

Large-Scale Adsorption and Chromatography

Volume I

Author

Phillip C. Wankat, Ph.D

471

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PREFACE

My major goal in writing this book has been to present a unified, up-to-date development of operating methods used for large-scale adsorption and chromatography. I have attempted to gather together the operating methods which have been used or studied for large-scale applications. These methods have been classified and compared. The main unifying principle has been to use the same theory, the solute movement or local equilibrium theory, to present all of the methods. Mass transfer and dispersion effects are included with the nonlinear mass transfer zone (MTZ) and the linear chromatographic models. More complex theories are referenced, but are not discussed in detail since they often serve to obscure the reasons for a separation instead of enlightening. Liberal use has been made of published experimental results to explain the operating methods.

Most of the theory has been placed in Chapter 2. I recommend that the reader study Sections II and IV.A and IV.B carefully since the other chapters rely very heavily on these sections. The rest of Chapter 2 can be read when you feel motivated. The remaining chapters are all essentially independent of each other, and the reader can skip to any section of interest. Considerable cross-referencing of sections is used to guide the reader to other sections of interest.

I have attempted to present a complete review of the open literature, but have not attempted a thorough review of the patent literature. Many commercial methods have been published in unconventional sources such as company brochures. Since these may be the only or at least the most thorough source, I have referenced many such reports. Company addresses are presented so that interested readers may follow up on these references. Naturally, company brochures are often not completely unbiased. The incorporation of new references ceased in mid-May 1985. I apologize for any important references which may have been inadvertently left out.

Several places throughout the text I have collected ideas and made suggestions for ways to reduce capital and/or operating expenses for different separation problems. Since each separation problem is unique, these suggestions cannot be universally valid; however, I believe they will be useful in the majority of cases. I have also looked into my cloudy crystal ball and tried to predict future trends; 5 years from now some of these predictions should be good for a laugh.

Much of this book was written while I was on sabbatical. I wish to thank Purdue University for the opportunity to take this sabbatical, and Laboratoire des Sciences du Genie Chimique, Ecole Nationale Supérieure des Industries Chimiques (LSGC-ENSIC) for their hospitality. The support of NSF and CNRS through the U.S./France Scientific Exchange Program is gratefully acknowledged. Dr. Daniel Tondeur, Dr. Georges Grevillot, and Dr. John Dodds at LSGC-ENSIC were extremely helpful in the development of this book. My graduate level class on separation processes at Purdue University served as guinea pigs and went through the first completed draft of the book. They were extremely helpful in polishing the book and in finding additional references. The members of this class were Lisa Brannon, Judy Chung, Wayne Curtis, Gene Durrence, Vance Flosenzier, Rod Geldart, Ron Harland, Wei-Yih Huang, Al Hummel, Jay Lee, Waihung Lo, Bob Neuman, Scott Rudge, Shirish Sanke, Jeff Straight, Sung-Sup Suh, Narasimhan Sundaram, Bart Waters, Hyung Suk Woo, and Qiming Yu. Many other researchers have been helpful with various aspects of this book, often in ways they are totally unaware of. A partial listing includes Dr. Philip Barker, Dr. Brian Bidlingmeyer, Dr. Donald Broughton, Dr. Armand deRosset, Dr. George Keller, Dr. C. Judson King, Dr. Douglas Levan, Dr. Buck Rogers, Dr. William Schowalter, and Dr. Norman Sweed. The typing and help with figures of Connie Marsh and Carolyn Blue were invaluable and is deeply appreciated. Finally, I would like to thank my parents and particularly my wife, Dot, for their support when my energy and enthusiasm plummeted.

THE AUTHOR

Phillip C. Wankat is a Professor of Chemical Engineering at Purdue University in West Lafayette, Ind. Dr. Wankat received his B.S.Ch.E. from Purdue University in 1966 and his Ph.D. degree in Chemical Engineering from Princeton University in 1970. He became an Assistant Professor at Purdue University in 1970, an Associate Professor in 1974, and a Professor in 1978. Prof. Wankat spent sabbatical years at the University of California-Berkeley and at LSGC, ENSIC, Nancy, France.

