Adsorption Process Development

Basics & Methodology

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Outline

- Objectives;
- Adsorbents;
- Terminology;
- Modeling;
- Equilibrium theory;
- Intraparticle kinetics – LDF model;
- Old models;
- Methodology;
- PSA and SMB techonologies.

Objectives

1) Purification

2) Recovery of solutes

3) Separations (bulk)

Examples

a. Phenolic waste waters
b. Recovery of antibiotics from fermantation broths
c. Parex/Sarex
d. Separation of enantiomeres
e. O_2 from air
f. N_2 from air
g. H_2 purification
h. Propane/propene
i. Landfill gas

Adsorbents

Types

- Activated carbons
- Carbon molecular sieves
- Zeolites
- Polymers
- Metal organic frameworks (MOFs)

Structures

- Homogeneous
- Porous
- Bidisperse

Adsorbents: properties

- Adsorption capacity;
- Adsorption kinetics;
- Selectivity;
- Mechanical stability;
- Thermal stability;
- Chemical stability.

Adsorption capacity: Amount of each species that is retained by the adsorbent

Adsorption kinetics: rate at which each species is retained by the adsorbent

Selectivity:

Equilibrium selectivity – ratio between the adsorption capacities of the more adsorbed species and the less adsorbed species;

Kinetic selectivity – ratio between the kinetic parameter of the more adsorbed species and the less adsorbed species;

Mechanical stability:

Resistance of the adsorbent – the formation of dust may damage the valves and contaminate the product.

It also limits the height of the columns.

Thermal stability:

It is important to know how the adsorbent reacts to temperature increase.

Some adsorbent can be damaged when increasing the temperature.

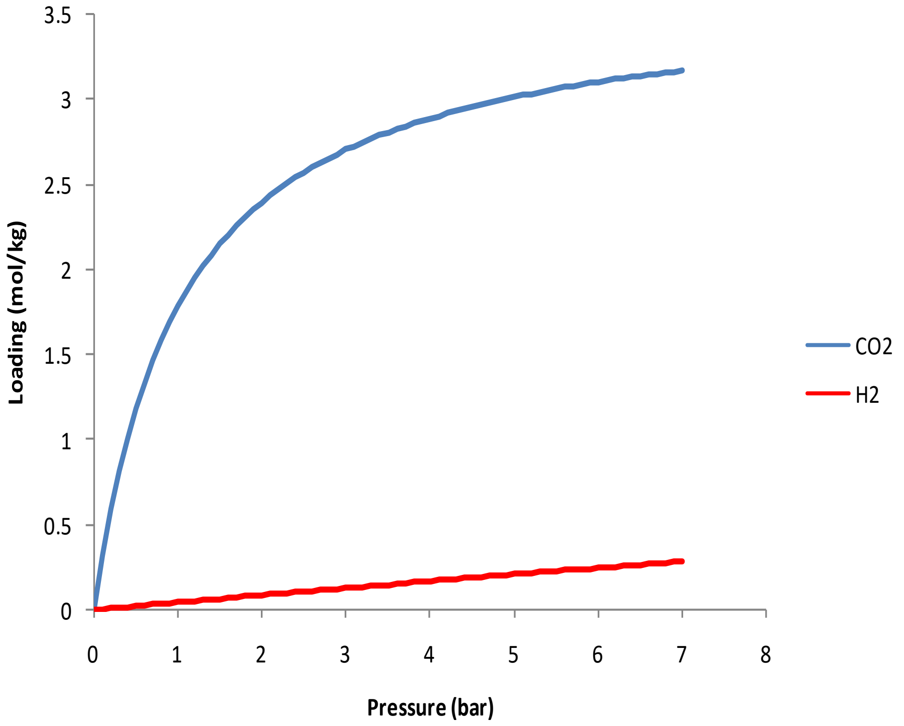
Chemical stability:

Some adsorbents can be damaged in presence of some species such as water vapour or ammonia.

It is important to know if these species are in the feed streams and if so, take measures to avoid the degradation of the adsorbent.

Equilibrium-controlled separation

Adsorption capacity of H_2 and CO_2 in activated carbon



Sircar, S.; Golden, T. C. Isothermal and Isobaric Desorption of Carbon-Dioxide by Purge. *Ind Eng Chem Res* ***1995, 34, 2881.***

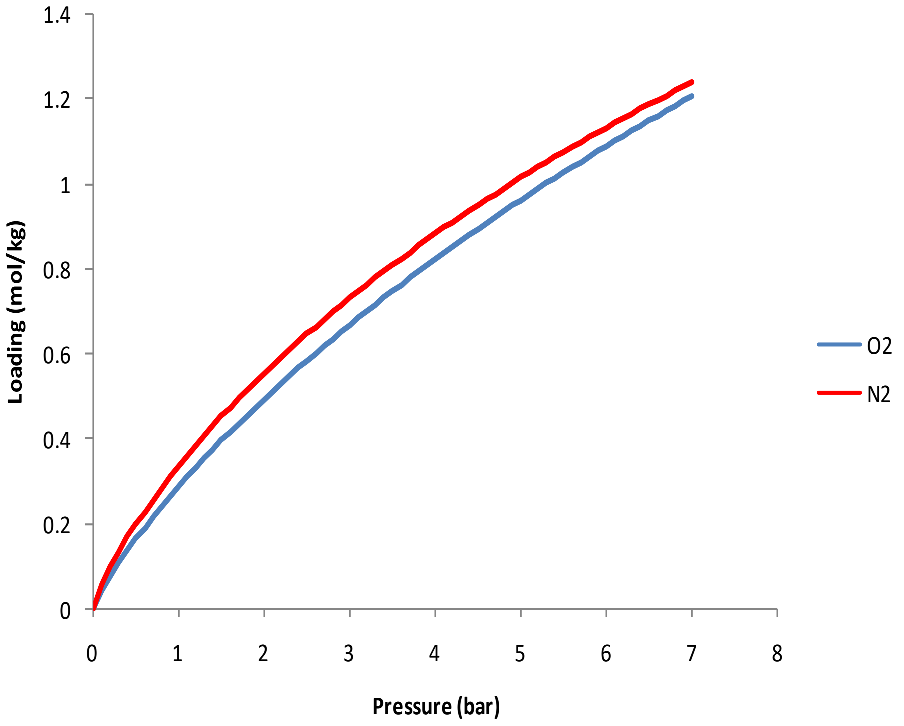
There is a significant difference between the H_2 and CO_2 adsorption capacities.

This difference in the adsorption capacity allows the separation of these two gases.

This is an equilibrium-controlled separation.

Kinetic-controlled separation

Adsorption capacity of N_2 and O_2 in a carbon molecular sieve



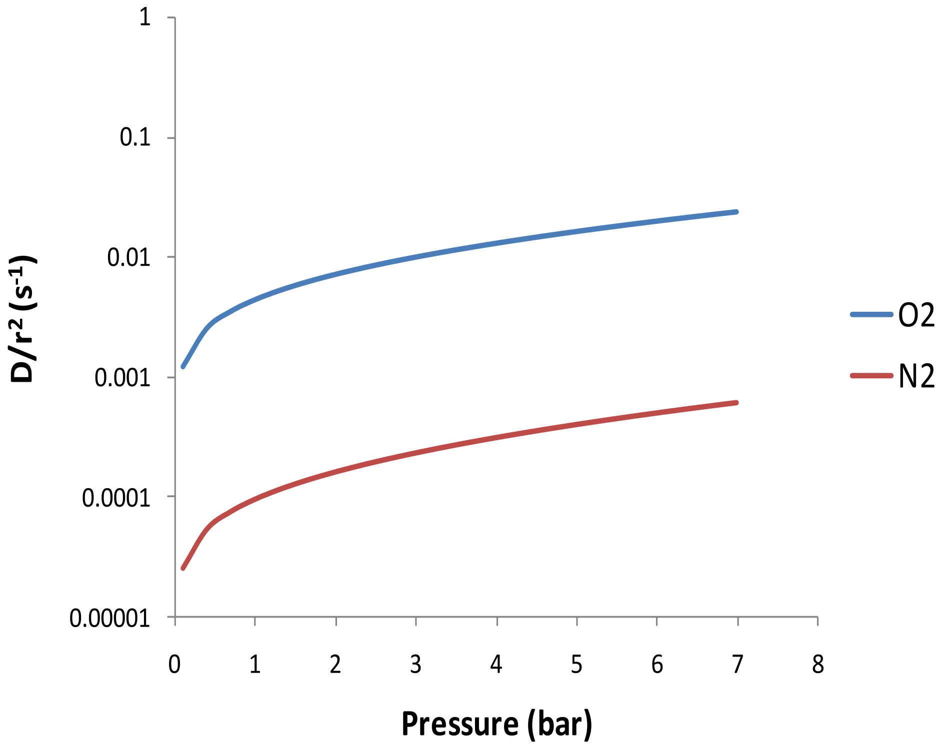
Jee, J. G.; Kim, M. B.; Lee, C. H. Pressure Swing Adsorption Processes to Purify Oxygen Using a Carbon Molecular Sieve. *Chem Eng Sci, 60, 869, 2005.*

Carbon molecular sieves are commonly used for nitrogen production from air by PSA.

How can the separation be possible if both gases adsorb almost the same?

Kinetic-controlled separation

Adsorption kinetics of N_2 and O_2 in a carbon molecular sieve



Oxygen diffuses faster than nitrogen.

This difference in the adsorption kinetics allows the separation of these two gases.

This is a kinetic-controlled separation.

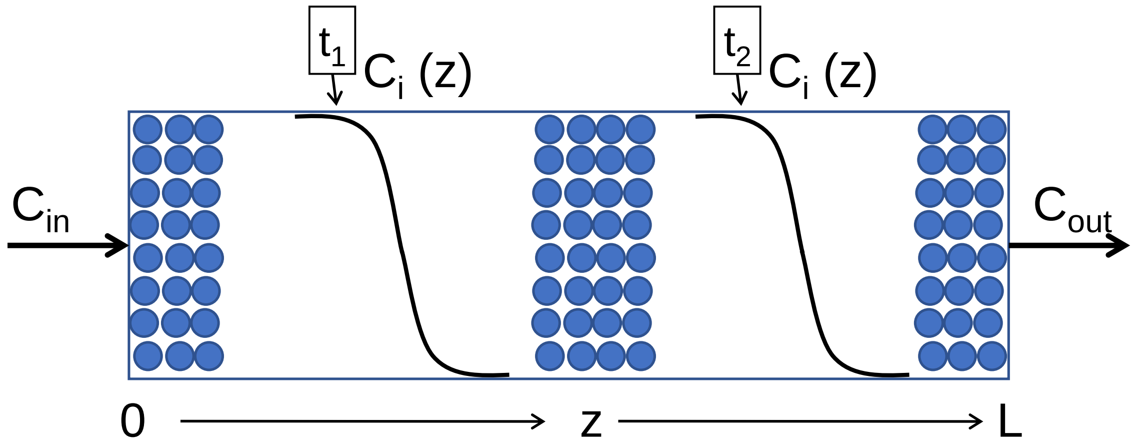
Jee, J. G.; Kim, M. B.; Lee, C. H. Pressure Swing Adsorption Processes to Purify Oxygen Using a Carbon Molecular Sieve. *Chem Eng Sci, 60, 869, 2005.*

Modelling, Simulation and Optimization

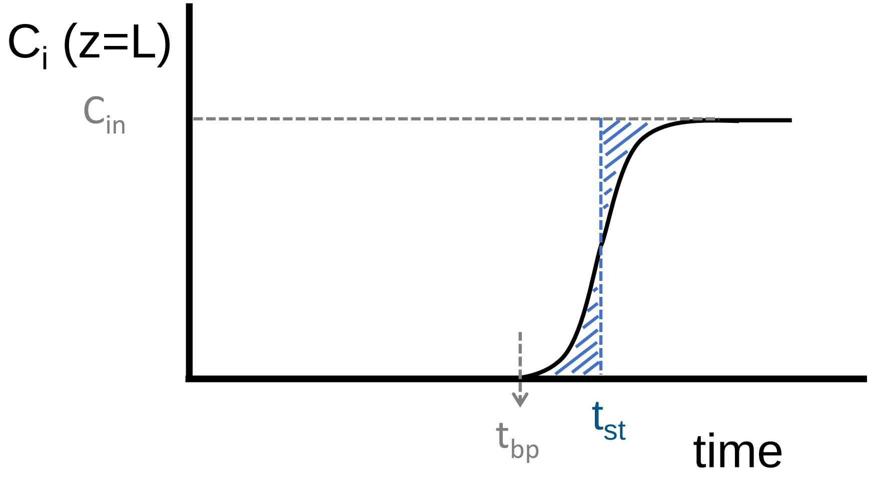
An accurate process simulator is an important tool for learning, designing and optimization purposes.

