Batch Distillation

Distillation is a common method of separating and purifying liquids. Its operation is based on differences in boiling points between components of a liquid mixture. The lighter components move up the column and the heavier ones move down. Batch distillation is utilized when relatively small amount of product is required so that performing continuous distillation becomes economically unfeasible. In this experiment, you will use batch distillation to separate ethanol and isopropanol. In addition to ethanol and isopropanol, the feed contains a small amount of methanol.

System Overview

The batch distillation experiment is performed using the East Column. This column contains 12 stages (bubble cap trays). Other specifications for the column are as follows:

- Distance between plates: 12 inches
- Weir height 1 inch
- Hold-up on plates 2 lb/plate
- Hold-up in condenser 4.5 lb

Other key components of the system are a steam-heated reboiler, a water-cooled condenser, a bottoms tank, and a distillate product tank. Schematic of the system is shown in Figure 1. The column spans the first two floors of the lab. The other system components are located either on first or second floor:

1. First floor: reboiler, reboiler tap, and reboiler mixing pump.
2. Second floor: condenser, standpipe where the liquid is held up after being condensed, distillate and reflux lines, and a sample tap on the reflux line.

There are supply lines for steam (heating), water (cooling), and air (operating pneumatic valves) on both floors. The distillation column is controlled by a computer system located in the control room on the 2nd floor of the lab. Compositions of the feed and the products will be determined using a Gas Chromatograph (GC) which is also located in the control room.

The starting reboiler composition will be approximately 50% ethanol, 50% isopropanol, up to 4% methanol, and less than 1% other components including methyl isobutyl ketone, ethyl acetate, and naphtha solvent. A batch will be prepared in the steam-heated reboiler prior to the start of the lab period. You will start the column under total reflux conditions. After the system reaches a steady-state, it will be switched to the batch mode by drawing off part of the distillate product.
Figure 1. Diagram of the batch distillation system as it appears on the screen of the computer in the control room.

**Degrees of Freedom**

The main independent control variables available are

- Reflux ratio
- Flow rate and pressure of steam supplied to the reboiler
- Flow rate of water supplied to the condenser

You should carefully choose operating conditions before running an experiment. Remember that it takes a long time to perform a single experimental run, so choosing inadequate parameters will result in wasting a lot of time. One of the main goals of your preliminary report should be to choose sensible experimental conditions for the 2nd and 3rd days of experiments. The main goal of the 1st day of experiments is to collect the data necessary to make this choice.
Objectives

1. Determine the overall efficiency of the system.
2. Determine local efficiency of individual plates.
3. Investigate effect of reflux ratio on the product composition.
4. Investigate effect of distillate flow rate composition (controlled by steam flow rate and pressure) on the product.
5. Optional: investigate effect of changing flow rate of the cooling water.
6. Determine time-dependence of the following quantities at various distillate flow rates and reflux ratios:
   a. Distillate product composition
   b. Bottoms product composition
   c. Heat transfer rate from the condenser
   d. Heat transfer rate to the reboiler
   e. Stage by stage temperature profile (optional)
   f. Stage by stage composition profile (optional)
   g. Stage by stage flow profile (optional)

Schedule

Week 1
- Run the column at total reflux and different flow rates of fluids inside the column
- Determine the overall and local efficiencies. For this, you should measure composition of the top and bottom products, as well as of samples taken from individual trays.
- Calibrate the GC. It is recommended to work on calibration while the distillation column is warming up.
- Measure amount of liquid and its composition in the bottoms tank after the system has reached a steady state. This will give you information regarding initial conditions for the batch operation. You will need this information to develop an experimental plan in the preliminary report.
- Learn how to draw off distillate product, even if you do not perform this task the first week.

Weeks 2 and 3
- Implement the plan proposed in your preliminary report:
  Run the distillation column at different reflux ratios and steam flow rates.