His research interests have been in the area of separation processes with an emphasis on operating methods for adsorption and large-scale chromatography. He has published over 70 technical articles, and has presented numerous seminars and papers at meetings. He was Chairman of the Gordon Research Conference on Separation and Purification in 1983. He is on the editorial board of Separation Science. He is active in the American Institute of Chemical Engineers, the American Chemical Society, and the American Society for Engineering Education. He has consulted with several companies on various separation problems.

Prof. Wankat is very interested in good teaching and counseling. He earned an M.S.Ed. in Counseling from Purdue University in 1982. He has won several teaching and counseling awards, including the American Society for Engineering Education George Westinghouse Award in 1984.

TABLE OF CONTENTS

Volume I

Chapter 1	
Introduction	1
Chapter 2	
Physical Picture and Simple Theories for Adsorption and Chromatography	7
Chapter 3	
Packed Bed Adsorption Operations.....	55
Chapter 4	
Cyclic Operations: Pressure Swing Adsorption, Parametric Pumping, and Cycling Zone Adsorption	91
Appendix: Nomenclature	133
References.....	139
Index	177

Volume II

Chapter 5	
Large-Scale Chromatographic Separations.....	1
Chapter 6	
Countercurrent Systems: Moving Beds and Simulated Moving Beds	41
Chapter 7	
Hybrid Chromatographic Processes: Column Switching and Moving Ports	95
Chapter 8	
Two-Dimensional and Centrifugal Operating Methods	115
Appendix: Nomenclature	131
References.....	137
Index	175

Chapter 1

INTRODUCTION

The purpose of this book is to provide a unified picture of the large number of adsorption and chromatographic operating methods used for separation. The macroscopic aspects of the processes differ, but on a microscopic scale all of these separation methods are based on different velocities of movement of solutes. The solute velocities in turn depend upon the phenomena of flow through a porous media, sorption equilibria, diffusion, mass transfer, and sorption/desorption kinetics.

Since I do not read books serially from cover to cover, but instead skip to those sections I am most interested in, this book has been written for this type of selective reading. Except for Chapter 2, the chapters are essentially independent so that the reader can start anywhere. All of the chapters do rely heavily on the local equilibrium or solute movement theory. Thus, a review of Chapter 2 (Sections III.A and B, plus possibly Section IV) would be helpful before reading other parts of the book. The remainder of Chapter 2 can be picked up as needed.

We will first look (in Chapter 2) at a physical picture of solute movement in a packed column. For most systems the separation can be predicted by combining the average rate of solute movement and zone spreading effects. The average rate of solute movement will be derived for both linear and nonlinear isotherms. This average solute wave velocity depends upon the bed porosity, solvent velocity, and equilibrium conditions, and is essentially the fraction of time the solute is in the mobile phase times the fluid velocity. The solute velocity is easily calculated and easy to use to explain the macroscopic aspects of different operating methods. Nonlinear adsorption, thermal waves, changing gas velocities, and coupled systems will all be studied. The spreading of the solute zones depends on diffusion, mass transfer rates, and sorption/desorption kinetics. The amount of zone spreading is easily determined from theories for systems with linear isotherms. From these theories one obtains the familiar rule that zone spreading is proportional to the square root of the distance traveled. For nonlinear systems which form constant patterns, the mass transfer zone (MTZ) approach will be developed.

The pictures of solute movement and of zone spreading will be combined to explain the operating methods in Chapters 3 to 8. Where necessary, the results from more detailed theories will be used to explain experimental results. Chapters 3 to 8 describe different operating methods and use the theories from Chapter 2 to explain these methods. The division of different separation methods into chapters is somewhat arbitrary. Essentially, Chapters 3 to 5 cover fixed-bed systems while Chapters 6 to 8 cover moving or simulated moving beds. These six chapters are all independent and can be read in any order, although they are cross-referenced. The development of mathematical theories is mainly restricted to Chapter 2 and, to a lesser extent, Chapter 6.

The adsorption of a single solute with simple cycles is discussed in Chapter 3. The basic type of operating cycle used is shown in Figure 1-1. The adsorption of solute occurs for some period and then the solute is desorbed either with a hot fluid or a desorbent. This is a batch process with a large number of possible variations. The method has been applied for cleaning up gas streams using a hot gas for desorption, for solvent recovery from a gas stream using activated carbon and steam desorption, for liquid cleanup using either a hot liquid or a desorbent for the desorption step, and for wastewater treatment systems. General considerations are covered in Section II of Chapter 3 and specific separations are covered in the rest of the chapter. Section II.D in Chapter 3, on the effect of particle size, will probably be of interest to all readers. Many of the common commercial adsorption processes are briefly reviewed in this chapter.