Terminology

Concentration profiles - C_i (z) at a given t



Concentration histories - C_i (t) at a given z



At z=L Breakthrough Curve

t_{bp} – breakthrough time
t_{st} – stoichiometric time

Terminology

Overall balance

$$\underbrace{Q\,C_{i0}\,t_{st}}_{\text{Moles introduced in the column}} = \varepsilon\,C_{i0}\,V + (1-\varepsilon)\,q_{i0}\,V$$

$$\boxed{t_{st} = \tau(1+\xi)}$$

$$Total\,capacity = Q\int_0^{t_f}(C_{i0}-C_i)\,dt$$

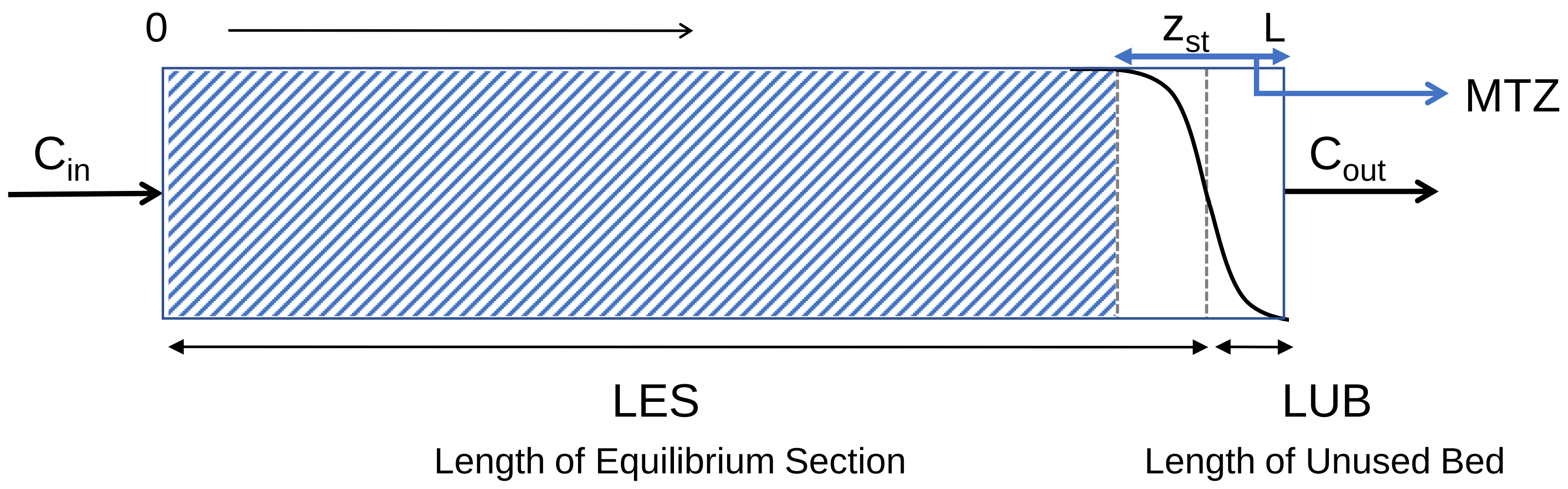
$$Useful\,capacity = Q\int_0^{t_{bp}}(C_{i0}-C_i)\,dt$$

$$\tau = \frac{\varepsilon V}{Q}$$ Space time

$$\xi = \frac{(1-\varepsilon)}{\varepsilon}\frac{q_{i0}}{C_{i0}}$$ Capacity factor

Terminology

Concentration profile at t = t_{bt}



$$L = LES + LUB$$

$$L = \frac{u_i}{1+\xi} t_{st}$$

$$z_{st} = LES = \frac{u_i}{1+\xi} t_{bp}$$

$$LUB = L - LES = L - \frac{L}{t_{st}} t_{bp}$$

$$\boxed{LUB = L\left(1 - \frac{t_{bp}}{t_{st}}\right)}$$

$$\boxed{MTZ = 2\,LUB}$$

"Le Génie Chimique c'est pas de la plomberie"

Modelling

Pierre Le Goff

a) conservation equations (mass, energy, momentum, electric charge)

b) equilibrium laws at the interface(s)

c) constitutive laws

d) kinetic laws of heat/mass transfer and reaction

e) initial and boundary conditions

f) optimization criterion

Modelling

Factors influencing the behavior of adsorptive processes

Heat transfer

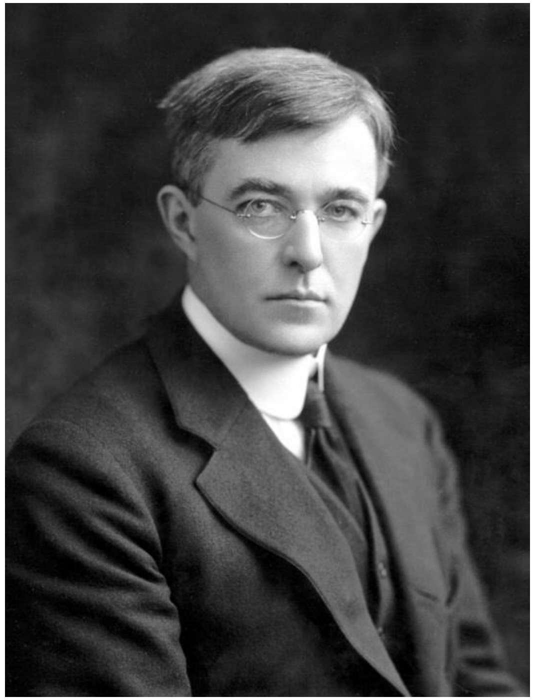
Intraparticle convection

Intraparticle diffusion

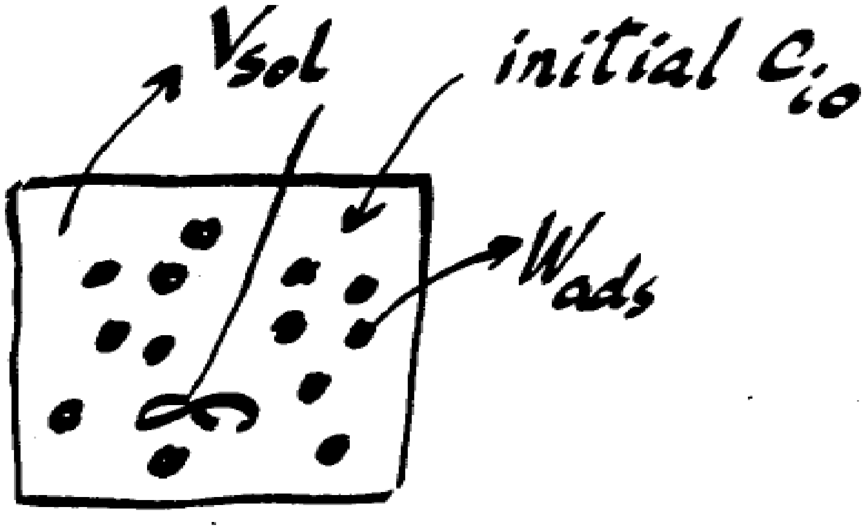
Film mass transfer

Hydrodynamics

Equilibrium



Batch adsorption (isothermal)



Mass balance in a batch adsorber

$$-V\frac{dC_i}{dt} = W\frac{dq_i}{dt}$$

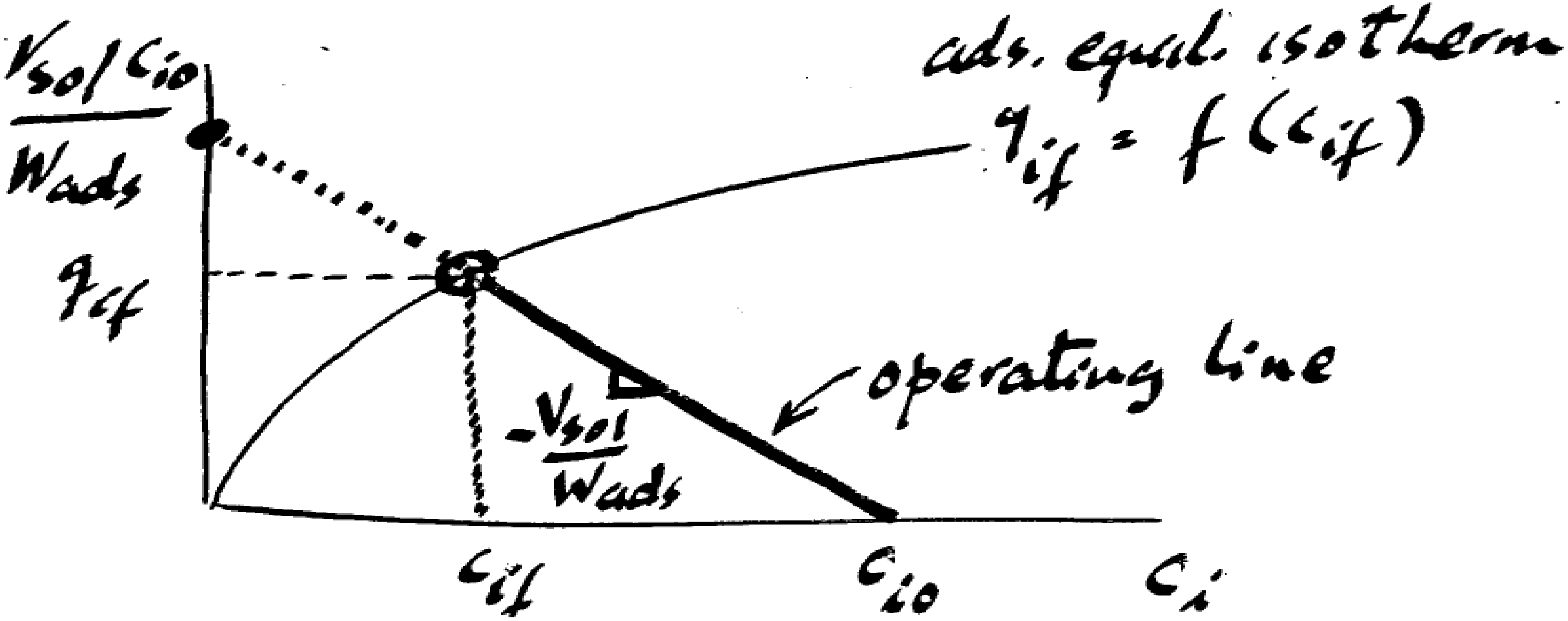
Operating Line

$$q_i(t) = \frac{V}{W}C_{i0} - \frac{V}{W}C_i$$

At equilibrium:

$$q_{ie} = \frac{V}{W}C_{i0} - \frac{V}{W}C_{ie}$$

$$q_{ie} = f(C_{ie})$$



Solve

$$\begin{cases} q_{if} = f(c_{if}) \\ q_{if} = \dfrac{V_{sol}\, c_{io}}{W_{ads}} - \dfrac{V_{sol}}{W_{ads}}\, c_{if} \end{cases} \longrightarrow c_{if}$$

