Separation Processes, 4M3

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Note:

- You may bring in any printed materials to the midterm; any textbooks, any papers, *etc*.
- You may use any calculator during the midterm.
- You may answer the questions in any order on all pages of the answer booklet.
- *Time saving tip*: please use bullet points to answer, where appropriate, and **never repeat the question** back in your answer.
- Please note, parts of this exam will require you to apply concepts you have learned in your other core courses, such as 2D, 2F and 3E.
- This exam requires that you apply the material you have learned here in 4M to new, unfamiliar situations, which is the level of thinking required from 4th and 5th year students.
- Time management on this exam is important.
- **Total marks**: 100 marks, 15% of course grade.
- Total time: 2 hours. There are 4 pages on the exam, please ensure your copy is complete.

Question 1 [10 = 1 + 1 + 1 + 1 + 1 + 1 + 1 + 2 + 1]

Provide single word, or short sentence answers to the following (no explanation, no equations).

1. What would be the expected separation factor for an ultrafiltration membrane separating proteins from liquid?
2. What is the MWCO?
3. If a cyclone is not achieving the desired cut size, the main way in which it is adjusted during operation is by altering ....
4. In membrane separations: what is the MSA?
5. In a centrifuge: what is the ESA?
6. The difference between thickening and clarification is that .... (don't just copy from the notes please; use your own words).
7. The terminal settling velocity of a solid, spherical particle is affected by its (a) mass, (b) density or (c) both mass and density.
8. Disk-bowl centrifuges are preferred to tubular bowl centrifuges in this application .....(name one) for the following reason: .....
9. A good general reference on separation processes is ....

Solution

1. Very high, or infinite
2. Molecular weight cut off (the molecular weight which will be retained by the membrane at 90% concentration in the retentate)
3. The underflow velocity; then the entry velocity
4. The membrane itself
5. The energy to drive to the centrifuge, i.e. electricity
6. Clarification aims for a clear overflow, thickening aims for a highly concentrated solids in the underflow

7. Both mass (via D_p) and density
8. Beverage clarification for aseptic operation.
9. Perry's Handbook, or any of the other references that keep appearing in the course notes.

Question 2 [3]

Give 3 advantages of using a centrifuge over sedimentation.

Solution

- Allows for cleaning in place (CIP)
- Shorter time to separate, i.e. higher throughput
- Can be temperature controlled
- Aseptic operation: prevent contaminants and release of solvents
- Smaller footprint
- Achieve separations that gravity separation could not achieve otherwise
- No flocculation is required

Question 3 [24]

An asymmetric ultrafiltration membrane is used with the aim of separating dyes from a liquid stream and to achieve a more concentrated dye-water mixture. The feed waste stream arrives at a flow rate of 2.2 m^3.hour^{-1} with concentration of 1.2 kg.m^{-3}. The membrane's operating characteristic was calculated from various experiments:

$$J_v = 0.04 \ln \left(\frac{15}{C} \right)$$

where the bulk concentration C has units of kg.m^{-3} and flux is measured in m^3.hour^{-1}.m^{-2}.

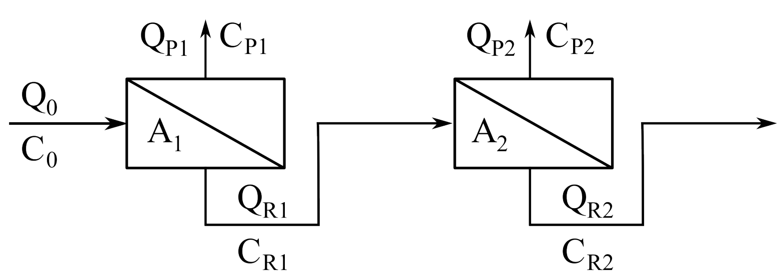
If two membrane modules, each of area 25 m^2, are simply placed in series, give reasonable estimates of:

1. the dye concentration from the first membrane module?
2. the permeate flow rate from the first membrane module?
3. the dye concentration from the final membrane module?
4. the permeate flow rate from the final membrane module?
5. Then explain whether the above answers seem reasonable.

Please show all calculations, assumptions and relevant details. (*Yes, this question is on new material; it is not hard; just think logically.*)

Solution

An illustration of the system is



recognizing that it is the retentate from the first module that is fed into the second membrane.