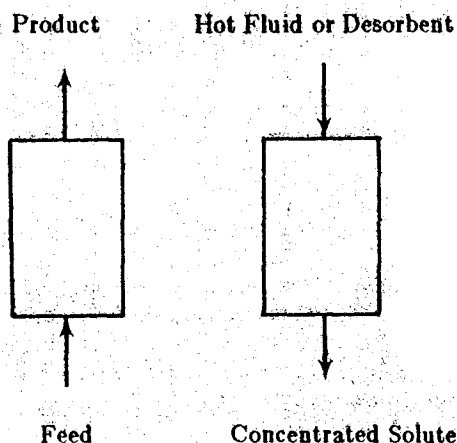


FIGURE 1-1. Basic cycle for adsorption of a single solute. (A) Adsorption step. (B) Desorption.

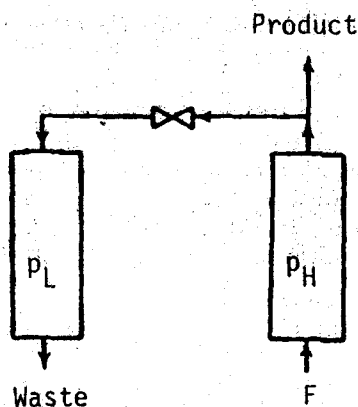


FIGURE 1-2. Basic pressure swing adsorption apparatus.

Chapter 4 covers cyclic operations which are somewhat more complex than those shown in Figure 1-1. Pressure swing adsorption (PSA) first adsorbs solute from a gas stream at elevated pressure and then desorbs the solute using a purge at much lower pressure. A very simple system is shown schematically in Figure 1-2. Since the volume of gas expands when depressurized, a larger volume but fewer moles of gas can be used for the purge step. Every few minutes the columns change functions. For liquid systems, parametric pumping and cycling zone adsorption are based on the shift in the equilibrium isotherm when a thermodynamic variable such as temperature is changed. Although this change in concentration is often small, a large separation can be built up by utilizing many shifts. A variety of cycles will be explored for both gas and liquid systems.

The separation or fractionation of more than one solute by large-scale chromatography is the subject of Chapter 5. The basic method and typical results are illustrated in Figure 1-3. Solvent or carrier gas is continuously fed into a packed column and a pulse of feed is injected intermittently. Since different solutes travel at different velocities, they exit the column at

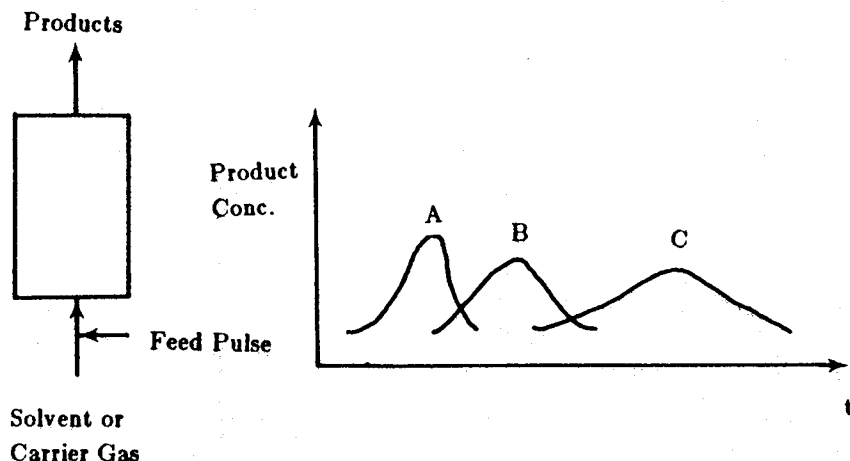


FIGURE 1-3. Apparatus and results for chromatographic separation.

different times. Large-scale liquid chromatography, size exclusion chromatography (SEC), gas-liquid chromatography (GLC), biospecific affinity chromatography, and ion-exchange chromatography will be explored.