Linear isotherm

$$c_{if} = \frac{c_{i0}}{1+\xi_m}$$

Factor de capacidade

$$\xi = \frac{(1-\varepsilon)}{\varepsilon}\frac{q_{i0}}{C_{i0}}$$

Rectangular

$q_{if} = Q$ for $c_{if} > 0$ $\qquad c_{if} = c_{i0}(1-\xi_m)$

Langmuir

$$q_{if} = \frac{Q K c_{if}}{1+K c_{if}} \qquad x_{if} = \frac{-[1+K'(\xi_m-1)]+\sqrt{[\cdots]^2+4K'}}{2K'}$$

$K' = K c_{i0}$

$x_{if} = c_{if}/c_{i0}$

Kinetics of batch adsorption

Mass balance

$$-V\frac{dC_i}{dt} = W\frac{dq_i}{dt} \qquad q_i = \frac{V(C_{i0} - C_{i)}}{W}$$

Kinetic law

$$\frac{dq_i}{dt} = k_h(q_i^* - q_i)$$

LDF model

Equilibrium law

$$q_i^* = KC_i$$

Linear isotherm

Initial condition

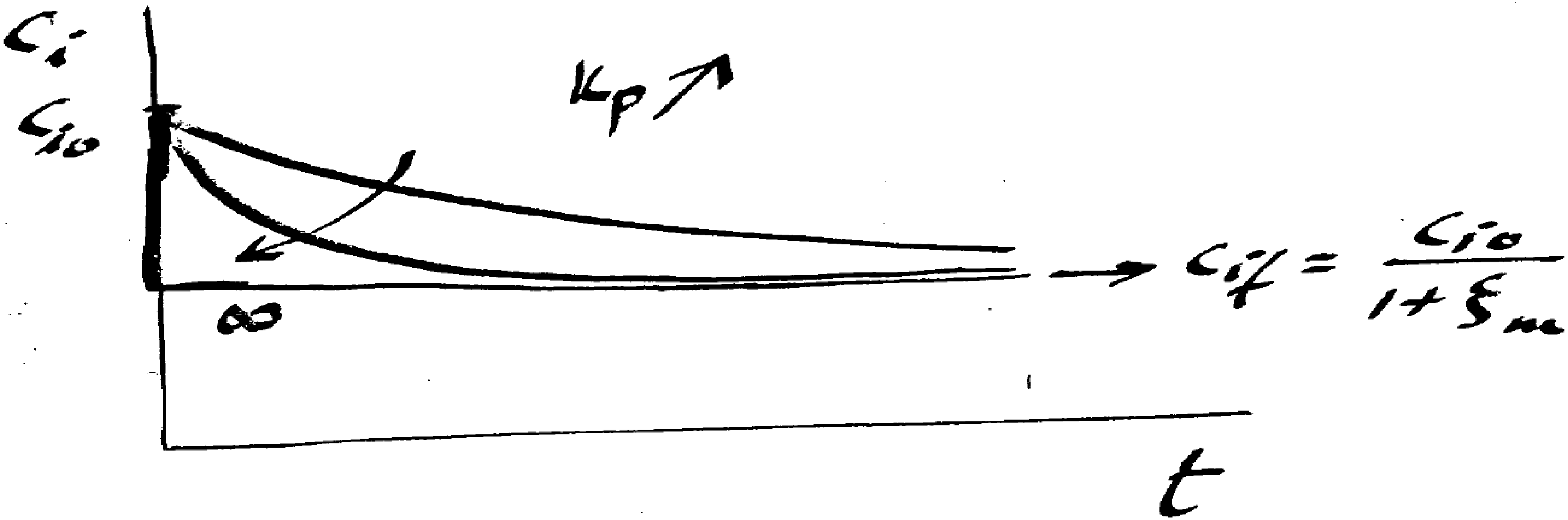
$$t = 0; C_i = C_{i0}; q_i = q_{i0}$$

Linear Driving Force model
Glueckauf

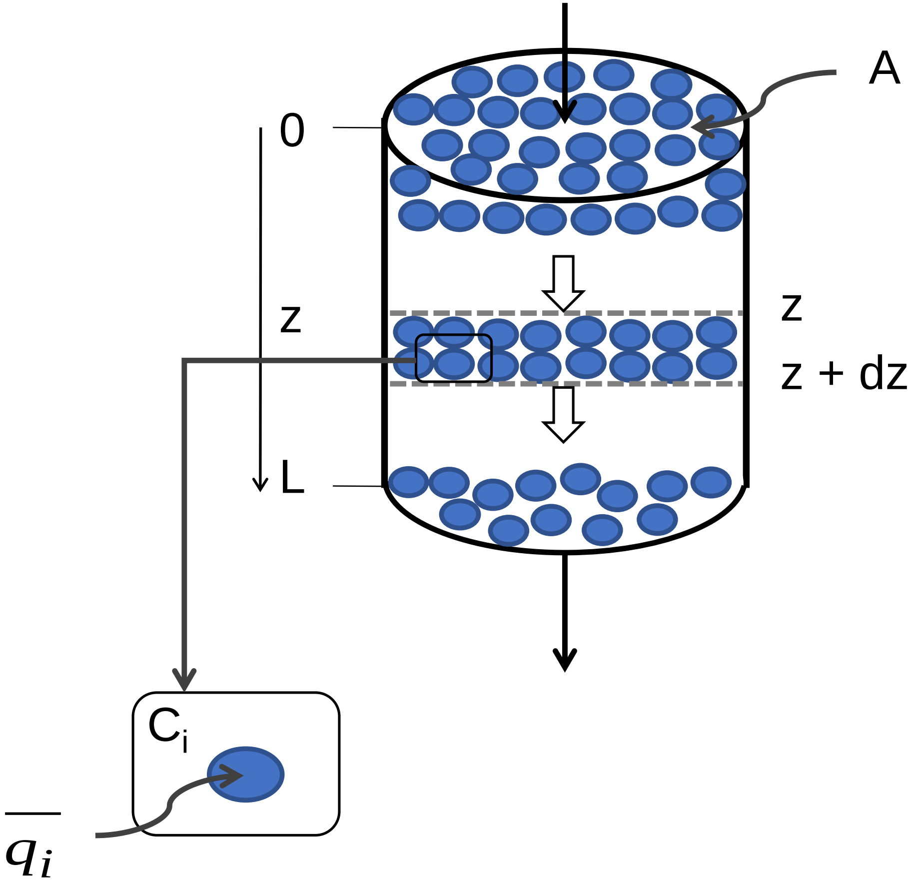
$$\frac{\partial\langle q\rangle}{\partial t} = k_h\left[q_s - \langle q\rangle\right]$$

$$k_h = \frac{15\,D_h}{{R_p}^2}$$

$$C_i(t) = \frac{C_{i0}}{1+x}\left(1 + x e^{-k_h(1+x)t}\right)$$



Mass balance for species i



Average adsorbed concentration in the particle (mol/m^3_{ads})

Isothermal operation

Axial dispersed flow

Flux in $\underbrace{}_{mol/s}$ $\qquad \underbrace{(\varepsilon A)}_{m^2} \; \underbrace{\varphi_z}_{mol/m^2s}$

Flux out $\qquad (\varepsilon A)\varphi_{z+dz}$

Acumulation

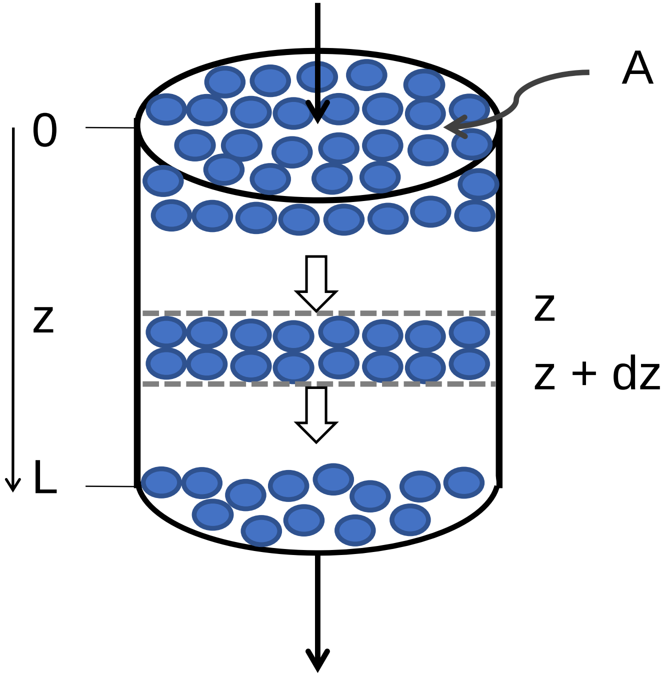
$Interparticle \qquad \varepsilon A \, dz \, \frac{\partial C_i}{\partial t}$

$Intraparticle \qquad (1-\varepsilon) A \, dz \, \frac{\partial \overline{q_i}}{\partial t}$

$$(\varepsilon A) \; \varphi_z = (\varepsilon A)\varphi_{z+dz} + \varepsilon A \, dz \, \frac{\partial C_i}{\partial t} + (1-\varepsilon) A \, dz \, \frac{\partial \overline{q_i}}{\partial t}$$

$$\boxed{0 = \frac{\partial \varphi_i}{\partial z} + \frac{\partial C_i}{\partial t} + \frac{(1-\varepsilon)}{\varepsilon} \frac{\partial \overline{q_i}}{\partial t}}$$

Mass balance for species i



Isothermal operation

Axial dispersed flow

$$0 = \frac{\partial \varphi_i}{\partial z} + \frac{\partial C_i}{\partial t} + \frac{(1-\varepsilon)}{\varepsilon}\frac{\partial \overline{q_i}}{\partial t}$$

$$\varphi = u_i\, C_i - D_{ax}\frac{\partial C_i}{\partial z}$$

$$u_i = \frac{u_0}{\varepsilon}$$

$$\boxed{\varepsilon\, D_{ax}\frac{\partial^2 C_i}{\partial z^2} = u_0\frac{\partial C_i}{\partial z} + \varepsilon\frac{\partial C_i}{\partial t} + (1-\varepsilon)\frac{\partial \overline{q_i}}{\partial t}}$$

Mass balance for species i

Dimensionless variables

$$x = \frac{z}{L} \qquad \theta = \frac{t}{\lambda} \qquad \tilde{C}_i = \frac{C_i}{C_{i0}} \qquad \tilde{q}_i = \frac{q_i}{q_{i0}}$$

$$\boxed{\frac{1}{Pe}\frac{\partial^2 \tilde{C}_i}{\partial x^2} = \frac{\partial \tilde{C}_i}{\partial x} + \frac{\partial \tilde{C}_i}{\partial \theta} + \xi \frac{\partial \overline{\tilde{q}_i}}{\partial \theta}}$$

Peclet number $\quad Pe = \dfrac{u_0 L}{\varepsilon D_{ax}} \qquad Pe \rightarrow \infty \quad$ Plug flow of fluid phase

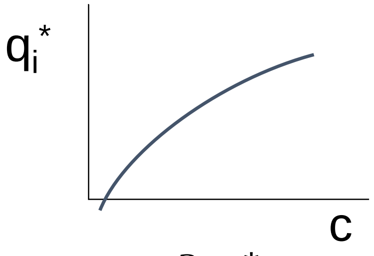
Capacity factor $\quad \xi = \dfrac{(1-\varepsilon)}{\varepsilon}\dfrac{q_{i0}}{C_{i0}} \qquad \xi \rightarrow 0 \quad$ No adsorption, inert packing

