UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2008/2009

November 2008

EKC 511 – Advanced Separation Process

Duration : 3 hours

Please check that this examination paper consists of FIVE pages of printed material and TWO pages of Appendix before you begin the examination.

Instructions: Answer **FOUR** (4) questions.

1. [a] Why application of pervaporation in chemical industry usually hybrid with distillation and reactor? Using simple sketches, describe the hybrids of pervaporation-distillation and pervaporation-reactor, respectively. In each type of the hybrid process, you should give two different types of layout and able to distinguish among them.

[10 marks]

[b] An ultrafiltrion tubular module is used to clarify apple juice. The ultrafiltration tubes have a diameter of 1 cm and a length of 2 m. The juice circulates inside the tubular membrane at a velocity of 1 m/s. Due to the presence of pectin in the surroundings of the tubular membrane wall, a gel layer is formed when the concentration of the pectin reaches a value of 5.5% (w/V). If the pectin content in the juice fed to the ultrafiltration module is 0.5% (w/V), determine:

[i] The flux equation for ultrafiltration.

[2 marks]

[ii] The number of the tubes needed if it is desired to-obtain 120 L/h of such juice.

[11 marks]

[iii] Since the presence of the pectin in the juice fed is unavoidable, suggest a method to reduce the forming of gel layer on the membrane wall.

[2 marks]

Data given for juice properties:
density 1025 kg/m^3
viscosity 1.4 kg/m.s
solute diffusivity 9×10^{-11} m^2/s

2. [a] Describe why pillared clays and carbon nanotubes are becoming important adsorbents nowadays?

[10 marks]

[b] A thermal swing adsorption process is used to remove traces of toluene from n-heptane using silica gel as adsorbent. The adsorber operates at 1 atm. The feed is 0.0011 wt. fraction toluene and 0.9989 wt. fraction n-heptane at 0°C. Superficial velocity of the feed is 10 cm/min. The absorber is 2 m long and adsorption is taking place at 0°C. The feed step is continued until breakthrough occurs. To regenerate, use counterflow of pure n-heptane at 80°C. Superficial velocity during purge is 10 cm/min. Column is cooled to 0°C before the next adsoption cycle.

Assume that wall heat capacities can be ignored, heat of adsorption is negligible, no adsorption of n-heptane. Using the solute movement theory, determine:-

[i] the breakthrough time for toluene during the feed step
[ii] time for thermal wave to breakthrough
[iii] time to remove all toluene from column
[iv] the outlet concentration profile of the regeneration fluid

[15 marks]

Data given: At low concentration isotherms for toluene:
Isotherms q = 17.46x at 0°C, and q = 1.23x at 80°C
q and x are in g solute/g adsorbent and g solute/g fluid, respectively
structural density of solid, $\rho_s = 2100$ kg/m^3
fluid density, $\rho_f = 684$ kg/m^3
heat capacity of the solid, $C_{p,s} = 2000$ J/kg°C
heat capacity of the fluid, $C_{p,f} = 1841$ J/kg°C
external porosity, $\varepsilon_e = 0.43$
internal or pore porosity, $\varepsilon_p = 0.48$
size exclusion parameter, $K_d = 1.0$

3. It is required to separate components A and B from a mixture F containing 30 mole % of A in order to obtain a concentrate of 95 mole% of A with not more than 4 mole % of A as bottoms. The feed flow-rate is 100 kmol/h. However, A and B form an azeotrope at approximately 64 mole % of A at atmospheric pressure making it impossible to separate under single atmospheric distillation processes. A two pressure distillation system as shown in Figure Q.3 is to be designed in order to achieve the separation. The column 1 is to operate at 1 atmosphere while the column 2 at 5 atmosphere. The equilibrium data for 1 atmosphere and 5 atmosphere are shown in Tables Q.3.i and ii respectively where x and y refers to the mole fraction of A. Estimate:

[a] Flow-rates B_1,B_2, D_1 and D_2

[10 marks]

[b] The number of plates in all sections of the column 1.