Countercurrent moving bed systems and simulated countercurrent systems are the subject of Chapter 6. For single solute removal the basic apparatus is shown in Figure 1-4. The function of the sorption and desorption chambers is the same as in Figure 1-1, but the countercurrent apparatus operates at steady state. To fractionate two solutes the moving bed arrangement shown in Figure 1-5 could be used. This is a steady-state apparatus for binary separation. Since it is difficult to move solids in a uniform plug flow, simulated moving bed (SMB) systems have been developed commercially. In an SMB the solid does not move. Instead, the location of each product and feed port is switched in the direction of fluid flow every few minutes. When a port location moves, an observer at that port sees the solid move in the opposite direction. Thus, countercurrent motion is simulated.

Chromatographic and simulated countercurrent processes both have advantages. These two processes are combined in hybrid chromatographic processes, which are discussed in Chapter 7. With column-switching procedures the products are removed at different locations in the column. In a moving-feed chromatograph the input location of the feed pulse moves up the column to follow the movement of solute. Then chromatographic development is used to completely separate the solutes. Moving port chromatography combines these two methods.

Two-dimensional and rotating methods are discussed in Chapter 8. The prototype two-dimensional system is the rotating annulus apparatus shown in Figure 1-6. The annulus is packed with sorbent while carrier gas or solvent flows continuously upward. Feed is added continuously at one point. The result is a steady-state separation similar to the results shown in Figure 1-3, but with the angular coordinate, θ , replacing time. Many other two-dimensional arrangements have been developed. Centrifugal chromatography, the chromatofuge, is also discussed.

Most real separation problems can be solved using any of several different operating methods. Hopefully, this compendium of operating methods will provide the designer with ideas for creating new schemes. The "best" scheme will vary depending on all the facets of the problem being solved.

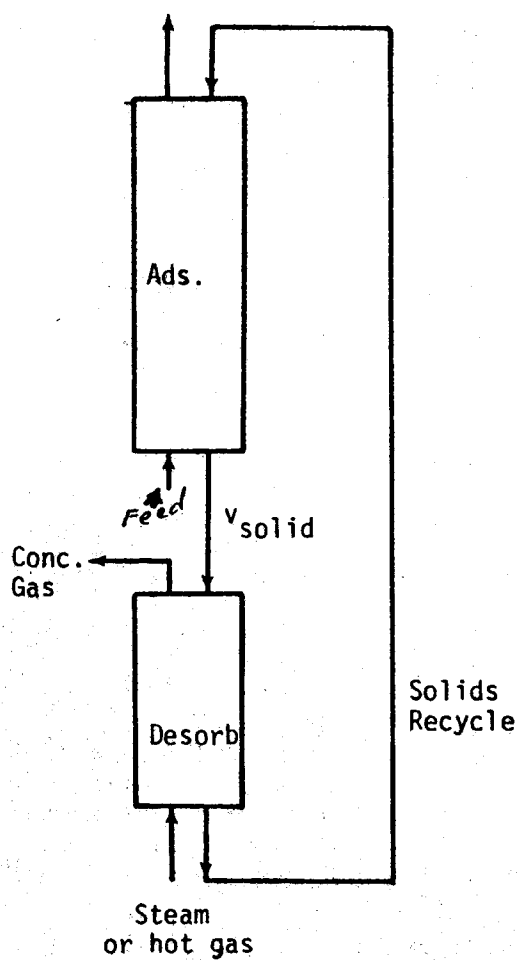


FIGURE 1-4. Countercurrent system for single solute.

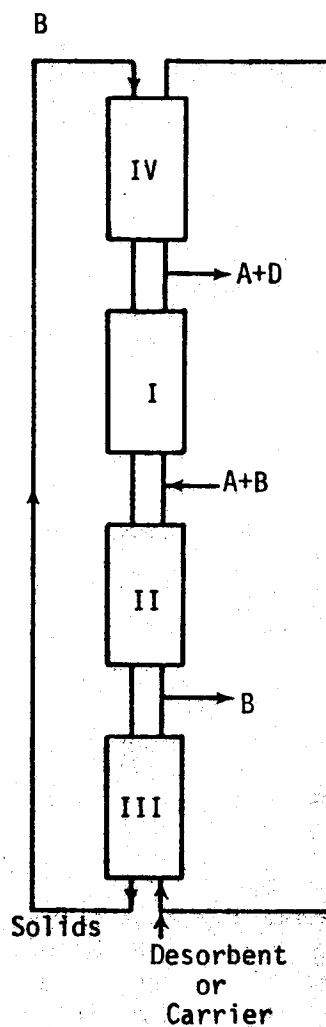


FIGURE 1-5. Countercurrent system for fractionation of two solutes.