Equilibrium law at interfaces

Adsorption equilibrium isotherm

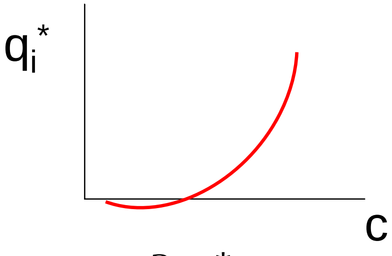
$$q_i^{\ *} = f(C_i)$$

Favorable isotherms



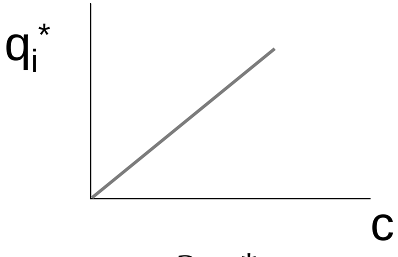
$$\frac{d^2 q_i^{\ *}}{d\,C_i^{\ 2}} < 0$$

Unfavorable isotherms



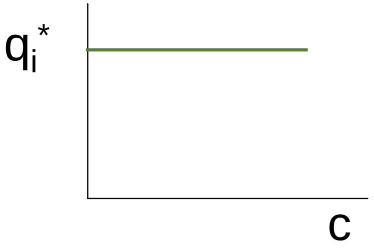
$$\frac{d^2 q_i^{\ *}}{d\,C_i^{\ 2}} > 0$$

Linear isotherms

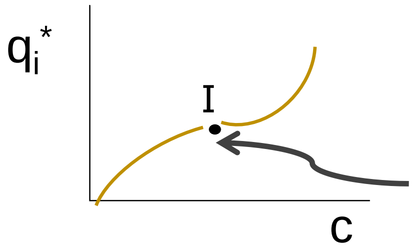


$$\frac{d^2 q_i^{\ *}}{d\,C_i^{\ 2}} = 0$$

Rectangular or irreversible



With an inflection point (BET)



$$\frac{d^2 q_i^{\ *}}{d\,C_i^{\ 2}} = 0$$

Equilibrium theory

The simplest model

- ✓ Isothermal operation
- ✓ Instantaneous equilibrium in each point of the bed - $\overline{q_i} = q_i^*$
- ✓ Plug flow
- ✓ No pressure drop (negligible)

a) Mass balance for i

$$u_0 \frac{\partial C_i}{\partial z} + \varepsilon \frac{\partial C_i}{\partial t} + (1-\varepsilon)\frac{\partial q_i^*}{\partial t} = 0$$

b) Equilibrium law

$$q_i^* = f(C_i)$$

Combining a) and b)

$$u_i \frac{\partial C_i}{\partial z} + \frac{\partial C_i}{\partial t} + \frac{(1-\varepsilon)}{\varepsilon} f'(C_i)\frac{\partial C_i}{\partial t} = 0$$

$$\boxed{u_i \frac{\partial C_i}{\partial z} + \left[1 + \frac{(1-\varepsilon)}{\varepsilon} f'(C_i)\right]\frac{\partial C_i}{\partial t} = 0}$$

Equilibrium theory

$$u_i \frac{\partial C_i}{\partial z} + \left[1 + \frac{(1-\varepsilon)}{\varepsilon} f'(C_i)\right] \frac{\partial C_i}{\partial t} = 0$$

Since $$\left(\frac{\partial z}{\partial t}\right)_C = -\frac{\left(\frac{\partial C}{\partial t}\right)_z}{\left(\frac{\partial C}{\partial z}\right)_t}$$

It results in:

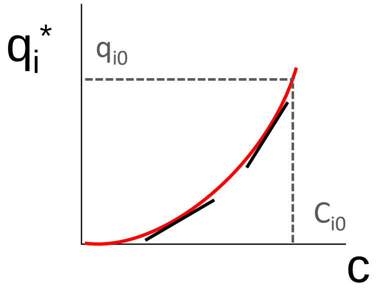
$$\boxed{u_c = \left(\frac{\partial z}{\partial t}\right)_C = \frac{u_i}{1 + \frac{1-\varepsilon}{\varepsilon} f'(C_i)}}$$

De Vault's equation (1943)

Adsorption as a wave phenomenon

The velocity of propagation of a concentration C, i.e. u_c, is inversely proportional to the local slope of the isotherm $f'(C_i)$

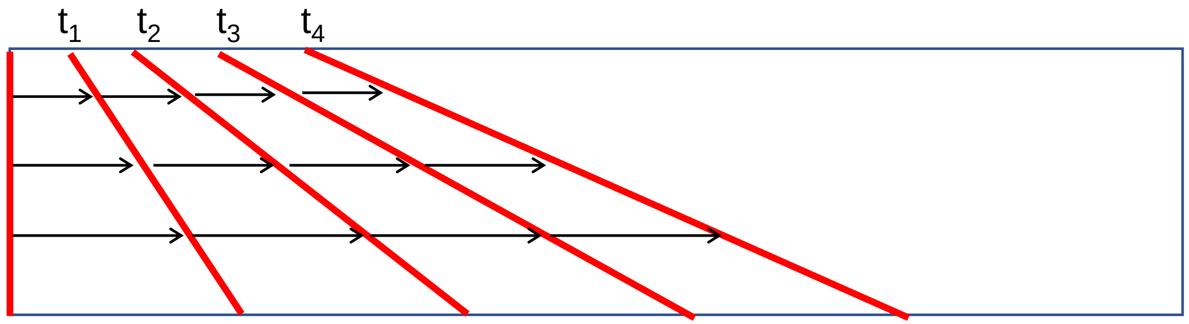
Unfavorable isotherms



As C_i $\nearrow$ the slope $f'(C_i)$ $\nearrow$ and u_c $\searrow$

Higher concentrations travel at lower velocities

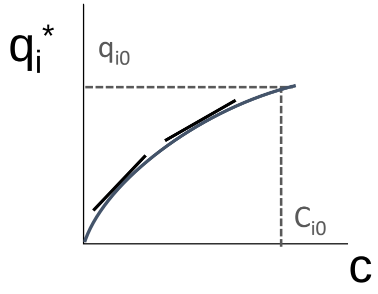
Step C_{i0}



Concentration profiles - C_i (z) at a given t

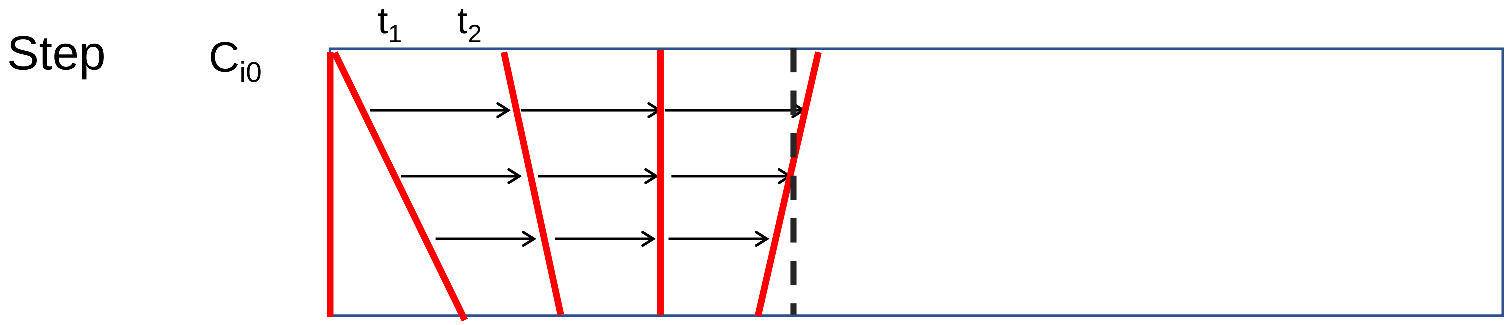
Equilibrium theory

Favorable isotherms



As $C_i \nearrow$ the slope $f'(C_i) \searrow$ and $u_c \nearrow$

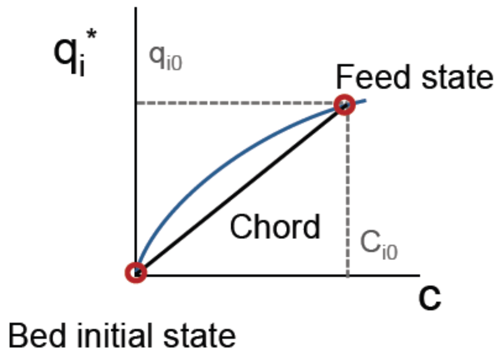
Higher concentrations travel at higher velocities



Physicaly not possible $\longrightarrow$ Compressive front $\longrightarrow$ **shock**

Equilibrium theory

Favorable isotherms



Shock velocity

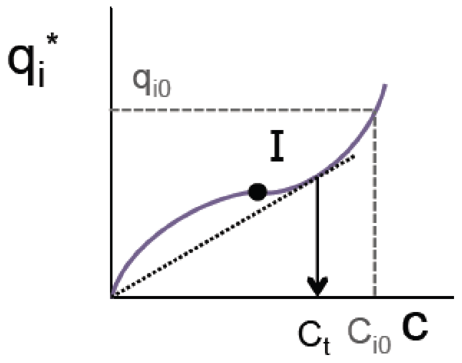
$$u_{\text{sh}} = \frac{u_i}{1 + \frac{1-\varepsilon}{\varepsilon} \underbrace{\frac{\Delta q_i}{\Delta c_i}}_{\text{Slope of the chord}}}$$

Equilibrium theory

Linear isotherms

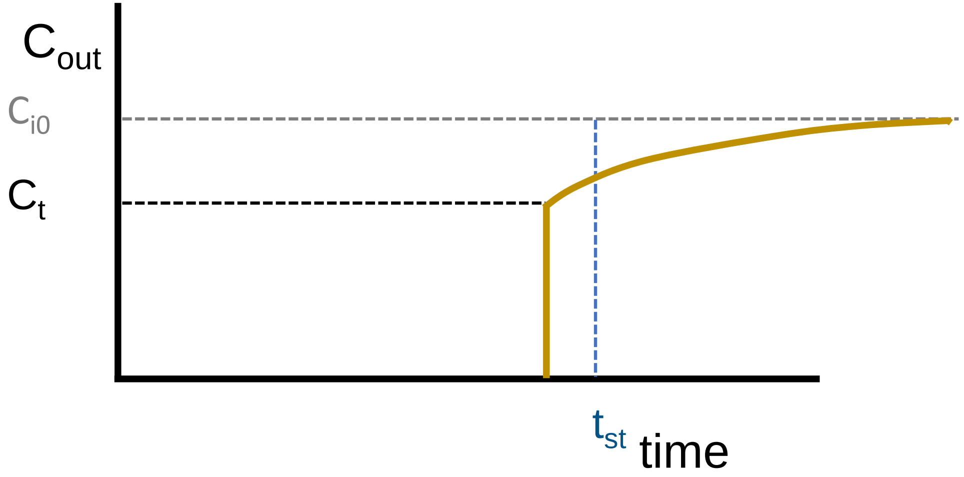
The input is just delayed without change

BET



Tangency point

Breakthrough curve – feed at C_{i0}

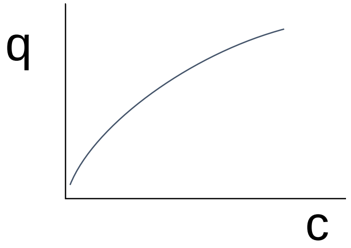


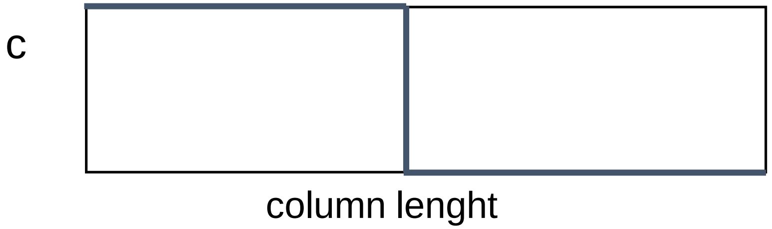
Composite front: shock up to C_t then dispersive

Equilibrium theory

The nature of the breakthrough curve is governed in first place by the adsorption equilibrium isotherm.