[15 marks]

Assume that the streams F and D_1 are boiling liquids and D_2 is a vapour at boiling point. The reflux ratio of the atmospheric pressure column is 1.5 times the minimum whereas are for the other column, the reflux ratio is 5 times the minimum. The compositions of streams D_1 and D_2 are 60 mole % and 50 mole % of A respectively.

Table Q.3.i : Equilibrium Data for A and B at 1 atmosphere

x	0	0.1	0.2	0.3	0.4	0.5	0.6	0.65	0.7	0.8	0.9	1
y	0	0.17	0.31	0.43	0.52	0.59	0.637	0.65	0.68	0.69	0.83	1

Table Q.3.ii : Equilibrium Data for A and B at 5 atmospheres

x	0.4	0.6	0.8	0.9	0.95	1
y	0.4	0.52	0.65	0.75	0.85	1

x and y are mole fractions of A in the mixture.

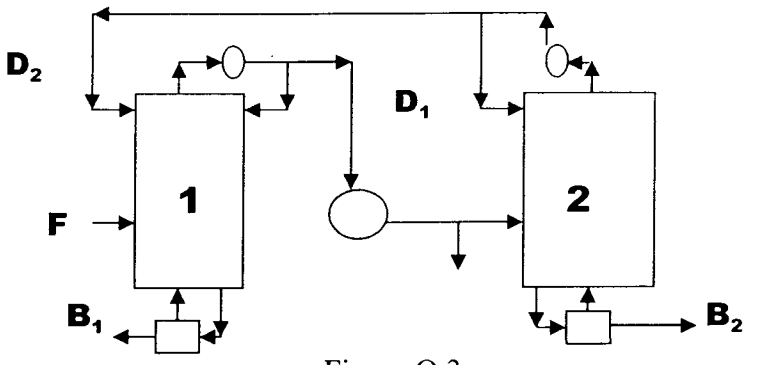

Figure Q.3

4. [a] A typical supercritical fluid extraction system using a packed bed is shown in Figure Q.4.[a]. The solubilities of the solute on the fluid on the low pressure (P_L) and high pressure (P_H) sides are C_{sh} (g/kg) and C_{sl} (g/kg) respectively. r % of the fluid entering the regeneration unit can be assumed to be lost thereby requiring a make-up of fresh solvent for the system as shown in the Figure. The mass transfer area exposed to the supercritical fluid is A_{EX} and the mass transfer coefficient is k_c (m/s). The density of the supercritical fluid on the high pressure side is ρ_H. Show that, under mass transfer controlled conditions for extraction, the rate of extraction R (g/s) of solute in the product P1 can be expressed as:

$$R = L_m.[C_{SH} - (1.0 - 0.01r)C_{SL}][1.0 - \exp(-\eta)]$$

where : $\eta = \left[A_{EX}.k_c \rho_{SH} / [L_m] \right]$

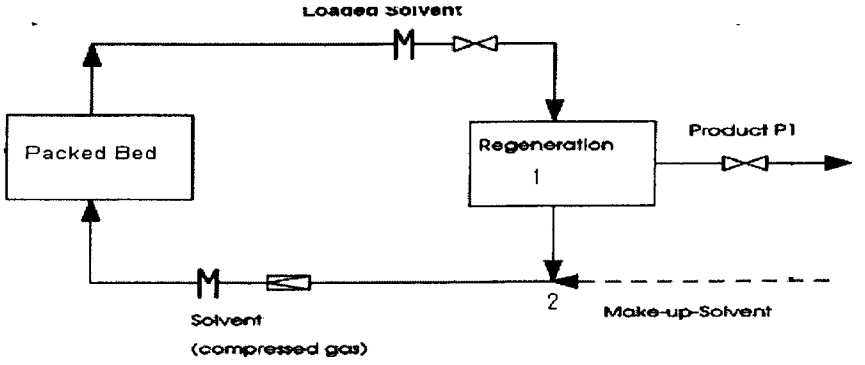

Figure Q.4.[a]