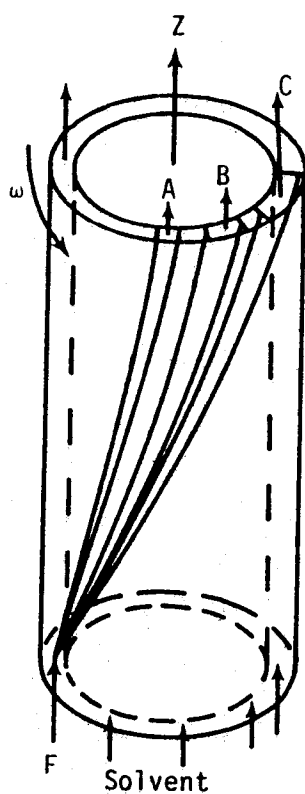


FIGURE 1-6. Apparatus and results for two-dimensional system.

Chapter 2

PHYSICAL PICTURE AND SIMPLE THEORIES FOR ADSORPTION AND CHROMATOGRAPHY

I. INTRODUCTION

What phenomena are involved in chromatographic and adsorptive separations? How do these phenomena combine to produce the desired separation? How can we simply predict the separation which will occur? In this chapter we will try to answer these and other questions. First, a physical picture of sorption will be presented. Then, equilibrium isotherms will be discussed. A physical argument will be used to explain movement of solute and energy in an adsorption column followed by a rigorous mathematical development of the equations. Zone spreading by mass transfer and diffusion will be explained mathematically for linear systems. The mass transfer zone (MTZ) or length of unused bed (LUB) approach will be introduced for nonlinear systems. The purpose of this chapter is to provide a physical understanding and relatively simple mathematical theories which will be useful in later chapters. This chapter is not a design manual; other sources should be consulted for design equations. 165,865,901,1015-1017

II. PHYSICAL PICTURE

Commercially significant sorption operations (including adsorption, chromatography, ion exchange, ion exclusion, etc.) use sorbents which are highly porous and have large surface areas per gram of sorbent. The sorbent particles are commonly packed in a column as illustrated in Figure 2-1. In general, the particles will be of different sizes and shapes. They will pack in the column and have an average interparticle (between different particles) porosity of α . In a poorly packed bed α may vary considerably in different parts of the column. This can lead to poor flow distribution or channeling and will decrease the separation. Since the particles are porous, each particle has an intraparticle (within the particle) porosity, ϵ , which is the fraction of the particle which is void space. If the packing is manufactured uniformly, ϵ will be the same for all particles. Approximately 2% of the surface area is on the outer surface of the packing; thus, most of the capacity is inside the particles. An alternate model using a single porosity is also commonly used and is discussed in Section V.B.

The pores are not of uniform size. Large molecules such as proteins or synthetic polymers may be sterically excluded from some of the pores. The fraction of volume of pores which a molecule can penetrate is called K_d . Very small molecules can penetrate all the pores and $K_d = 1.0$ while very large molecules can penetrate none of the pores and $K_d = 0$. For a nonsorbed species, K_d can be determined from a simple pulse experiment. Very small nonsorbed species will have available both the external void volume V_o and the internal void volume V_i . Thus, small molecules will exit at an elution volume, V_e , of

$$V_e = V_o + V_i = \alpha V_{col} + (1 - \alpha)\epsilon V_{col} \quad (2-1)$$

where V_{col} is the volume of the packed column. Since large molecules have available only the external void volume V_o , their elution volume is