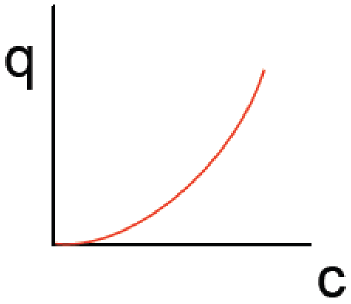
Favorable isotherms $\longrightarrow$ Compressive fronts (shock)

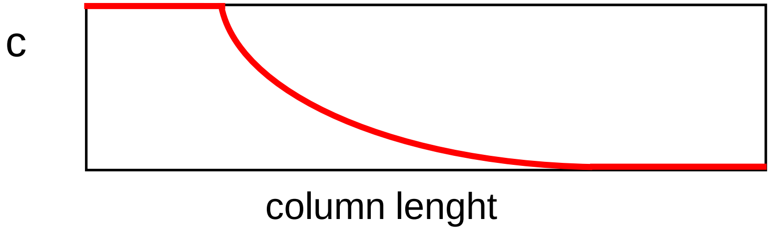




$$u_{sh} = \frac{u_i}{1 + \frac{1-\varepsilon}{\varepsilon}\frac{\Delta q_i}{\Delta c_i}}$$

Unfavorable isotherms $\longrightarrow$ Dispersive fronts





$$u_{c_i} = \frac{u_i}{1 + \frac{1-\varepsilon}{\varepsilon} f'(c)}$$

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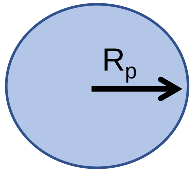
Intraparticle kinetics

Homogeneous particle



$$\frac{\partial q}{\partial t} = D_h \frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2 \frac{\partial q}{\partial r}\right)$$

Parabolic profile

$$q = a_0 + a_2 r^2 \qquad \frac{\partial q}{\partial r} = 2 a_2 r$$

$$q_s = a_0 + a_2 {R_p}^2 \qquad \left.\frac{\partial q}{\partial r}\right|_{R_p} = 2 a_2 R_p$$

$$\langle q \rangle = a_0 + a_2 \frac{3}{5} {R_p}^2$$

Averaging

$$\frac{{R_p}^3}{3}\frac{\partial \langle q \rangle}{\partial t} = D_h \left[r^2 \frac{\partial q}{\partial r}\right]_0^{R_p} = D_h {R_p}^2 \left.\frac{\partial q}{\partial r}\right|_{R_p}$$

$$\frac{\partial \langle q \rangle}{\partial t} = D_h \frac{3}{R_p} 2 a_2 R_p = 6\ a_2\ D_h$$

Linear driving force – LDF (Glueckauf)

$$\boxed{\frac{\partial \langle q \rangle}{\partial t} = k_h \left[q_s - \langle q \rangle \right]}$$

$$\boxed{k_h = \frac{15 D_h}{{R_p}^2}}$$

$$6\ a_2\ D_h = k_h \left[a_0 + a_2 {R_p}^2 - a_0 + a_2 \frac{3}{5} {R_p}^2 \right]$$

Intraparticle kinetics

Porous particle

$$\varepsilon_p \frac{\partial C_p}{\partial t} + \frac{\partial q}{\partial t} = D_{pe} \frac{1}{r^2} \frac{\partial}{\partial r}\left(r^2 \frac{\partial C_p}{\partial r} \right)$$

$$q = f\left(C_p\right)$$

Per volume of particle

Averaging

$$\varepsilon_p \frac{\partial \langle C_p \rangle}{\partial t} + \frac{\partial \langle q \rangle}{\partial t} = k_p \left(C_{ps} - \langle C_p \rangle \right)$$

$$k_p = \frac{15 D_{pe}}{{R_p}^2}$$

Linear isotherms

$$q = K C_p$$

$$\frac{\partial \langle C_p \rangle}{\partial t} = \frac{k_p}{\varepsilon + K}\left(C_{ps} - \langle C_p \rangle \right)$$

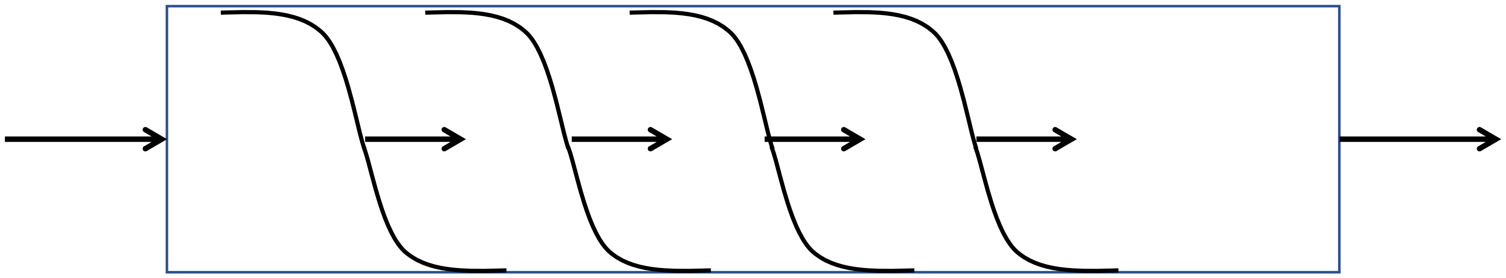
Equivalence with homogeneous particles

$$\boxed{\frac{\partial \langle q \rangle}{\partial t} = k_h \left[q_s - \langle q \rangle \right]}$$

$$\boxed{k_h = \frac{k_p}{K_h} = \frac{k_p}{\varepsilon_p + K}}$$

Constant patern

Stationary front



$$C_i(z+dz, t+dt) = C_i(z,t)$$

$$\left.\frac{\partial C_i}{\partial z}\right|_t = -\frac{1}{\lambda}\left.\frac{\partial C_i}{\partial t}\right|_z$$

$$\lambda = \left.\frac{\partial z}{\partial t}\right|_{C_i} = \frac{L}{t_{st}} = \frac{u_i}{1+\xi}$$

Velocity of the stationary front

Old models

I) Chemical kinetic type

II) Physical kinetic type

Old models

Chemical kinetic type – Thomas model

Mass balance

$$u_0 \frac{\partial C_i}{\partial z} + \varepsilon \frac{\partial C_i}{\partial t} + (1-\varepsilon)\frac{\partial \overline{q_i}}{\partial t} = 0$$

Kinetic law

$$\frac{\partial \overline{q_i}}{\partial t} = k_1\left[C\left(Q-\overline{q_i}\right) - \frac{1}{K}\overline{q_i}\left(C_0 - C_i\right)\right]$$

Boundary and initial conditions

$$z = 0 \quad C = C_0 \quad \forall t$$

$$t = \frac{z}{u_i} \quad q_i = 0 \quad \forall z$$

$$\boxed{\frac{C_i}{C_0} = \frac{J(rN, NT)}{J(rN, NT) + [1 - J(N, rNT)] \times e^{(r-1)N(T-1)}}}$$

$$r = \frac{1}{K}, \quad N = k_1\, Q\, \frac{z}{u_0}(1-\varepsilon), \quad NT = k_1\, C_0\left(t - \frac{z}{u_i}\right)$$

Old models

Chemical kinetic type – Simplifications

$r = 0 \qquad K \rightarrow \infty$

Bohart

$$\frac{C_i}{C_0} = \frac{e^{NT}}{e^{NT} + e^{N} - 1}$$

$r = 1$

Walter

$$\frac{C_i}{C_0} = J(N, NT)$$

$high \;\; r$

(Unfavorable isotherm K<1)

$$\frac{C_i}{C_0} = \frac{\sqrt{\frac{r}{T}} - r}{1 - r}$$

$r = 1, \; N \; and \; NT \; high$

Klinkenberg

$$\frac{C_i}{C_0} = \frac{1}{2}\left[1 + erf\left(\sqrt{NT} - \sqrt{N}\right)\right]$$

Old models

Physical kinetic type – Rosen model

Mass balance

$$u_0 \frac{\partial C_i}{\partial z} + \varepsilon \frac{\partial C_i}{\partial t} + (1-\varepsilon)\frac{\partial \overline{q_i}}{\partial t} = 0$$

Kinetic law for film mass transfer

$$\frac{\partial \overline{q_i}}{\partial t} = k_f \; a \left(C_i - C_i{}^s\right)$$

Intraparticle diffusion

$$\frac{\partial \, q_i(r,z,t)}{\partial t} = D_h \left[\frac{\partial^2 q_i}{\partial r^2} + \frac{2}{r}\frac{\partial q_i}{\partial r}\right]$$

Average

$$\overline{q_i}(z,t) = \frac{3}{R_p}\int_0^{R_p} q_i(r,z,t) r^2 dr$$

Isotherm

$$q_i = \frac{Q}{C_0} C_i = m C_i$$

$$\frac{C_i}{C_0} = \frac{1}{2} + \frac{2}{\pi}\int_0^{\infty} e^A \sin B \frac{d\lambda}{\lambda}$$

A and B depend on the model parameters

$$Bi = \frac{k_f R_p}{D_h}, \quad N_f = k_f \; a \; \tau, \quad m$$

Methodology

Prior to any simulation, necessary data must be gathered independently:

I. measurement of single adsorption isotherms;

II. measurement of intraparticle diffusivities (ZLC);

III. measurement of film mass transfer (shallow bed);

IV. measurement of axial dispersion (tracer);

V. measurement of breakthrough curves for single and feed mixtures;

VI. modeling breakthrough curves and model validation;

VII. modeling/simulation of process (PSA; SMB);

VIII. lab-scale operation of process and model validation;

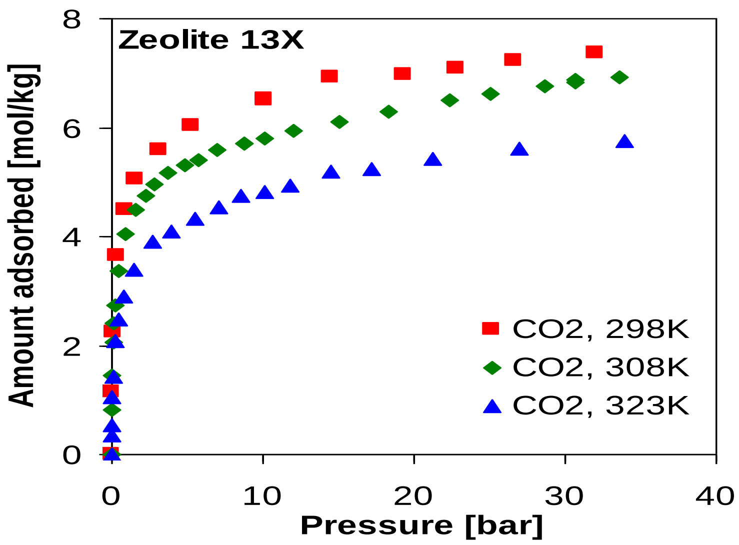
IX. sizing and scale-up of industrial process.