We make the assumption, common in ultrafiltration, that the permeate concentration leaving each module is approximately zero. The flux J_v refers to the flux *through* the membrane, in other words the flux of the permeate. The flux equation is in terms of the bulk concentration in the membrane, which is ultimately the concentration leaving, i.e. C_{R1} and C_{R2} for module 1 and 2 respectively.

We solve the membrane modules in series, because there are no recycle loops or coupling between the modules. We can cascade down from module to module. Mass balance on module 1:

$$Q_0 C_0 = Q_{R1} C_{R1} + Q_{P1} \cancelto{0}{C_{P1}} \tag{1}$$

Volume balance on module 1:

$$Q_0 = Q_{R1} + Q_{P1} \tag{2}$$

Solving for C_{R1}, the first part of the question, using (1) and substituting in (2):

$$C_{R1} = \frac{Q_0 C_0}{Q_{R1}} = \frac{Q_0 C_0}{Q_0 - Q_{P1}} \tag{3}$$

however we can use the flux equation to substitute for Q_{P1}:

$$J_v = \frac{Q_{P1}}{A_1} = 0.04 \ln\left(\frac{15}{C}\right)$$

Leading to

$$C_{R1} = \frac{Q_0 C_0}{Q_0 - 0.04 A_1 \ln\left(\frac{15}{C_{R1}}\right)}$$

simplifying and substituting in known values

$$2.2 C_{R1} - C_{R1}(0.04)(25)\ln\left(\frac{15}{C_{R1}}\right) - (2.2)(1.2) = 0 \tag{4}$$

where equation (4) is in the form $f(C_{R1}) = 0$. A lower bound for C_{R1} is 1.2, while an upper bound is 15 kg.m^{-3}. Using guess-and-check, or a calculator with root-finding capabilty:

$$\begin{aligned} f(C_{R1} = 3) &= -0.84 \\ f(C_{R1} = 8) &= 9.9 \qquad \text{obviously too high a guess} \\ f(C_{R1} = 4) &= 0.87 \qquad \text{still too high} \\ f(C_{R1} = 3.5) &= -0.03 \qquad \text{close enough} \\ \text{So } C_{R1} &= 3.5 \text{ kg.m}^{-3} \end{aligned}$$

From which we can solve for the remaining unknowns in module 1: $Q_{R1} = 0.75$ m^3.hour^{-1} and $Q_{P1} = 1.45$ m^3.hour^{-1}. A similar construction leads to: $C_{R2} = 9.4$ kg.m^{-3}, $Q_{R2} = 0.28$ m^3.hour^{-1} and $Q_{P2} = 0.47$ m^3.hour^{-1}.

The dye concentrations are being upgraded, as expected: from 1.2 to 3.5 kg.m^{-3} across module 1 and from 3.5 to 9.4 kg.m^{-3} in module 2. The corresponding flows leaving in the retentate decrease: from 2.2 m^3.hour^{-1} in the feed to 0.75 and then down to 0.28 m^3.hour^{-1} in the last module.

The permeate stream, essentially pure solvent is the balance of the volume: 1.45 + 0.47 = 1.92 m^3.hour^{-1} of pure liquid.

Grading for this question:

- 5 marks to set up the diagram with the correct flow connections, or it must be *very* clear from your calculations what the connections would have been
- 2 marks for the dye mass balance across module 1
- 2 marks for the volume balance across module 1
- 2 marks for correctly using the flux equation (correct flow term and concentration term)
- 2 marks for substituting equations into each other to attempt to solve for C_{R1}
- 2 marks for attempting to solve the non-linear equations by guess-check or calculator
- 2 marks for the solving for the correct values of C_{R1}, Q_{R1}, Q_{P1}
- 3 marks for setting up the equations for solving module 2
- 2 marks for solving for the actual values in module 2
- 2 marks for interpretation in the final part

Question 4 [8]

Perry's handbook gives an approximate correlation for the capital cost of a sedimentation vessel as cost [U$] $= 147d^{1.38}$, where diameter d ranges between 10 and 225 feet.

We stated at the start of the course that any separation unit is generally more expensive if it has a higher separation factor. Carefully explain whether the correlation from Perry's logically matches that statement for treating a given wastewater feed.