$$V_e = V_o = \alpha V_{col} \quad (2-2)$$

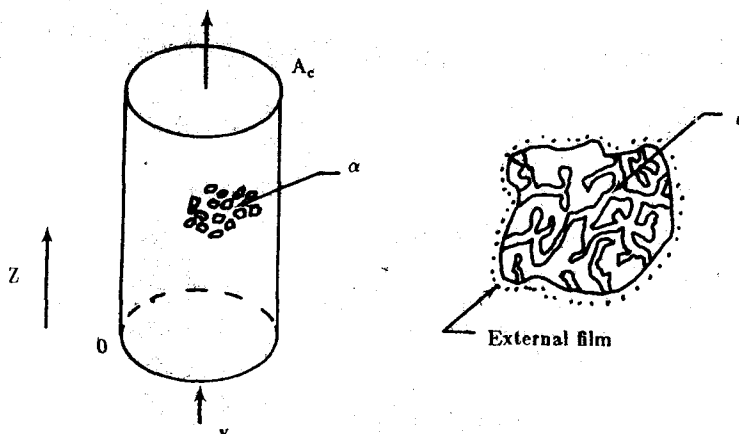


FIGURE 2-1. Particles packed in a bed.

Equations 2-1 and 2-2 allow determination of V_o , α , V_i , and ϵ from one experiment with large molecules and one experiment with small molecules. Molecules of intermediate size can penetrate some of the pores. For these nonsorbed molecules, K_d can be determined from:

$$K_d = \frac{V_o - V_i}{V_i} \quad (2-3)$$

Size exclusion (gel permeation or gel filtration) separations are based entirely on differences in K_d .

This picture is somewhat too simple for some sorbents. Some activated carbons^{79,2,107,3} and ion exchange resins¹⁸ have two types of pores: macropores and micropores. These particles have two internal porosities: one for macropores and one for micropores. The two internal and one external porosity can be measured by three experiments. Very small molecules will permeate all pores; large molecules will permeate only the macropores; very large molecules will stay in the external void volume. The solute movement theory presented later could be adjusted for these more complex systems, but K_d can be used to approximately include the two types of internal pores.

Molecular sieve zeolites differ from both these pictures.^{105,168,645,865} The zeolite crystals form a porous three-dimensional array and have a highly interconnected, regular network of channels and cavities of very specific sizes. Thus the crystal geometry is well defined. Commercial zeolite adsorbents are pelleted agglomerates of zeolite crystals and binder. The binders have large pores and relatively little sorption capacity compared to the zeolite crystals. Typical values for void fractions are⁶⁴⁵ interpellet = 32%, intercrystal = 23%, and intracrystal = 19% based on the fraction of the entire bed.

For the system shown in Figure 2-1, the processes which occur during a separation are as follows: Fluid containing solute flows in the void volume outside the particles. The solute diffuses through an external film to the particle. Here the solute may sorb on the external surface or (more likely) diffuse into the stagnant fluid in the pores. If the pores are tight for the solute this diffusion will be hindered. The solute finds a vacant site and then sorbs by physical or electrical forces or by a chemical reaction. While sorbed the solute may diffuse along the surface. The solute desorbs and diffuses through the pores, back across the external film, and into the moving fluid. A given molecule may sorb and desorb many

times during its stay inside a single particle. Once in the moving fluid the solute is carried along at the fluid velocity until the solute diffuses into another particle and the whole process is repeated. As far as migration down the column is concerned, the particle is either moving at the interstitial velocity, v , of the fluid or it has a velocity of zero when it is inside a particle.

A large number of adsorbents, ion exchange resins, partition chromatography supports, and size exclusion packings have been developed. The properties of these are available in the following sources: ion exchangers,^{18,202,549,1016,1017,1076} size exclusion media,^{549,827,1076,1109} activated carbon,^{616,792,1016,1017} silica gel,^{112,616,1016,1017} activated alumina,^{112,438,616,1016,1017} chromatographic packings,^{827,930,1076,1126} and molecular sieves.^{105,112,168,616,645,865,1017} An extremely complete annotated bibliography of adsorption up to 1953 was compiled by Dietz.^{321,322}

III. EQUILIBRIUM ISOTHERMS

A wide variety of equilibrium isotherms have been published.^{18,105,168,645,792,825,865,1016,1017} A few of these will be reviewed for gas and liquid systems.

