



NTNU – Trondheim
Norwegian University of
Science and Technology

Department of chemical engineering

Examination paper for TKP4105 (Separation Technology)

**Academic contact during examination: May-Britt Hägg Phone: 930 80834
Sigurd Skogestad, Phone: 931 71669**

Examination date: 16.12.13

Examination time (from-to): 09:00 – 13:00

**Permitted examination support material: No printed or handwritten material
permitted. Simple calculator code D
accepted.**

Other information:

Language: English

Number of pages: 5 (frontpage included)

Number of pages attachment enclosed: 2

mm-paper is needed!

Checked by:

Date Signature

Problem 1 Gas separation with membranes 25%

A feed stream with mixed gas contains 50% of gas A and 50% of gas B. These gases are to be separated with a membrane. Gas A is the fastest permeating gas, while gas B is the valuable gas which we want to be as clean as possible. (This mixed gas can for instance be biogas, in other words, A = CO₂ and B = CH₄)

Use ideal mixing model for the calculations. Useful information about the system is given below, and also equations in the Attachment 1.

- Calculate the minimum concentration of component A which can be achieved in the retentate stream. How clean is in this case gas B?
- Assume that we are, in our case, able to achieve a concentration of A in the retentate which is 25% higher than the one you have calculated in a). What will then be the composition of the permeate stream? How much of gas B (give as vol%) will be lost in the permeate stream?
- How large membrane area is needed to perform this separation?
- Do you have any suggestions on how to reduce the loss of B by doing some changes to the process?

Gitt for systemet:

Feed stream, $q_f = 2 \text{ m}^3(\text{STP}) / \text{min}$
Feed pressure: 10 bar
Permeate pressure: 1 bar (1 bar = 76 cm Hg)
Membrane thickness: $t = 10^{-6} \text{ m}$
Permeability of A: $P'_A = 400 \text{ Barrer}$ (1 Barrer = $10^{-10} \text{ cm}^3(\text{STP}) \cdot \text{cm} / (\text{s} \cdot \text{cm}^2 \cdot \text{cm Hg})$)
Selectivity of P'_A/P'_B : $\alpha = 30$
Fraction of the feed permeated: $\theta = 0,53$

