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 $9 \times 10^{-11}$ 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$^o$C. Superficial velocity of the feed is 10 cm/min. The absorber is 2 m long and adsorption is taking place at 0$^o$C. The feed step is continued until breakthrough occurs. To regenerate, use counterflow of pure n-heptane at 80$^o$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

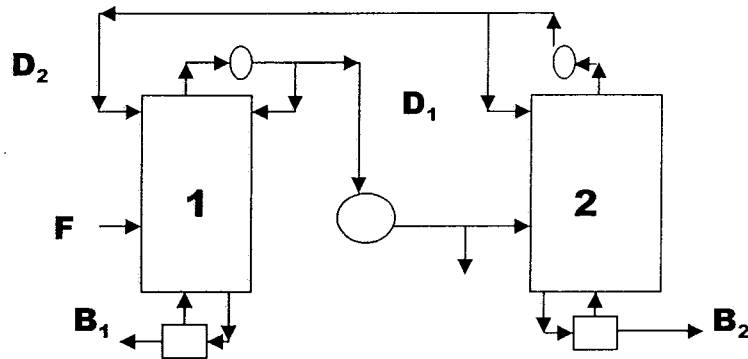| x | 0.4 | 0.6 | 0.8 | 0.9 | 0.95 | 1 |
|---|-----|-----|-----|-----|------|---|
| y | 0.4 | 0.52 | 0.65 | 0.75 | 0.85 | 1 |

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 $\rho_H$. 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:

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

$$\text{where}: \quad \eta = \left[ A_{EX} \cdot k_c \rho_{SH} \Big/ [L_m] \right]$$
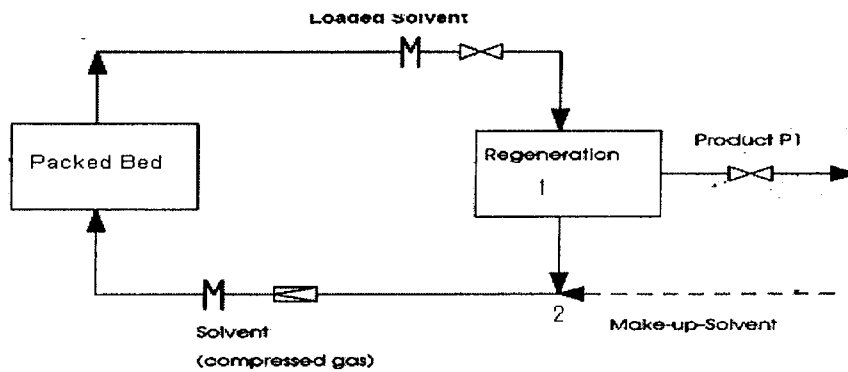
Loaded Solvent



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.

...5/-

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]

- ooo O ooo -
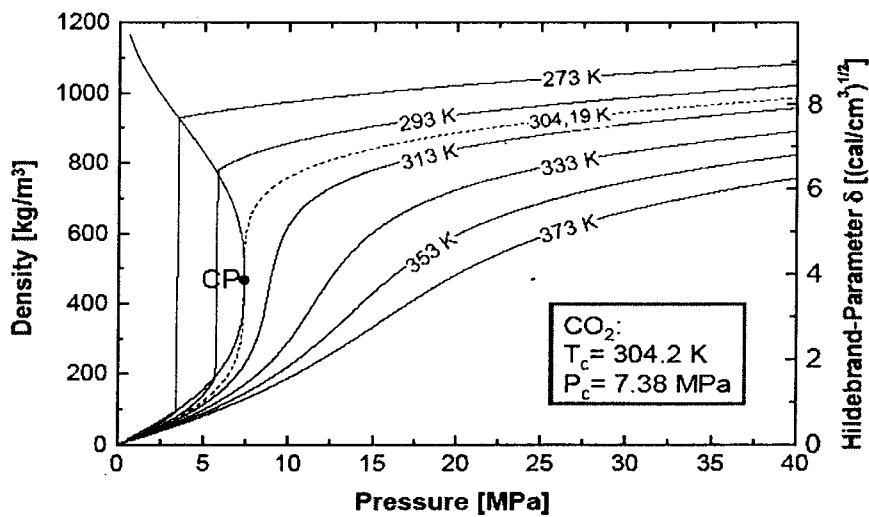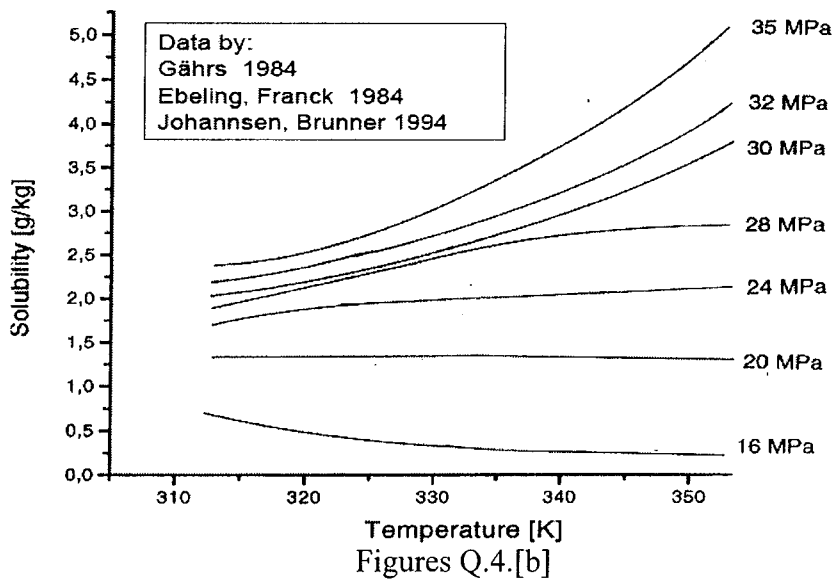
## Appendix

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,

$\rho$    - density, $\mathrm{kg/m^3}$

$u_b$    - bulk velocity in tube, m/s

$d_t$    - diameter of tube, m

$\mu$    - viscosity, kg/m·s

$D$    - diffusivity, $\mathrm{m^2/s}$

$k$    - 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, $\mathrm{m^3/m^2{\cdot}s}$ or $\mathrm{L/m^2{\cdot}h}$

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

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

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

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