Methodology

Single adsorption isotherms - Liquid phase in batch systems

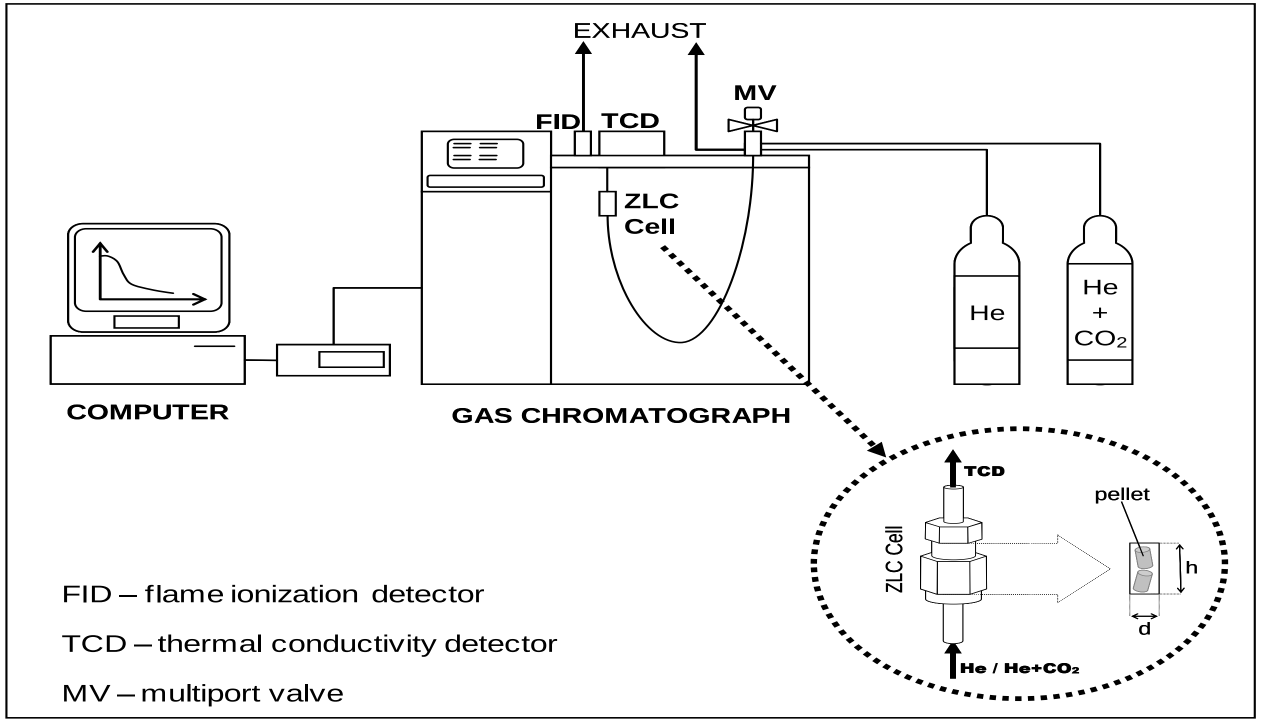
Methodology

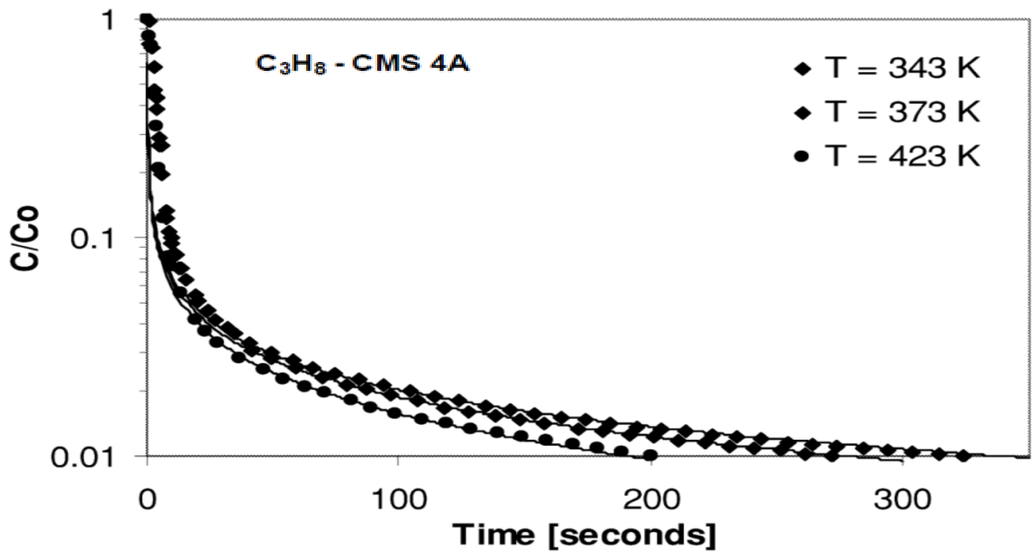
Single adsorption isotherms - Gas phase in Rubotherm magnetic balance



Methodology

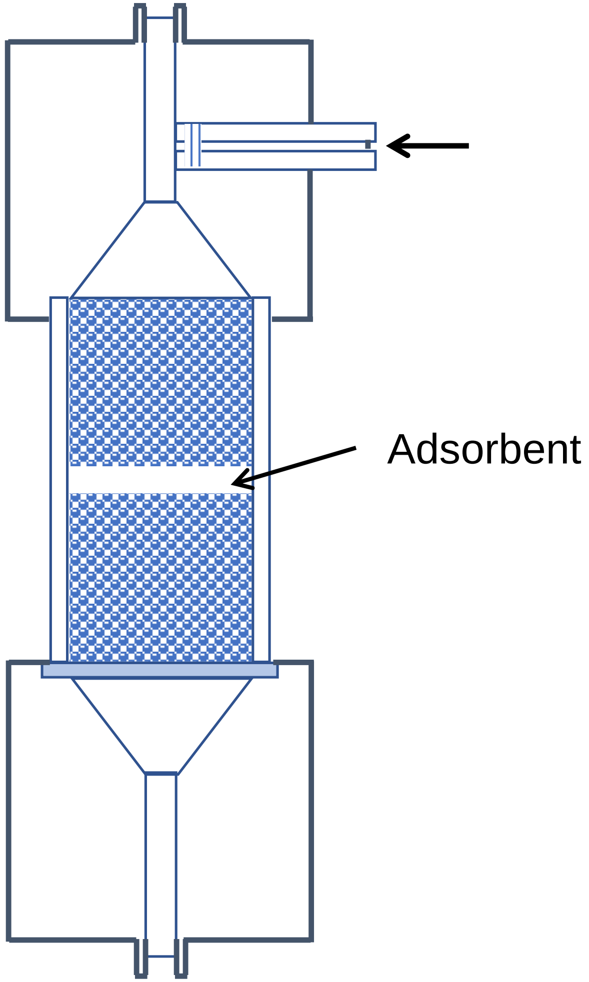
Intraparticle diffusivities - Zero-length column (ZLC) method

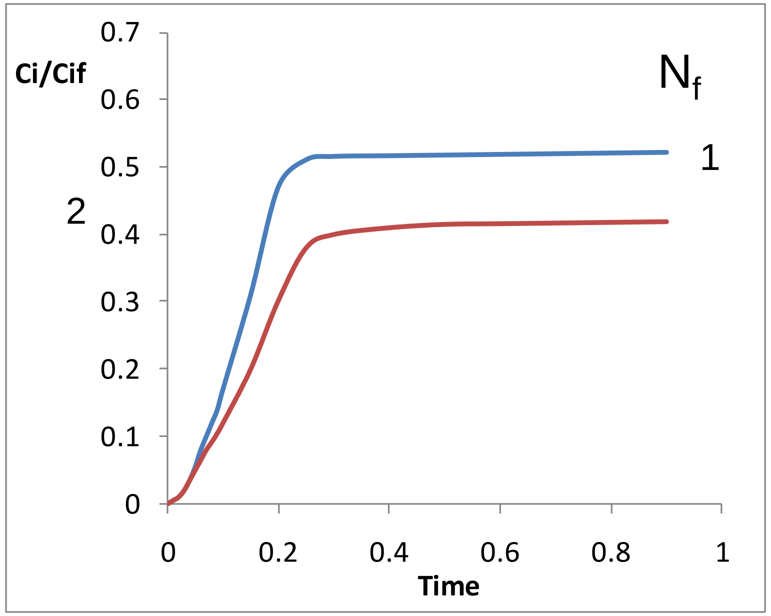




Methodology

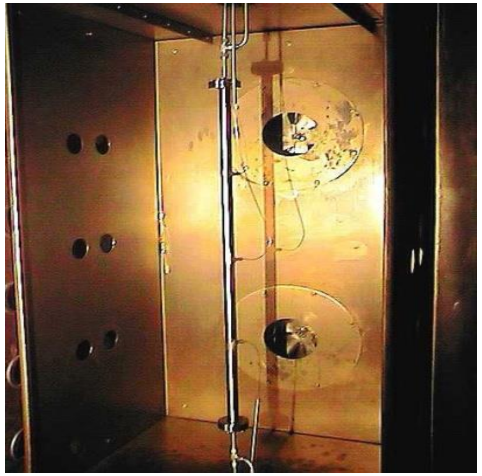
Film mass transfer - shallow-bed technique

