

Extended Homework Project, Part 2
due December 4, 2008

The objective of the extended homework project is to demonstrate your ability to apply material and energy balances to a more complicated overall process. Quizzes have tested individual concepts; this assignment will assess combining many concepts together within an overall solution to a problem.

The project involves a modification to the process described in chapter 4 problem 38 of Felder Rousseau. Specifically,

- Each group will consider a different mass of solvent fed to the first mixing tank (step 1 in the problem description). *The per-group conditions will be listed on the class web page.*
- Each group will consider a different amount of extraction solvent fed to the third mixing tank (step 3 in the problem description).
- The extraction product stream that contains drug (D), ethanol (E), and solvent F (step 5 in the problem description) will be considered further in the project. Specifically, the drug D will need to undergo a reaction and purification in order to make the final product. (See below.)
- The mass of drug will be taken into account to enable the reaction to be analyzed properly. An average yield of (1 kg drug/1000 kg leaf) should be used to estimate the amount of drug that is available.

As a reminder, the following steps have been added to the process:

7. The extract product stream from the extractor (step 5) is fed to a reactor. There it mixes with a stream containing another reactant G, which is present at 10 mass% in solvent F. The G and drug are required in a 1:1 mass ratio. The drug is transferred completely to this extract stream. The reaction is described further in step 12, below. The reaction products D' and G' have the same masses as the reactants D and G.

8. The reactor products are cooled in a crystallizer, and most of the drug precipitates out. (The reaction changes its solubility.) The two products are the solid drug and the filtrate. Each group will consider a different filtering temperature and thus a different drug recovery efficiency.

9. The drug is dried by passing an air stream over it to remove residual solvents E and F and reaction product G'. The outlet air stream is passed through a flare to burn the E, F, and G', with the heat available for use elsewhere in the process.

The second part of the project involves **solving the material balances for each unit on the corrected flowsheet.** Reminder: this means that flow rate and mass (or mole) fraction variables must be calculated for all streams and compounds. This includes the amount of drug in each stream and the products of the combustion process in the flare. Incorporate both the 4.38 problem and the new steps into your flowsheet.

In addition to solving the material balances, answer the following questions:

- (a), (b), (c) See problem 4.38 statement in the text.
- (d) Clearly report the mass balance results for all individual units in the process.
- (e) Calculate the bubble point temperature at 1 atm pressure for the raffinate stream of the extractor. Assume that solvent F has volatility properties (i.e. Antoine equation parameters) and molecular weight equal to those of diethyl ether.
- (f) Describe a possible use for the 20% ethanol stream that emerges from the stripping column.
- (g) Describe a possible use for the filtrate that emerges from the product filter (step 8).
- (h) What fraction of the drug fed to the process (as part of the leaves) is recovered (i.e. obtained as an output) from the dryer? What steps could be taken to improve the recovery further?
- (i) What is the role of the ethanol in the extraction solvent? Why isn't pure water used instead? Hint: what property(ies) probably change as a result of having the ethanol present? What effect does that have on the material balances?
- (j) Speculate on the financial value of the drug molecule (\$/kg) compared to the ethanol and solvent F.

The following additional information is available (and required!) for solving the material balances.

10. The amount of drug transferred from the leaves depends the mass fraction of drug in the leaf and in the extracting solvent. The phase equilibrium lab of the company has recently quantified this relationship as

$$w_{\text{solvent}}/w_{\text{leaf}} = K_{\text{eq}}$$

with a value of $K_{\text{eq}} = 3.56$. In other words, at equilibrium there is enough drug extracted from the leaf such that the mass fraction of drug in the ethanol/water mixture (which is used as the extraction solvent) is 3.56 times larger than the mass fraction of drug in the leaf. Since the mass fraction of drug is so small in both systems (0.001 in the original leaf), the statement about the mass of drug having a negligible impact on the total mass and volume of spent leaf and filtrate continues to be true. In other words, the mass fraction of dissolved drug can be approximated as (mass of drug / mass of solvent); for the solid phase, the mass fraction is approximately (mass of drug / mass of leaves). Technically these are called "mass ratios". Hint: the mass fractions inside a unit can be calculated from the amounts flowing in, i.e. they reach this mass fraction before the streams flow out.

In addition, the lab has reported that the rate of attaining phase equilibrium is slow unless there is a lot of agitation. Fortunately the mixing tanks have good mixers, and experiments have shown that equilibrium is reached there. The holding tank doesn't have a mixer, though, so further transfer of drug between the liquid phase and the remaining suspended leaves is negligible there.

11. The phase equilibrium lab has also determined a three-phase diagram for the ethanol (E) / water (W) / solvent F system, under the temperature and pressure conditions that are found in the extraction unit. The resulting triangle diagram was distributed in class and is available on the class web page.

12. The reaction between the drug and molecule G is an isomerization that leaves the molecular weights and masses of drug D and reactant G unchanged. The products D' and G' can be distinguished from D and G by infrared spectroscopy but not by material balances. The reaction goes to completion if D and G are fed in a 1:1 mass ratio.

13. In the crystallizer, the F/E/G mixture is cooled down to a temperature between 0 and 10°C. (Each group will have a different temperature.) Between these temperatures, the phase equilibrium lab reports that the equilibrium drug solubility in the F/E/G mixture can be described by the following equation:

$$\log w_{\text{drug}} = -\frac{7726}{T} + 21.30$$

The temperature is in degrees Kelvin. Any drug above the mass fraction w_{drug} precipitates into the (desired) solid phase; the rest remains dissolved in the filtrate. Note that this equation does not apply at higher temperatures.

5 mass% of the mass of the filter cake is solvent (E, F, and G) that is held up in the solid drug. (Think of damp coffee grounds.) The solvent components E, F, and G are at the same relative concentrations as in the filtrate. The drug dissolved in the solvent held up in the solid phase can be ignored.

14. The amount of air used to dry the filter cake is controlled so there is six times as much air present compared to the amount required for the combustion reaction that occurs in the flare (i.e. 500% excess oxygen). This large excess is used to ensure that the vapor phase concentrations of E, F, and G do not become too large. Assume that F has properties identical to those of diethyl ether, as above. Assume that G has a molecular weight and properties equivalent to those of normal dodecane. (It isn't normal dodecane, but you are told that those properties are similar enough for these calculations.) Assume that the dryer removes all solvent and no drug from the solid phase.