Solution

Separation factors, recall, are defined so that $S_{ij} = \dfrac{x_{i,1}/x_{j,1}}{x_{i,2}/x_{j,2}} = \dfrac{x_{\text{solid,under}}/x_{\text{liquid,under}}}{x_{\text{solid,over}}/x_{\text{liquid,over}}}$, where i and j are selected so that $S_{ij} \geq 1$.

A given feed implies the physical properties are fixed, which implies the terminal settling velocity is constant (note: a misconception that some people have is the TSV changes with an increased tank diameter; we explicitly learned this is not the case, else we wouldn't be able to scale up from lab tests to industrial scale). So if we are treating a given wastewater feed, by increasing the tank diameter we expect the following logic to hold:

- we will pay more for a larger diameter tank, (based on the correlation), so a
- larger tank diameter, implies
- larger tank area, implies
- slower *horizontal* velocity component for a given throughput Q, implying
- more time for solids to settle (i.e. a longer residence time), implying
- $x_{\text{solid,under}}$ increases and $x_{\text{solid,over}}$ decreases, implying
- S in the above equation gets larger and larger.

This sequence of logical events matches our expectation.

Question 5 [12]

1. Spherical particles of solid coffee extract of 400μm are falling in an air stream that is at 422K. The particles have a density of 1030 kg.m^{-3}; what is the terminal settling velocity?

The density of air at this temperature is 0.83 kg.m^{-3} and viscosity of air is approximately $\mu = \dfrac{C_1 T^{1.5}}{C_2 + T}$ where $C_1 = 1.46 \times 10^{-6}$ kg.m^{-1}.s^{-1}.K$^{-0.5}$ and $C_2 = 110.4$ K.

2. We have however a complete distribution of particle sizes, where the average particle size is 400 μm; should we design a separation unit based on the upper end (larger size) particles or based on the lower end (smaller size) particles?

Solution

1. Assume Stokes' law applies, then:

$$\begin{aligned} v_{\text{TSV}} &= \frac{(\rho_p - \rho_f)\, g D_p^2}{18\mu_f} \\ &= \frac{(1030 - 0.83)\,(9.81)(400 \times 10^{-6})^2}{(18)(2.38 \times 10^{-5})} \\ &= 3.78 \text{ m.s}^{-1} \end{aligned}$$

Checking the Reynolds number: $\text{Re} = \dfrac{\rho_f v_{\text{TSV}} D_p}{\mu_f} = \dfrac{(0.83)(3.78)(400 \times 10^{-6})}{2.38 \times 10^{-5}} = 52.6$, which exceeds 1. So we use the updated drag coefficient estimate $C_D = \frac{24}{52.6}\left(1 + 0.15 \times 52.6^{0.687}\right) = 1.5$.

Then the update estimate of the terminal velocity is:

$$\begin{aligned} v_{\text{TSV}} &= \sqrt{\frac{4\,(\rho_p - \rho_f)\, g D_p}{3 C_D \rho_f}} \\ &= \sqrt{\frac{4\,(1030 - 0.83)\,(9.81)(400 \times 10^{-6})}{(3)(1.5)(0.83)}} \\ &= 2.1 \text{ m.s}^{-1} \end{aligned}$$

which results in a Reynolds number of $\dfrac{(0.83)(2.1)(400 \times 10^{-6})}{2.38 \times 10^{-5}} = 29$.

One final iteration to fine-tune the estimate:

$C_D = \frac{24}{29}\left(1 + 0.15 \times 29^{0.687}\right) = 2.1$ and $v_{\text{TSV}} = \sqrt{\dfrac{4\,(1030 - 0.83)\,(9.81)(400 \times 10^{-6})}{(3)(2.1)(0.83)}} = 1.75$ m.s^{-1}, which has a Reynolds number of 24.4.

2. Separation units are designed based on the smaller size particles: these particles experience smaller inertial and/or gravitational forces, so they are the limiting factor in the design.

Question 6 [6]

Separation processes are of global importance in society. Please name three general separation applications that will be important to society's well-being during our life time.

Solution

- Carbon (and other green house gas) capture and sequestration
- Wastewater treatment for recycling
- Desalination
- *other reasonable answers that have global impact are acceptable*

Question 7 [15]

A continuous operating disk-bowl centrifuge has $\Sigma = 5256$ m^2 when operated at $\omega = 5000$ rpm for separating bacteria from a bioreactor broth. The bacteria's density is 1050 kg.m^{-3} with average particle size of 30 μm and a concentration of 20 kg.m^{-3}.