A. Gas Systems

For gas systems the adsorbed phase has essentially the same density as a liquid. This makes pure component equilibrium data very easy to obtain. The weight of the sorbent or the pressure can easily be measured. Many different adsorption isotherms have been developed to fit the results obtained. One of the simplest is the Langmuir isotherm.^{589,629,692,809,865,1114} This isotherm is also appealing because either a simple physical picture^{629,865} or a statistical mechanical argument^{809,865} can be used to develop the isotherm. Langmuir assumed that at most a monolayer of adsorbate could cover the solid surface. If q is the adsorbent loading and \bar{p} is the partial pressure, the Langmuir isotherm is

$$q = \frac{q_{\max} K_A \bar{p}}{1 + K_A \bar{p}} \quad (2-4)$$

where K_A is the sorption equilibrium constant and q_{\max} is capacity at monolayer coverage. The shape of Equation 2-4 is shown in Figure 2-2A. This is known as a "favorable" isotherm, because this shape leads to sharp breakthrough curves. Since K_A is a reaction equilibrium constant, it should follow the Arrhenius relationship:

$$K_A = K_0 \exp [-\Delta H/RT] \quad (2-5)$$

The Langmuir isotherm is applicable for dilute, single-component adsorption with an inert gas present or for pure gases at low pressures.

At very low partial pressure the Langmuir equations takes the linear form:

$$q = K_A q_{\max} \bar{p} \quad (2-6)$$

The linear isotherm is extremely important not only because it is a limiting form of more complex isotherms when the solute is dilute, but also because the mathematical theories become much more tractable when the isotherm is linear.

If several solutes can adsorb the Langmuir isotherm can be extended to:^{589,692,809,1114}

$$\frac{q_i}{q_{i\max}} = \frac{K_i \bar{p}_i}{1 + \sum_{i=1}^n (K_i \bar{p}_i)} \quad (2-7)$$

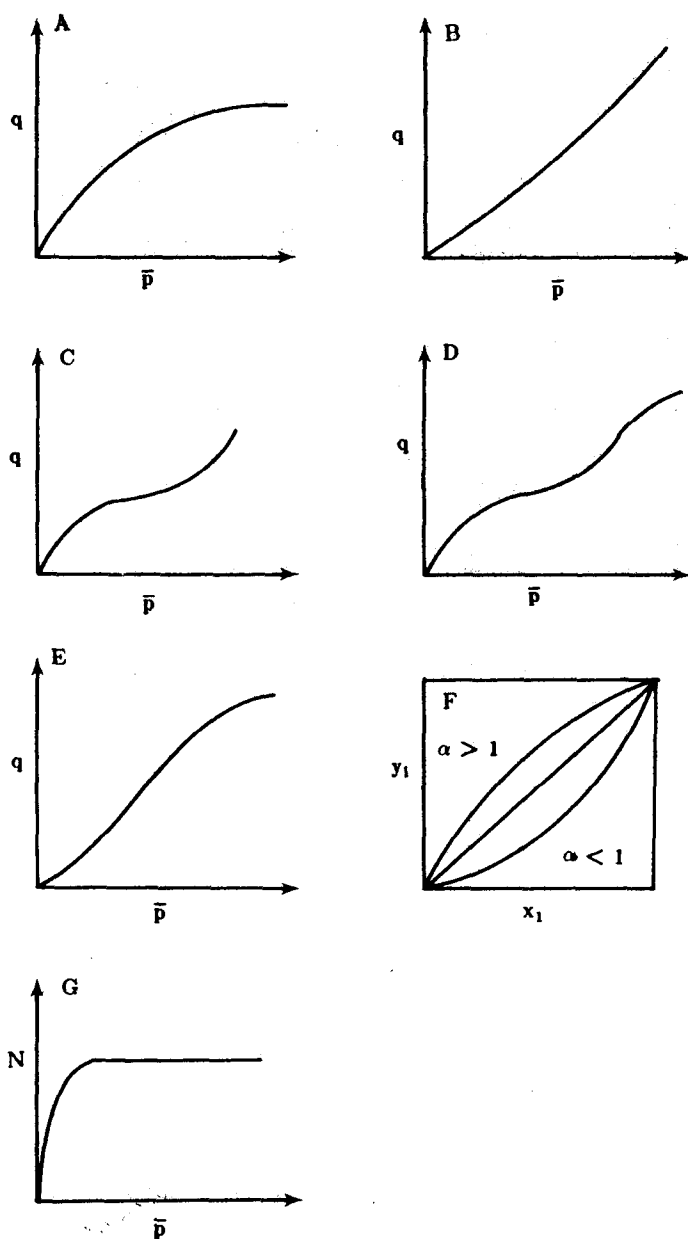


FIGURE 2-2. Equilibrium isotherms. (A) Langmuir; (B) BET type 2 behavior ($K > 1$); (C) BET type 3 behavior ($K < 1$); (D) BET type 4 behavior; (E) BET type 5 behavior; (F) Lewis correlation, constant α ; (G) molecular sieve isotherm.