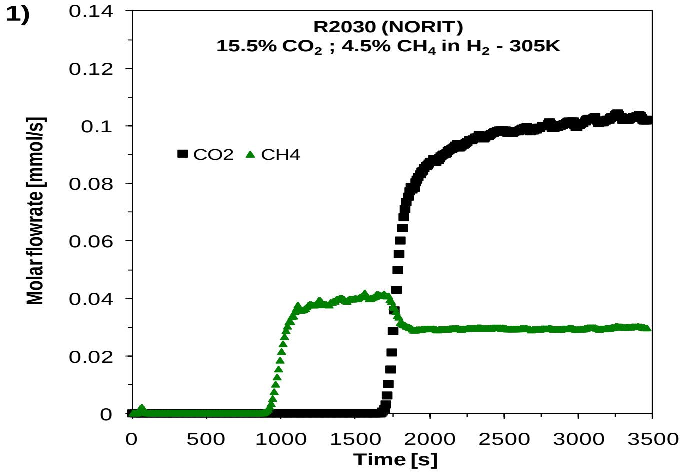




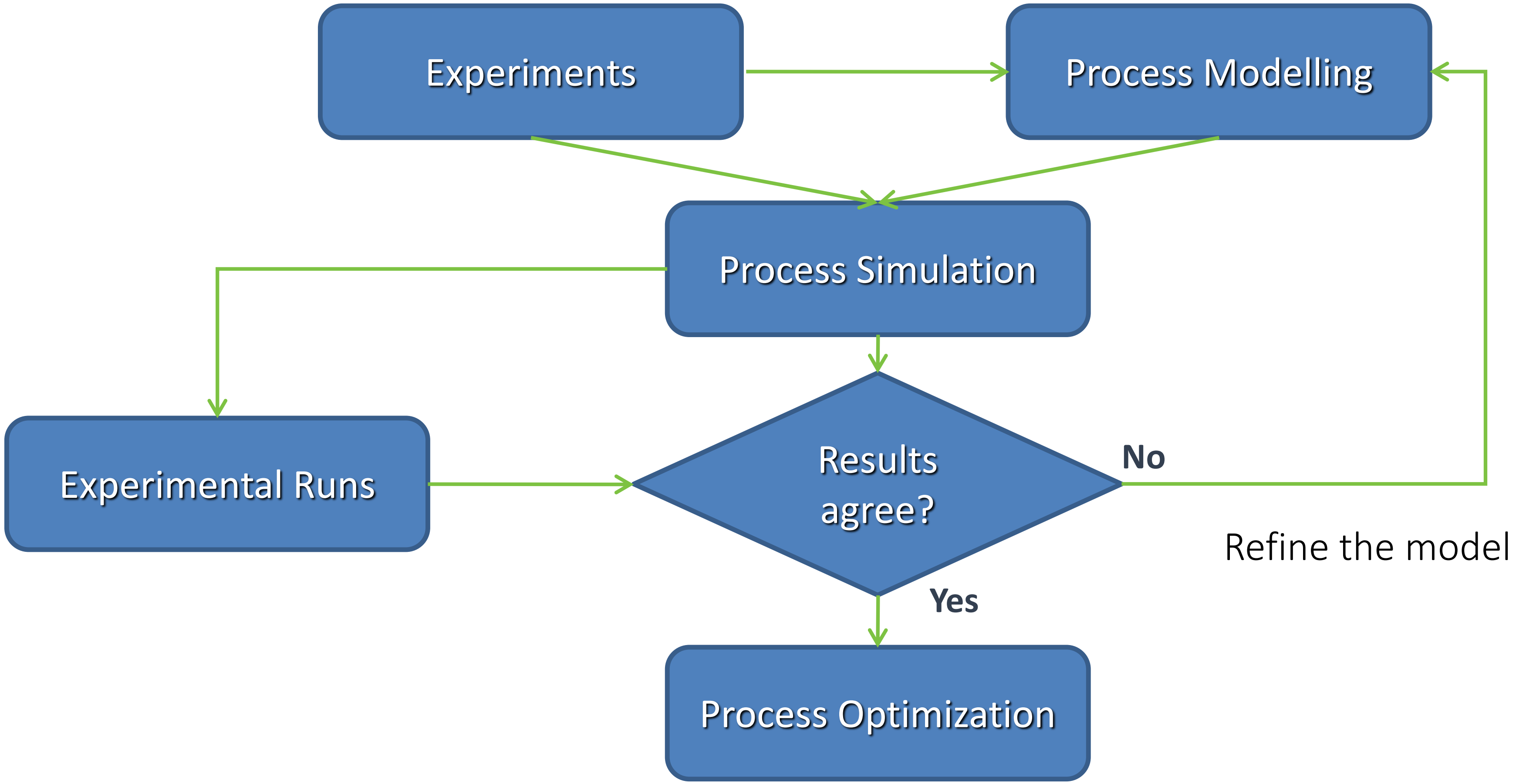
Methodology

Axial dispersion / model validation - tracer experiments and breakthrough curves for single and feed mixtures



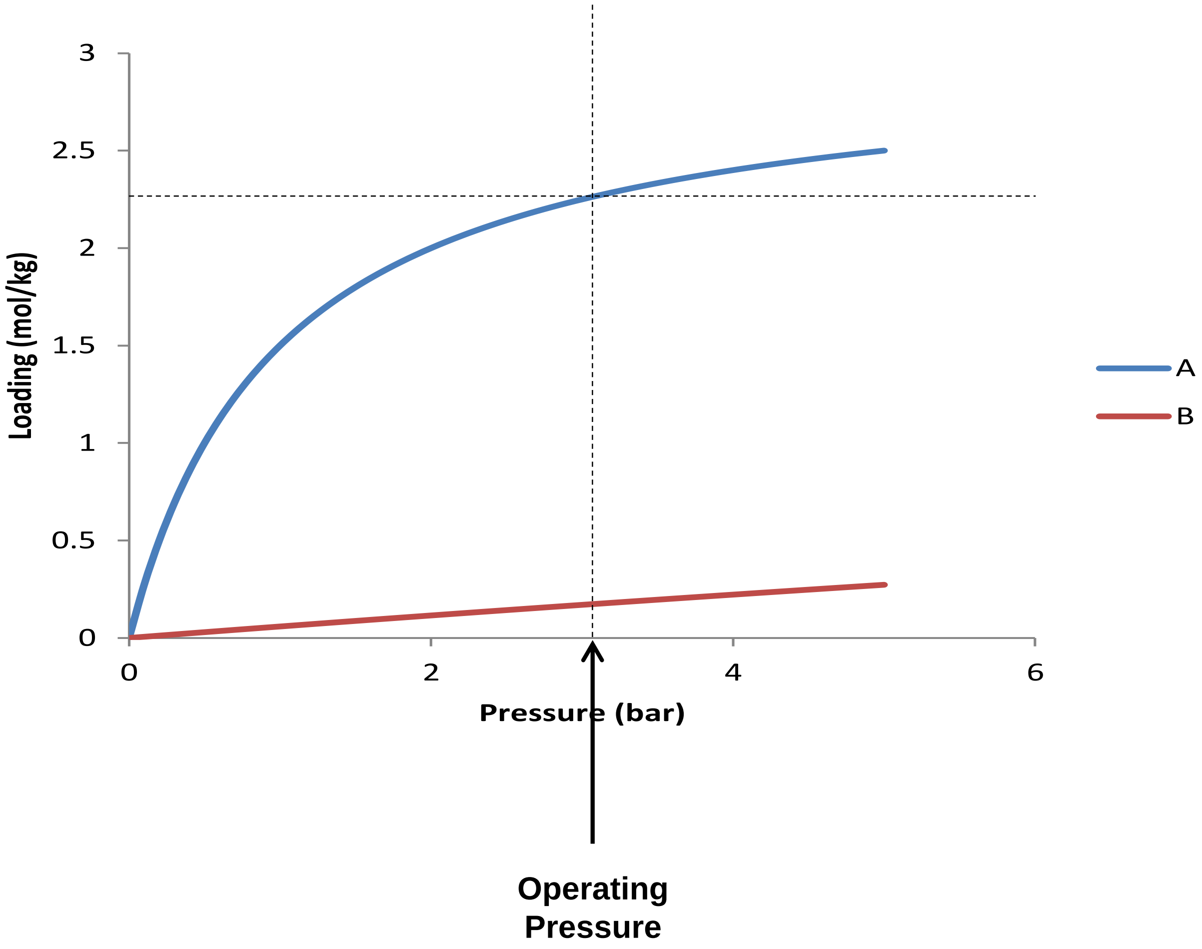


Methodology



Pressure Swing Adsorption

How can we obtain B from a mixture of A+B?

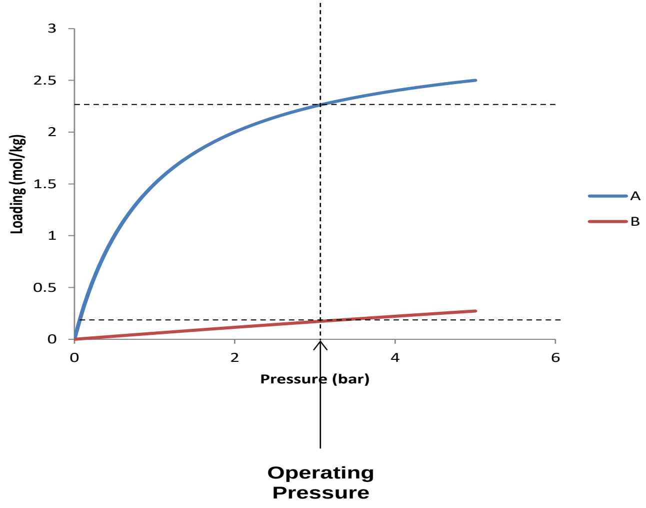


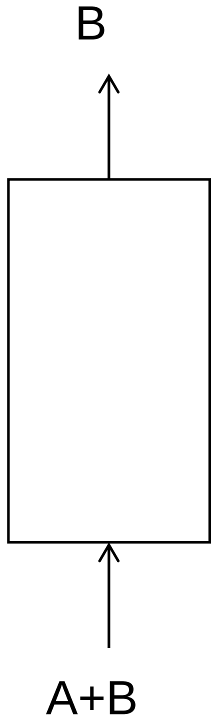
Let's choose an operating pressure considering:

- Acceptable capacity and selectivity;
- Maximum pressure allowed by the adsorbent and unit/equipment;
- Energy consumption;
- Product requirements.

Pressure Swing Adsorption

How can we obtain B from a mixture of A+B?





If we pass a stream containing A+B at the operating pressures, through a bed packed with this adsorbent:

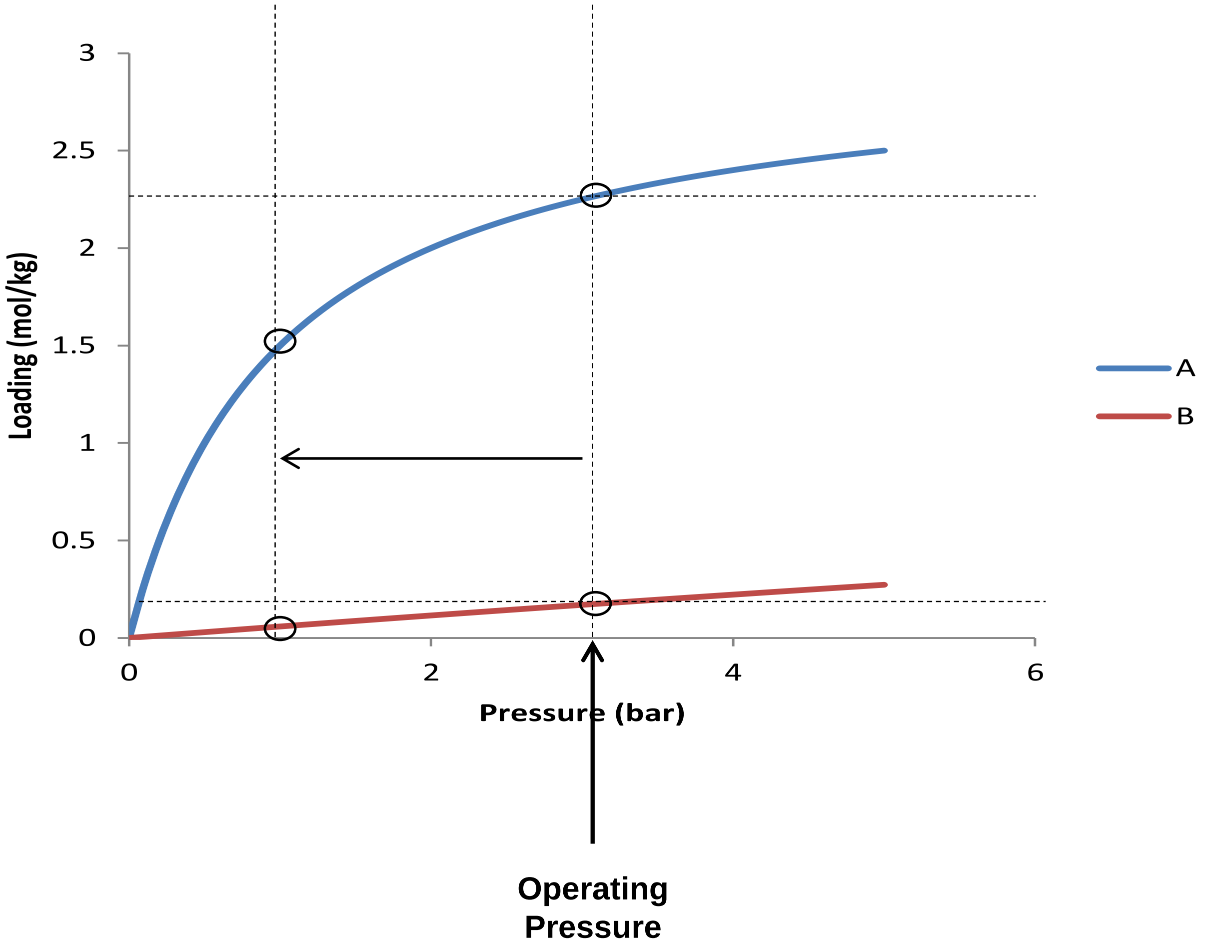
- A will be preferentially adsorbed (retained by the adsorbent);
- B will move through the bed and leave it at the top.

This will happen until the adsorbent is saturated with A.