See also Attachment 1 for equations

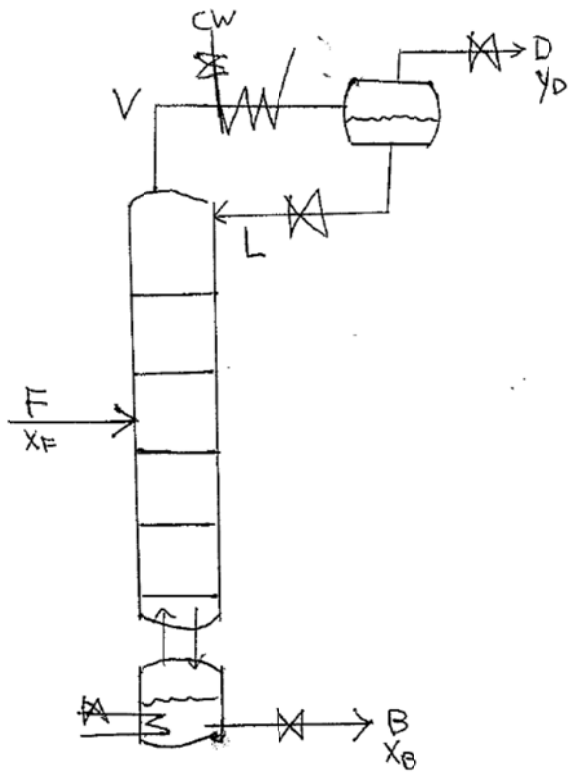
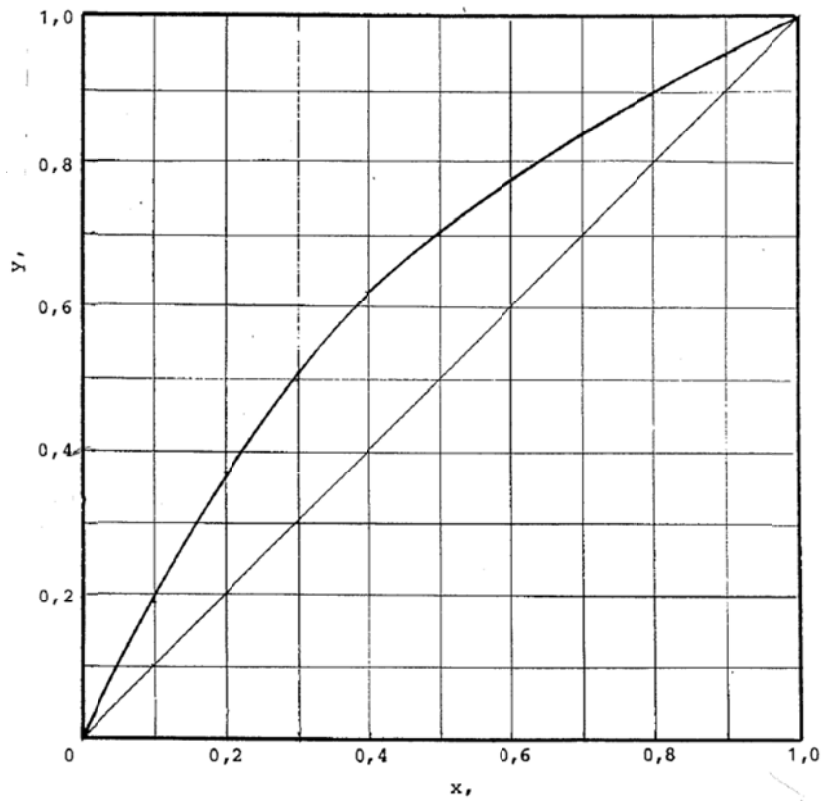
Problem 2 Distillation 25%

A distillation column has 7 theoretical stages (reboiler + 3 ideal plates below the feed + 2 ideal plates over the feed + partial condenser). The feed is 10 kmol/h and is a binary saturated liquid with $x_f=0.5$ (mole fraction A). The column operates at 2 atm, and the xy equilibrium data at 2 atm is given in the figure.

Operation specifications: The top product (D) should have $y_D=0.90$ (mole fraction A) and the molar liquid to vapor ratio in the upper part of the column should be $L/V=0.86$.

- Find the mole fraction x_B of component A in the bottom product and find the amount of desitillate D and bottom product B [kmol(h)].
 - How does the answer change if the feed is moved one stage down (so that there are 3 ideal plates above the feed)?
- Control (can be done independently): Suggest a control structure which satisfies the operation specifications. (What are the MVs, CVs and suggested pairings?)

(See next page for 2 figures given)



Figures for Problem 2

Problem 3 - Absorption 25%



$$\frac{y_1 - y_1^*}{y_2 - y_2^*} = A^N \quad (1)$$

$$A = \frac{L/V}{m} \quad (2)$$

$$A = \frac{y_1 - y_2}{y_1^* - y_2^*} \quad (3)$$

The flue gas (V_1) from a coal power plant is 3000 mol/s (84 kg/s) at 1bar and contains 0.2 mol% SO₂ ($y_1=0.002$). We want to remove at least 95% of the SO₂ ($y_2 < 0.0001$) by absorption with 90000 mol/s (1600 kg/s) of sea water ($x_2=0$) at 25 C (wet scrubbing); see the Figure.

Equilibrium data (Henry's constant for SO₂ in water; $p_{SO_2} = Hx$ where x is mole fraction of SO₂):
 $H=115$ bar (50°C), $H=46$ bar (25°C), $H=22$ bar (5°C).