In addition to the previous assumptions, it is necessary to assume that the only interaction of solutes is competition for sites on the adsorbent. In order for Equation 2-7 to be thermodynamically consistent, the monolayer coverages, q_{imax} , of all components must be equal.⁶⁵² If the monolayer coverages differ, extra terms are required for thermodynamic consistency.⁶⁵² Equation 2-7 predicts that less solute is adsorbed when other solutes are present. A few

systems will show cooperative adsorption where the presence of other solutes aids adsorption.¹¹⁰⁰ Competitive adsorption is much more common.

Langmuir's isotherm is based on a specific physical picture of adsorption. The Langmuir isotherm agrees with data for some systems but not for others. (This is generally true of all isotherms. There are none that fit all systems.) When different physical pictures are used, different isotherms result. For example, by assuming that the forces involved in adsorption and in condensation are the same, and allowing for more than one molecular layer, Brunauer et al.^{188,189} derived the BET isotherm. The simplest form of the BET equation is

$$\frac{q}{q_{\text{mon}}} = \frac{K \bar{p}}{[p^0 + (K - 1) \bar{p}][1 - \bar{p}/p^0]} \quad (2-8)$$

where q_{mon} is the adsorbate concentration for monolayer coverage and p^0 is the vapor pressure of pure solute at the adsorption temperature. The shape of Equation 2-8 depends on the values of the constants. If $K < 1$ the isotherm is unfavorable throughout and is shown in Figure 2-2B, while if $K > 1$ the isotherm is favorable at low concentrations and unfavorable at higher concentrations (Figure 2-2C). Other possible shapes which require a more complex equation^{188,809,1114} are shown in Figures 2-2D and E. If $K \gg 1$ and $\bar{p} \ll p^0$ Equation 2-8 reduces to the Langmuir form. In the limit of very dilute systems Equation 2-8 becomes linear. The adsorption isotherms for many gas systems can be fit by one of the BET forms. This is true for both adsorption of a pure gas and adsorption of a gas when an inert carrier is present. Of course, just because the isotherms fit a BET form is not proof that the mechanism postulated in the derivation is correct.

As layers build up, eventually capillary condensation will occur. This will cause an inflection point in the isotherm as in Figures 2-2C and D. In the capillary condensation regime, results for adsorption and desorption will differ. Several plausible physical pictures for this hysteresis have been suggested.⁸⁶⁵

For binary systems where both gases adsorb, Lewis et al.^{589,651} developed a correlation for adsorption from the mixture compared to pure component adsorption. This equation is

$$\frac{q_1}{q_1^0} + \frac{q_2}{q_2^0} = 1 \quad (2-9)$$

where q_i^0 is the amount adsorbed from a pure gas and q_i is the amount adsorbed from the mixture. With the additional assumption that the ratio

$$\alpha_{12} = \frac{y_1/x_1}{y_2/x_2} = \frac{y_1/q_1}{y_2/q_2} = \frac{\bar{p}_1/q_1}{\bar{p}_2/q_2} \quad (2-10)$$

is a constant, Equations 2-9 and 2-10 can be used for predictions; y_i and x_i are the mole fractions in the gas and solid phase, respectively; α_{12} is a separation factor which is essentially the same as the relative volatility of a liquid vapor mixture. The shape of these isotherms is shown in Figure 2-2F.

Isotherms can also be derived from a thermodynamic argument.⁸⁰⁹ For example, for the two-dimensional surface the pressure is replaced by the spreading pressure and the volume by the area. For an ideal adsorbed solution Myers and Prausnitz⁷⁵² showed that mixture equilibrium isotherms could be predicted from pure component isotherms. The procedure has been extended to multilayer adsorption.^{865,914} The ideal adsorbed solution theory has also been applied to heterogeneous surfaces.^{749,1125} Unfortunately, these theories do not give a single equation which can be substituted into theories of adsorption.

Molecular sieve zeolite adsorbents behave differently than other adsorbents. Instead of