What should we do next?

Pressure Swing Adsorption

How can we obtain B from a mixture of A+B?

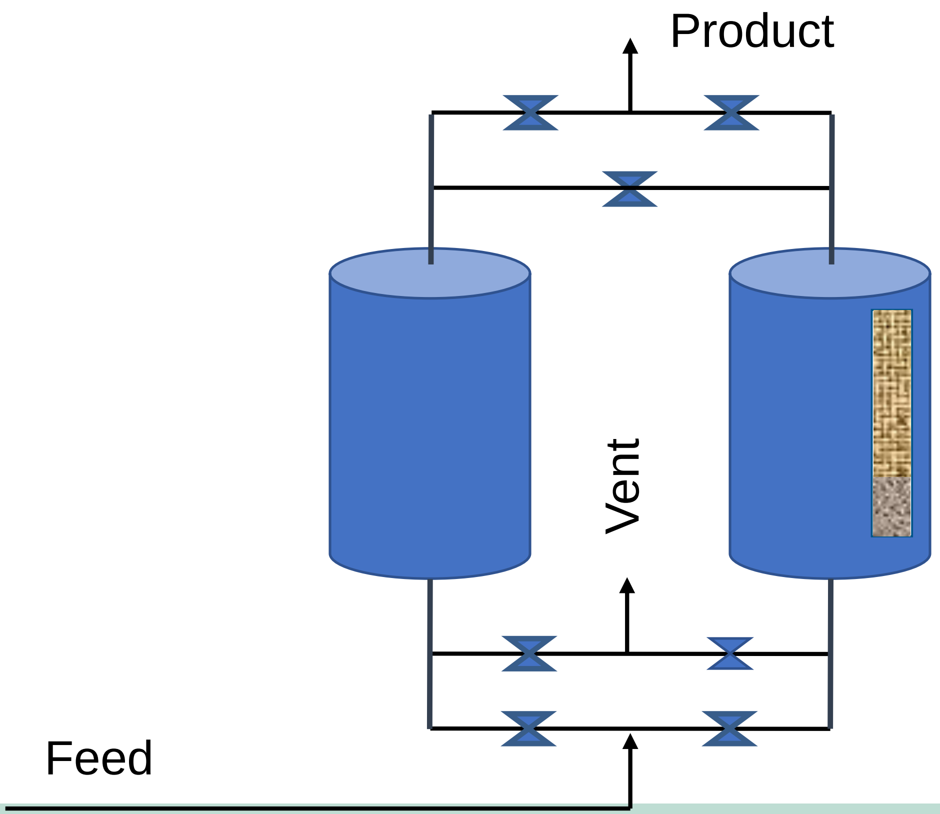


If we decrease the pressure the adsorption capacity also decreases.

For example, if we open the bed to the atmosphere and let the gas out, the final pressure will be 1 bar.

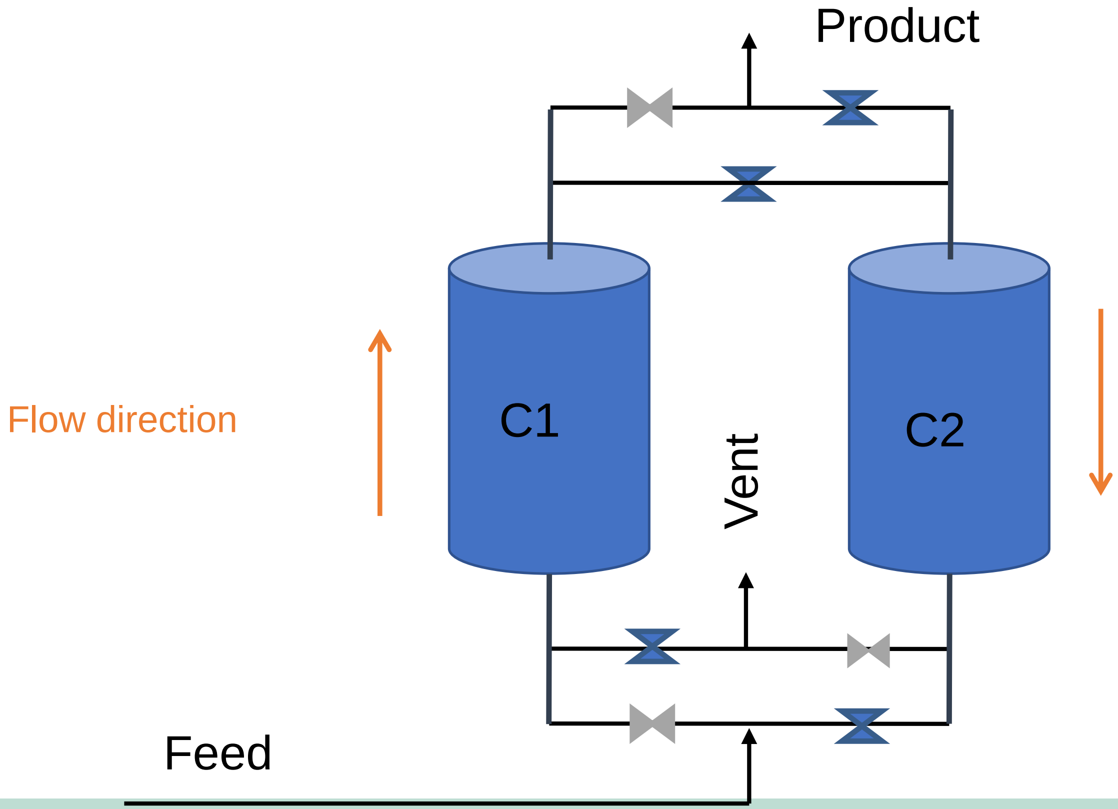
Pressure Swing Adsorption

PSA units are usually composed by two or more columns packed with one or more adsorbents



Pressure Swing Adsorption

PSA units are usually composed by two or more columns packed with one or more adsorbents

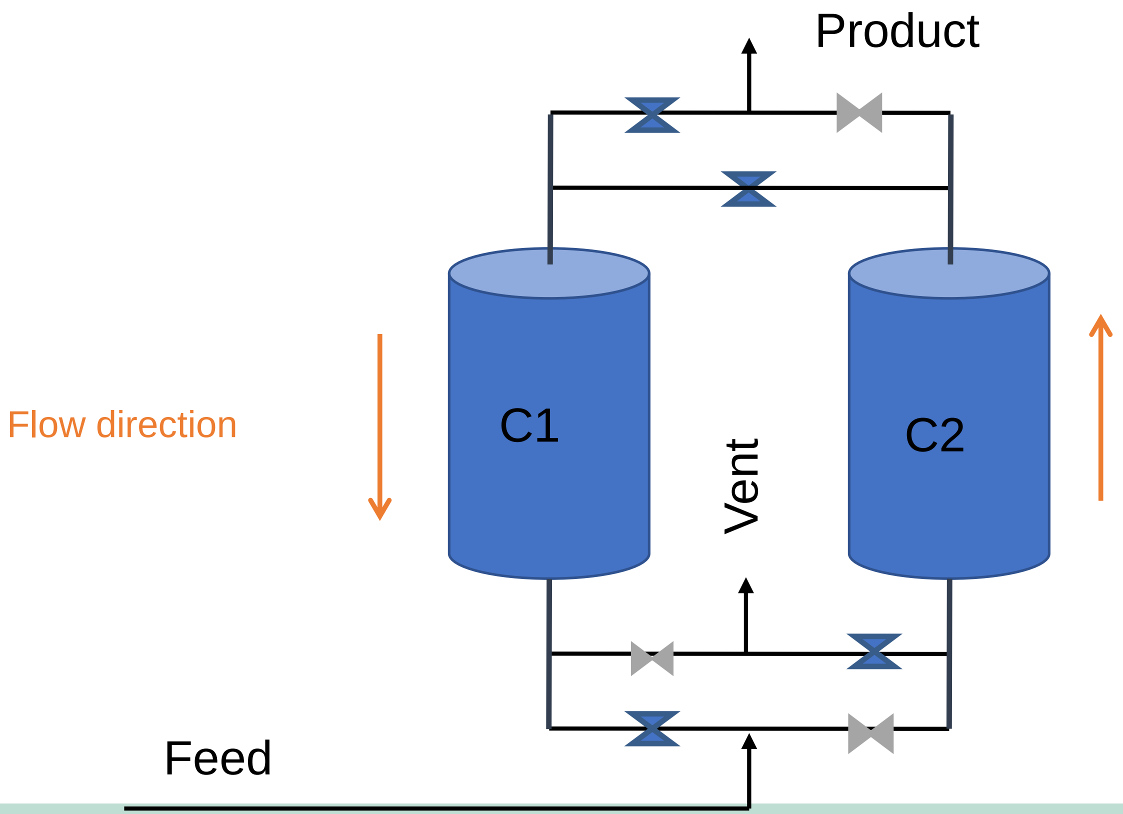


C1 – Production

C2 - Regeneration

Pressure Swing Adsorption

PSA units are usually composed by two or more columns packed with one or more adsorbents



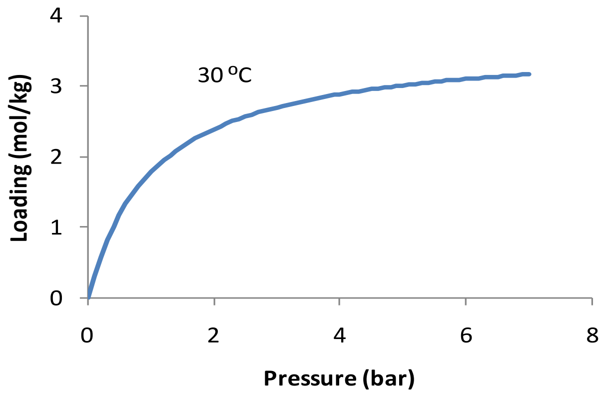
C1 – Regeneration

C2 - Production

Adsorbent regeneration - PSA, TSA, CSA

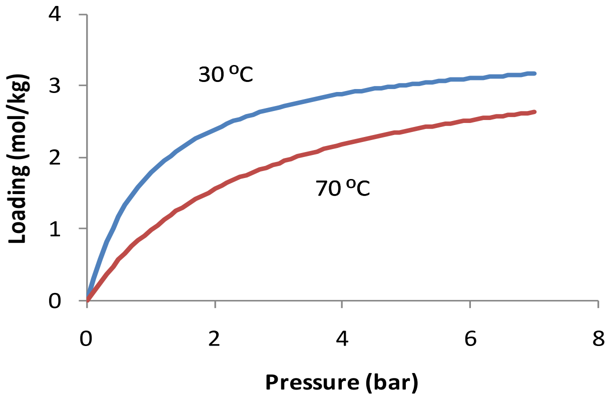
Pressure Swing Adsorption

The adsorbent is regenerated by lowering the pressure.



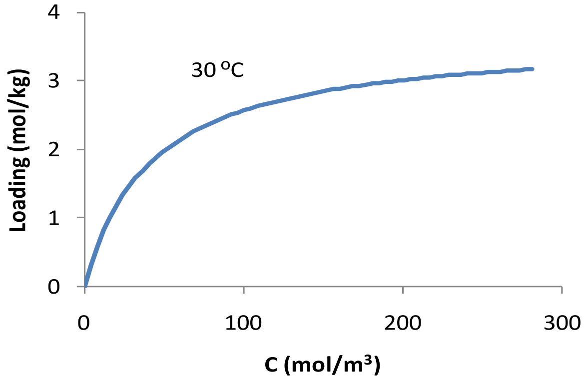
Temperature Swing Adsorption

The adsorbent is regenerated by increasing the temperature.



Concentration Swing Adsorption

The adsorbent is regenerated by lowering the concentration.



SMB principle (Sorbex Processes-UOP)