[10 marks]

[b] A laboratory pilot extraction unit for extraction of caffeine from beans using supercritical CO_2 as represented in Figure Q.4.[a] has been used .The percentage yield of caffeine is shown in Table Q.4. The solubility data of caffeine in supercritical CO_2 and densities of supercritical CO_2 are shown in Figures Q.4.[b] and Q.4.[c]. The high and low operating pressures of the system are 32MPa and 16MPa. The operating temperature of both sides is kept constant at 340 K. Assume the CO_2 leaving the regenerator is saturated with caffeine at the regenerator pressure and temperature. The extraction bed makes use of 50kg of beans having 0.31 g/kg of beans. Flowrate of the supercritical fluid on the high pressure side is 0.02 m^3/h. 5% of the carbon dioxide may be assumed to be lost on regeneration.

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Table Q.4.

Time Hrs	2	4	6	8	10	12	14	16	18
Yield %	13.6	24.7	33.8	39	43	48	51	52.5	56

[i] Draw a plot indicating the total extract of caffeine (g) vs time

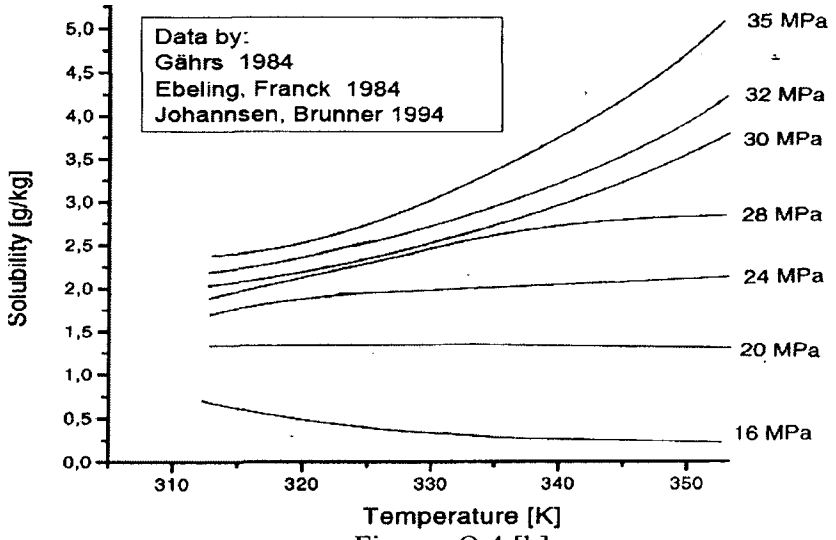
[5 marks]

[ii] Estimate the time at which the mass transfer in extraction deviates from the film mass transfer control to diffusion control conditions.

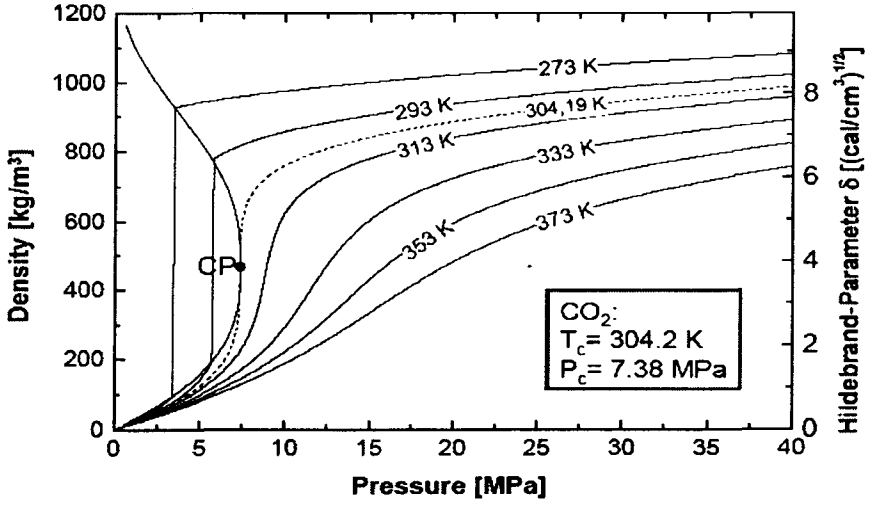
[5 marks]