1. What is the interpretation of this Σ value?
2. Your company plans to increase the throughput of the existing centrifuge. A person has suggested that diluting the feed will allow for higher recovery of bacteria. So the plan is to install a large holding tank before the centrifuge and dilute the feed 5-fold, down to a concentration of 4 kg.m^{-3}. What might their thinking have been to suggest this? What will be the *quantitative result* on protein recovery of doing this?
3. The operator wants to repurpose this same centrifuge, but operated at 6500 rpm, for separating sand-like particles ($\rho = 1200$ kg.m^{-3}) suspended in a large batch of contaminated oil ($\rho = 800$ kg.m^{-3}, $\mu = 0.3$ N.s.m^{-2}). Provide some guidance as to the expected throughput the operator can achieve, if the mean particle diameter is 2μm.

Solution

1. Σ is the area that would be required to achieve the same separation in a sedimentation vessel.
2. The person might have been confused with membrane separations where are a dilute feed reduces the cake resistance; or cyclones were a dilute feed stream does affect recovery. All the matters is residence time of the particle in the centrifuge. Diluting the feed means it will just take longer to process the material for the same residence time in the centrifuge. This will just end up increasing your operating costs. However none of the equations for centrifuges are a function of the feed concentration, so this additional step would just be a waste of money.
3. The new Σ at a higher operating speed would be $\Sigma \times \dfrac{6500^2}{5000^2} = 8883$ m^2.

   The expected flow through the reactor to capture particles of 2μm is given by the cut-size throughput:

$$
\begin{aligned}
Q_{\text{cut}} &= 2v_{\text{TSV}}\Sigma \\
&= 2\left(\frac{(\rho_p - \rho_f)\, g D_{p,cut}^2}{18\mu_f}\right)\cdot\Sigma \\
&= 2\left(\frac{(1200-800)\,(9.81)(2\times10^{-6})^2}{18\mu_f}\right)\cdot\Sigma \\
&= 5.16\times10^{-5}\ \text{m}^3.\text{s}^{-1} \\
&\equiv 4.46\ \text{m}^3.\text{day}^{-1}
\end{aligned}
$$

Grading for this question:

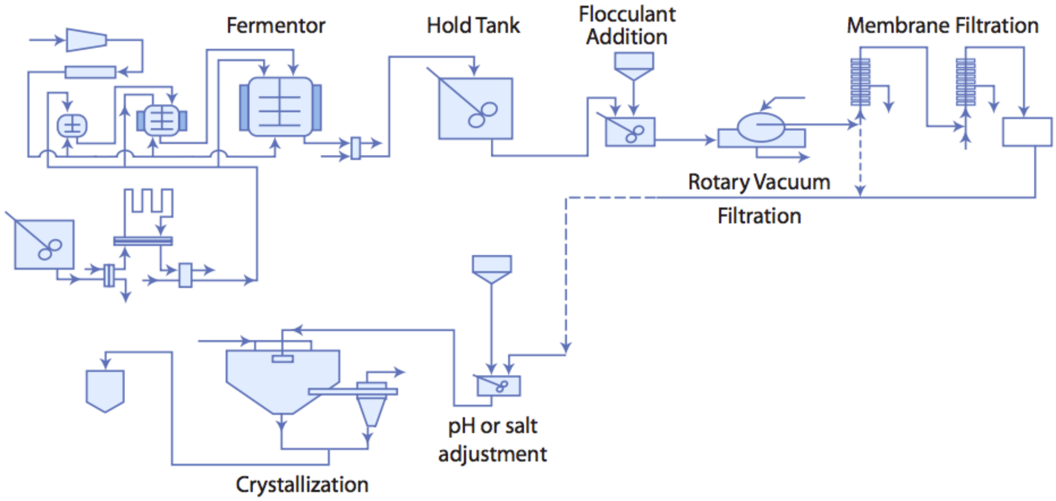
- Part 1 was 2 marks
- Part 2 was 5 marks: 2 for explaining why the person might have thought dilution was a good idea, but 3 marks to explain why it has no effect **on recovery**.
- Part 3 was 8 marks: 2 for the new Σ, and 6 for calculating the expected flow rate.

Question 8 [12]

This flowsheet is from the MIT OpenCourseware website, "Separation Processes for Biochemical Products", taught in 2005. It shows the downstream steps for recovery of alkaline protease, a biological enzyme.