- Even with a large column (with a large number of equilibrium stages, N) one was not able to reach the specification. Can you explain why? What is the SO₂ contents (y_2) in the exit gas if you assume a very large column ($N \rightarrow \infty$)?
- To remove more SO₂, it is suggested build an additional column, that is, the exit gas (y_2) is send to a second identical absorption column (which also has 90000 mol/s of sea water and a large number of stages). Make a flowsheet of the proposed process. Does this help? What is the SO₂ contents (y_2') after the second absorption column with a large column ($N \rightarrow \infty$)?
- Instead of building a second column, the following modifications for the original design have been suggested:
 - Increase the sea water flow by a factor 2
 - Decrease the temperature from 25°C to 5°C.
 - Increase the pressure from 1 bar to 2 bar

Would any of these modifications give acceptable SO₂ removal? Which modification would you suggest?

- For calculations in part (c) you may use the Kremser equation (1) given in the figure with $N=10$.
 - What are the assumptions behind equation (1)?
 - How are y_1^* and y_2^* defined?
 - A is defined by (2), but it is also given by (3). Use an xy -diagram with operation line and equilibrium line (McCabe-Thiele) to prove that (3) is correct.

Problem 2 Adsorption 25%

Using molecular sieves, water vapor is removed from N₂ gas in a packed bed column. At 28°C. The column height is 0.3 m and has a bulk density of the solid equal to 712.8 kg/m³. The initial water concentration in the solid is 0.01 kg H₂O / kg solid, and the mass velocity of the N₂ gas is 4052 kg/(m²·h). The initial water concentration coming in with the gas is $c_0 = 926 \times 10^{-6}$ kg H₂O / kg N₂

The break through data for mass transfer zone is given below. A value of $c/c_0 = 0.02$ is preferred for the break point.

t (h)	0	9	9.2	9.6	10	10.4
c (kg H ₂ O/kg N ₂ × 10 ⁶)	<0.6	0.6	2.6	21	91	235
t (h)	10.8	11.25	11.5	12.0	12.5	12.8
c (kg H ₂ O/kg N ₂ × 10 ⁶)	418	630	717	855	906	926

- Draw the curve c/c_0 as function
- Determine the break point time, and the height of “unused bed”
- Determine the fraction of total capacity used up to this point.
- Determine the loading capacity of the column for uptake of water up to the break point.

3.4 COMPLETE-MIXING MODEL FOR GAS SEPARATION BY MEMBRANES

3.4A Basic Equations Used

In Fig. 13.4-1 a detailed process flow diagram is shown for complete mixing. When a separator element is operated at a low recovery (i.e., where the permeate flow rate is a small fraction of the entering feed rate), there is a minimal change in composition. Then the results derived using the complete-mixing model provide reasonable estimates of permeate purity. This case was derived by Weller and Steiner (W4).

The overall material balance (Fig. 13.4-1) is as follows:

$$q_f = q_o + q_p \quad (13.4-1)$$

where q_f is total feed flow rate in $\text{cm}^3(\text{STP})/\text{s}$; q_o is outlet reject flow rate, $\text{cm}^3(\text{STP})/\text{s}$; and q_p is outlet permeate flow rate, $\text{cm}^3(\text{STP})/\text{s}$. The cut or fraction of feed permeated, θ , is given as

$$\theta = \frac{q_p}{q_f} \quad (13.4-2)$$

The rate of diffusion or permeation of species A (in a binary of A and B) is given below by an equation similar to Eq. (6.5-8) but which uses $\text{cm}^3(\text{STP})/\text{s}$ as rate of permeation rather than flux in $\text{kg mol}/\text{s} \cdot \text{cm}^2$.

$$\frac{q_A}{A_m} = \frac{q_p y_p}{A_m} = \left(\frac{P'_A}{l} \right) (p_h x_o - p_l y_p) \quad (13.4-3)$$

