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 more volatile components move up the column and the less volatile 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 isopropanol and 2-butanol.

System Overview

Schematics of the Batch Distillation system is shown in Figure 1. The distillation is performed using the East Column (EC) containing 12 bubble cap trays and spanning the first two floors of the lab. Other specifications for the column are:

- 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:

- Steam-heated reboiler (located on the 1st floor)
- Distillate tank T4 (located on the 1st floor)
- Water-cooled condenser and standpipe, in which distillate is held up after being condensed (located on the 2nd floor)

![Diagram of the batch distillation. Product lines are shown in black (liquid) and gray (vapor). Steam and steam condensate lines are shown in red and water lines are shown in blue.](image)
The distillation column is controlled by a distributed control system DeltaV with operator stations located in the control room on the 2\textsuperscript{nd} floor of the lab. Compositions of the feed and the products are determined using a Gas Chromatograph (GC) which is also located in the control room.

**Degrees of Freedom**

The main independent control variables available are

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

You should carefully choose operating conditions before running an experiment. Remember that it takes a long time (1 hour or more) to perform a single experimental run, so choosing inadequate parameters will result in wasting a lot of time. One of the main goals of the 1\textsuperscript{st} day of experiments is to collect the data necessary to choose sensible experimental conditions for the 2\textsuperscript{nd} day of experiments.

**Objectives**

1. Determine the overall efficiency of the column.
2. Determine local efficiency of individual trays.
3. Investigate effect of reflux ratio on the product composition.
4. Investigate effect of distillate flow rate (controlled by flow rate of steam to the reboiler) on the product composition.
5. Optional: investigate effect of changing flow rates 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 and composition profiles (optional)

**Schedule**

**Week 1**

- Run the column at total reflux and 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.
- Investigate dependence of the reflux flow rate on the steam flow rate.
- Develop an experimental plan for Week 2.

**Week 2**

- Perform distillation at finite reflux using reflux ratios and steam flow rates determined from your analysis of the data from Week 1.