[iii] The value of factor $k_c.A_{EX}$ for the extraction bed. (Use equation derived in part [i] of this question

[5 marks]



Figures Q.4.[b]



Figures Q.4.[c]

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Appendix

Useful formulas:

Reynolds numbers, $\mathrm{Re} = \dfrac{\rho\, u_b\, d_t}{\mu}$

Schmidt number, $\mathrm{Sc} = \dfrac{\mu}{\rho\, D}$

Sherwood number, $\mathrm{Sh} = \dfrac{d_t k}{D} = 0.023\,\mathrm{Re}^{0.83}\,\mathrm{Sc}^{1/3}$, turbulent flow

Concentration polarization module, $M = \dfrac{x_w}{x_r} = \exp\left(\dfrac{J_{solv}}{k}\right)$

Where,

ρ - density, kg/m^3
u_b - bulk velocity in tube, m/s
d_t - diameter of tube, m
μ - viscosity, kg/m·s
D - diffusivity, m^2/s
k - mass transfer coefficient, m/s
x_w - wt. fraction solute at membrane wall
x_r - wt. fraction solute in retentate
J_{solv} - volumetric flux of solvent, m^3/m^2·s or L/m^2·h

$$v_{inter} = v_{super} / \varepsilon_e$$

$$u_{s,i} = \frac{v_{inter}}{1 + \dfrac{(1-\varepsilon_e)}{\varepsilon_e}\varepsilon_p K_{d,i} + \dfrac{(1-\varepsilon_e)(1-\varepsilon_p)}{\varepsilon_e}\dfrac{\rho_s}{\rho_f}K'_{A,x}}$$

$$u_{th} = \frac{v_{inter}\rho_f C_{p,f}}{\left\{\left[1 + \left(\dfrac{1-\varepsilon_e}{\varepsilon_e}\right)\varepsilon_p\right]\rho_f C_{p,f} + \dfrac{(1-\varepsilon_e)(1-\varepsilon_p)}{\varepsilon_e} C_{p,s}\rho_s + \dfrac{W}{\varepsilon_e A_c} C_{p,w}\right\}}$$

$$\frac{c(T_2)}{c(T_1)} = \left[\frac{1}{u_s(T_1)} - \frac{1}{u_{th}}\right] \Big/ \left[\frac{1}{u_s(T_2)} - \frac{1}{u_{th}}\right]$$

where

v_{inter} – *interstitial velocity, m/s, cm/s, or cm/min*

v_{super} – *superficial velocity, m/s, cm/s, or cm/min*

$u_{s,i}$ – *average solute velocity of adsorbate, i, m/s, cm/s, or cm/min*

u_{th} – *thermal wave velocity, m/s, cm/s, or cm/min*

$K_{d,i}$ – *size exclusion parameter of adsorbate, i, dimensionless*

$K'_{A,x}$ – *linearized adsorption equilibrium constant, for system in wt. fraction,* $q_A = K'_{A,x} x_A$

W – *weight of column per lenght, kg/m*

A_c – *cross sectional area of column,* m^2

$C_{p,w}$ – *heat capacity of the wall,* $J/kg^\circ C$

$c(T_1)$ – *concentration at temperature* T_1, *wt. fraction*

$c(T_2)$ – *concentration at temperature* T_2, *wt. fraction*

$u_s(T_1)$ – *average solute velocity at temperature* T_1, *m/s, cm/s, or cm/min*

$u_s(T_2)$ – *average solute velocity at temperature* T_2, *m/s, cm/s, or cm/min*