You can view these interesting lecture notes at http://ocw.mit.edu/courses/chemical-engineering/10-445-separation-processes-for-biochemical-products-summer-2005/lecture-notes/lecture_10.pdf after the midterm (they tie in nicely with 4N4 also).

1. What is the general purpose of flocculation?



2. What is the purpose of flocculation in this flowsheet?
3. What is the purpose of the membrane step here?
4. Why are these membrane units in series?
5. Is it the retentate or permeate that is of interest?

Solution

1. Flocculation is a chemical treatment of the feed to cause particle agglomeration; these larger size particles settle faster, allowing the feed to be easily dewatered and concentrated
2. Flocculation here likely has the same purpose: to bring the biological suspension together, form flocs, and aid the subsequent filtration step. Larger particles have more open spaces, so there is a potential reduction of energy requirements in the filtration step. Large particles will also be more likely to be retained from passing through the subsequent membrane step's pores. It also leads to a reduced volume of material to treat in the membrane.
3. To increase the enzyme concentration and reduce the volume of solvent (broth) in the downstream crystallization step. It will also remove particles not trapped by flocculation.
4. The individual membrane module is not able to achieve the desired concentration; the second module accepts the retentate from the first module and increases its concentration further.
5. The enzymes will be in the retentate. The permeate may have some value, but this is not subsequently processed in the given flowsheet.

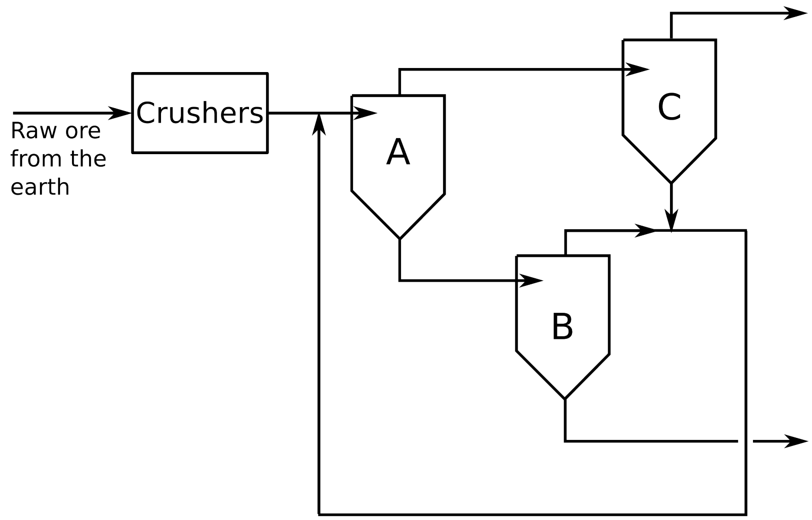
Question 9 [10]

The following flowsheet shows a typical arrangement of cyclones that you would see in the mining industry. Ore consisting of gangue (sand) plus valuable minerals enters as the feed to A from the upstream crushers. The cyclones all have the same geometry.

For example, in the gold mining industry, the aim is to achieve very small ore particles, e.g. $< 50\mu$m, so the gold crystals are exposed on the outer surface. Later these particles are placed into in a downstream solution of cyanide which dissolves (leaches out) the exposed gold from the ore [so note to yourself: keep gold rings and jewellery away from cyanide solutions].

Describe what might be the purpose of **each of the three** cyclones and why they are connected as they are. Also state where the two outlets from the flowsheet might go to next.

Give your answer in the context of the above example. Note: there is no need to describe the answer in terms of grade efficiency curves, simply give a qualitative explanation of what is happening.



Solution

Cyclone A is performing a rough separation of the recycled material and fresh feed. The finer overflow from A leaves to cyclone C where it is separated again to improve the cut. The fines leaving in the overflow of **cyclone C** will be the material going to cyanide leaching (cyanidation).

Cyclone B receives the heavier underflow from A. The heavier material from B will be the larger of the large particles that initially entered. These particles are too big and will likely be recycled back to the crushers, or some other unit operation to reduce their size. They will then likely be returned back to this flowsheet for another attempt at separating them based on size.

The overflow from B and underflow from C are midsize particles from the broad central area of the size distribution curves. These are particles that may be the right size for cyanidation or particles that are too large and require size reduction. Either way, they are recycled over and over through the cyclones until they leave in an appropriate stream.

The end.