollutants from industrial waste water, the adsorption increases with increasing pH.

Normally the adsorption reactions are exothermic, which means that the adsorption will increase with decreasing temperature, although small variations in temperature do not tend to alter the adsorption process to a significant extent.

The application of adsorption

Adsorption can be used to remove several organic compounds, such as phenol, alkylbenzene-sulphonic-acid, dyestuffs and aromatic compounds from waste water by the use of activated carbon. Logsdon et al. (1973) suggest application of activated carbon for removal of mercury. 1 mg/l of powdered carbon is needed for each 0.1 µg/l mercury to be removed. Furthermore, activated carbon can remove chloramines, which means that a combination of chlorination and treatment with activated carbon can be used for removal of ammonium. However, this is a rather expensive process as more than an equivalent amount of chlorine must be used. In practice, it is recommended to apply 10 mg chlorine per mg of ammonium nitrogen. This indicates that this method of removing ammonium can only be recommended when it is a question of removal of a few mg of ammonium per litre of waste water.

Scaramelli et al. (1973) have examined the effect (on effluent quality) of adding powdered activated carbon to an activated sludge system. They found that 100 to 200 mg/l was able to reduce TOC from about 20 mg/l to 7 mg/l.

Activated carbon has also been suggested as an adsorbent for the removal of refractory dyestuffs (Eberle et al., 1976).

Adsorption is also used for the removal of phosphate from waste water. In this case the adsorbent is activated aluminium oxide. Investigations have, however, shown that this process is a surface exchange of ions rather than a pure adsorption. It is more economical to use activated aluminium oxide for phosphate removal, especially when small concentrations of phosphate are present, than chemical precipitation.

Regeneration of granular carbon

In very small plants it may be feasible to use granular carbon on a use throw away basis, although economics probably would favour the use of powdered carbon in a sequential operation. The use of granular activated carbon involves the regeneration and reuse of carbon, with some exceptions. This regeneration can be carried out with sodium hydroxide provided that high molecular weight colloids are removed before the treatment of the activated carbon. It is possible with this chemical regeneration to recover for example phenols (Jørgensen, 1976b) and to remove colour bodies (Mulligan et al., 1976); (Chamberlin et al., 1975).

Also solvent can be used for regeneration of activated carbon, as indicated by Rovel (1972).

When sodium hydroxide or solvent is used, the adsorbate is passed through the carbon bed in the opposite direction to that of the service cycle until all is removed. The bed is then drained and the regenerated carbon is ready to go back into the stream.

Juhola and Tupper (1969) have studied the thermal regeneration of granular activated carbon, which consists of three basic steps, (1) drying, (2) baking of adsorbate and (3) activation by oxidation of the carbon residues from decomposed adsorbates. Drying requires between 100 and 700°C and activation a temperature above 750°C. All of these three steps can be carried out in a direct fired, multiple hearth furnace. This is the best commercial equipment available for regeneration of carbon for use in combination with waste water treatment. The capacity of the activated carbon will generally decrease by approximately 10% during the first thermal regeneration and another 10% during the next 5-10 regenerations.

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