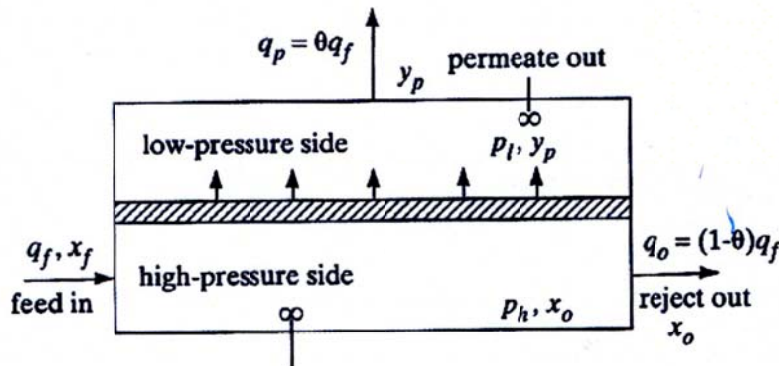


FIGURE 13.4-1. Process flow for complete mixing case.

where P'_A is permeability of A in the membrane, $\text{cm}^3(\text{STP}) \cdot \text{cm}/(\text{s} \cdot \text{cm}^2 \cdot \text{cm Hg})$; q_A is flow rate of A in permeate, $\text{cm}^3(\text{STP})/\text{s}$; A_m is membrane area, cm^2 ; t is membrane thickness, cm ; p_h is total pressure in the high-pressure (feed) side, cm Hg ; p_l is total pressure in the low-pressure or permeate side, cm Hg ; x_o is mole fraction of A in reject side; x_f is mole fraction of A in feed; and y_p is mole fraction of A in permeate. Note that $p_h x_o$ is the partial pressure of A in the reject gas phase.

A similar equation can be written for component B.

$$\frac{q_B}{A_m} = \frac{q_p(1 - y_p)}{A_m} = \left(\frac{P'_B}{t}\right)[p_h(1 - x_o) - p_l(1 - y_p)] \quad (13.4-4)$$

where P'_B is permeability of B, $\text{cm}^3(\text{STP}) \cdot \text{cm}/(\text{s} \cdot \text{cm}^2 \cdot \text{cm Hg})$. Dividing Eq. (13.4-3) by (13.4-4)

$$\frac{y_p}{1 - y_p} = \frac{\alpha^*[x_o - (p_l/p_h)y_p]}{(1 - x_o) - (p_l/p_h)(1 - y_p)} \quad (13.4-5)$$

This equation relates y_p , the permeate composition, to x_o , the reject composition, and the ideal separation factor α^* is defined as

$$\alpha^* = \frac{P'_A}{P'_B} \quad (13.4-6)$$

Making an overall material balance on component A

$$q_f x_f = q_o x_o + q_p y_p \quad (13.4-7)$$

Dividing by q_f and solving for the outlet reject composition,

$$x_o = \frac{x_f - \theta y_p}{(1 - \theta)} \quad \text{or} \quad y_p = \frac{x_f - x_o(1 - \theta)}{\theta} \quad (13.4-8)$$

Substituting $q_p = \theta q_f$ from Eq. (13.4-2) into Eq. (13.4-3) and solving for the membrane area, A_m ,

$$A_m = \frac{\theta q_f y_p}{(P'_A/t)(p_h x_o - p_l y_p)} \quad (13.4-9)$$

13.4C Minimum Concentration of Reject Stream

If all of the feed is permeated, then $\theta = 1$ and the feed composition $x_f = y_p$. For all values of $\theta < 1$, the permeate composition $y_p > x_f$ (H1). Substituting the value $x_f = y_p$ into Eq. (13.4-5) and solving, the minimum reject composition x_{oM} for a given x_f value is obtained as

$$x_{oM} = \frac{x_f \left[1 + (\alpha^* - 1) \frac{p_l}{p_h} (1 - x_f) \right]}{\alpha^*(1 - x_f) + x_f} \quad (13.4-12)$$

Hence, a feed of x_f concentration cannot be stripped lower than a value of x_{oM} even with an infinitely large membrane area for a completely mixed system. To strip beyond this limiting value a cascade-type system could be used. However, a single unit could be used which is not completely mixed but is designed for plug flow.