

CHME 401 CHEMICAL ENGINEERING LABORATORY II
EXPERIMENT 401-7
CONTINUOUS DISTILLATION

OBJECTIVE

The objective of the experiment is to understand and apply basic concepts in distillation of binary mixtures. Continuous distillation of ethanol-water mixture will be investigated. Efficiency of the column will be estimated and effect of reflux ratio on separation will be observed

PRELIMINARY WORK

1. Study basic fundamentals of phase equilibrium for binary mixtures (Liquid Vapor).
2. Obtain T,x,y data for ethanol in the ethanol-water system and plot boiling point and equilibrium diagram for ethanol.
3. Study distillation column design for binary mixtures .
4. Visit the lab. in advance to experiment and familiarize yourself with the experimental set-up with the consent of the teaching assistant.

DESCRIPTION OF THE EXPERIMENTAL SET-UP

Distillation unit is used to demonstrate the effect of different operation modes on distillation. Batch and continuous distillation at different reflux ratios can be studied. The pressure in the column can be reduced by connecting the unit to a vacuum pump. Experiments done with this unit provides opportunity to study energy balance over condenser and re-boiler sections and comparison between theoretical and real efficiency of the distillation column.

The equipment mainly consists of borosilicate re-boiler tank, heater, condenser, 3 tube-tube heat exchangers, borosilicate distillate collection tank and bottom product collection tank. Reflux ratio is selected by using a solenoid valve and a timer. The column is made up of 7 bubble-cap plates. Equipment is given in Figure 1.



Figure 1. Continuous Liquid- Liquid Distillation Set-up

EXPERIMENTATION

Temperature of the system will be recorded continuously through out the column, re-boiler and condenser by the help of thermocouples placed in the column. Temperature measurements will be used to understand whether the system is at steady state and to estimate equilibrium concentrations. Besides top and bottom product concentrations will also be measured by using an Abbe Refractometer.

Procedure

- 1) Obtain a calibration curve for Ethanol content in water by using Abbe Refractometer (prepare solns.: 0, 10, 30, 50, 80 and 100 mole % ethanol).
- 2) Shut-off valves V2, V3,V4, V6, V9, V15, V16 and open valves V1, V5, V7, V8, V10, V11, V12, V13, V14.
- 3) Prepare 10 L of ethanol- water solution (20%).

- 4) Fill both feed tank and re-boiler (until it overflows to D3) with the ethanol solution.
- 5) Open cooling water and adjust flow rate to 150l/h by using V6.
- 6) Switch J1 on to heat the re-boiler and adjust it to 1600 W wait until mixture reaches to boiling point and decrease the power of the heater to 1000 W.
- 7) Operate at total reflux for 5 minutes (until stationary temperature values are achieved).
- 8) Start Pump G1 (start to pump feed solution) by pushing start button on its keyboard and set it to 2L/h (pump can achieve flow rate of 7.1 L/h when it is set to 100%).
- 9) Set timers on the main panel to 10 sec stop and 1 sec start then start reflux timer to obtain a reflux ratio of 10:1.
- 10) Wait until steady state conditions are obtained it may take a while.
- 11) After steady state conditions are obtained drain the distillate and bottom product (D2 and D3) wait for another 5 minutes to collect distillate samples in tank D2 and D3 then take samples (measure volumes) to measure their ethanol contents using Abbe Refractometer.
- 12) Repeat step 11 for 3 times to obtain convenient results.
- 13) Turn reflux timer off set timers to 5 sec stop and 1 sec start and repeat steps 10-12.

Analysis

- 1) By using the temperatures of re-boiler and condensate determine ethanol concentrations in bottom and top product and compare with your results (use boiling point diagrams).
- 2) Make material and energy balance around the column.
- 3) Determine the theoretical stage number for the runs conducted and calculate the column efficiency.
- 4) Discuss the effect of the reflux ratio in distillation columns and how the changing the reflux ratio affected your results.