Distillation Experiment--Flooding

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[NOTE: The report has been minimally edited for clarity and consistency.]

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Abstract

Experimenters examined a binary distillation column that was used to separate ethanol from water. The distillation column was operated at total reflux for five different steam flow rates, but data from only four rates were analyzed. The goal of this experiment was to determine the point at which flooding occurred by observing changes in operating conditions at the different steam flow rates. Operating conditions that were observed included tray temperatures, pressure drop across the column, froth height as observed through the view-port, reboiler liquid level, and liquid compositions at both the top and the bottom of the tray. The values of the steam flow rates at 50%, 75%, 100%, and 125% of flooding were determined to be 0.44 kg/min, 0.65 kg/min, 0.87 kg/min, and 1.09 kg/min, respectively. The predicted flood point of the column was determined to be 0.87 kg/min, while the experimentally determined flood point of the column was determined to be 0.82 kg/min. This discrepancy showed a 6.1% difference between the predicted and experimentally determined flood point of the column. These two values may differ due to the fact that the energy balance equation around the reboiler did not take into account the heat lost to the surroundings. Another possible cause for the difference between these two values could be tray fouling.

> Notice how the authors move from the general to the specific. They use 4 sentences for the procedure, 3 sentences for the results, and 2 sentences to explain why the results occurred.

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Distillation Experiment--Flooding

Introduction

The purpose of the experiment was to determine the point at which flooding occurred in a binary distillation column used to separate ethanol from water. The column, which was operated at atmospheric pressure, contained a feed stream that consisted of 30 wt% ethanol at 70°C. The overhead product had to be at least 80 wt% ethanol, and the bottoms product could not be more than 5 wt% ethanol. In this experiment, the distillation column was operated at total reflux at four steam rates. The flood point was determined by observing changes in operating conditions at the different steam rates.

This introduction concisely explains the purpose and parameters of the experiment.

Methods

Fig. 1 shows a P&ID of the column that was used throughout this experiment:



Figure 1. Process and Instrumentation Diagram of the binary distillation column.

The distillation column was operated at five steam flow rate values. The point of flooding was identified by observing changes in operating conditions at each steam

rate. Variables such as tray temperatures, pressure drop across the column, froth height observed through the view-port, reboiler liquid level, and liquid compositions at the top and bottom of the tray were used to determine the onset of flooding. The experiment procedures followed the Lab Handout (Simpson, 2005). Before operating the column, the exhaust fan was checked to ensure that it was on. The main cooling water supply valve was then turned on. The reboiler level was verified to make sure that it was 15 cm or greater. The nitrogen supply cylinder was turned on and the outlet gauge was set to approximately 5 psig. The block valves on either side of the steam control valve were opened. The steam bypass valve was opened halfway. The column was then allowed to warm up. The block valves around the reflux control valve were opened. The level in the accumulator was verified as being constant at the set point before any data were recorded. The pressure on the reflux pump was adjusted to 50 psig. The block and bypass valves around the bottom product valve were opened and the bottoms pump was turned on. After steady state was reached, the temperature was verified to be in the range of 43°C to 52°C and the bottoms pump pressure was verified to be 50 psig. The column was operated at five values of steam flow. The five values of steam flow were chosen as a percentage of the predicted flooding vapor rate of the column. The operating data at each steam rate was collected from the overview display when steady state was reached. However, data at the steam flow rate of 0.75 kg/min were dropped from certain correlations since complete data (for figures 4 - 7) could not be collected at that value; the remaining four values were used in all the analysis.

The experimenters followed a number of safety precautions. Safety glasses and hard hats were worn when working with the distillation column. Caution was taken when climbing the column stairs. Experimenters were careful not to back into the stairs when working under the column because the stairs have sharp edges. Experimenters were also careful to avoid any hot surfaces when working with the column. Ethanol is extremely flammable and the appropriate precautions were taken when handling the chemical. The cooling water was verified to be flowing before the steam valve was opened. If the accumulator level had reached above 20 cm, the reflux pump would have been checked to ensure that it was on. If the accumulator level had dropped below 6 cm, the reflux pump would have been turned off. The bottoms pump pressure should have been reduced if TI-072 was above 52°C. Because information was being shared between two different teams, all members made sure that both teams were aware of the distillation column operating conditions.

When a distillation column is run at total reflux, the vapor rate is equal to the liquid rate at any point in the column. Under steady state conditions, the experimental internal reflux rate, or the liquid rate at the top of the column, is calculated from the chosen steam rate. The internal vapor rate can be calculated using the following methods (Simpson, 2005). The first method takes into account the external reflux for subcooling. The following relationship describes this aspect:

$$\mathbf{R}_{i} = \mathbf{R}_{e} \left[1 + (\mathbf{T}_{i} - \mathbf{T}_{e}) \mathbf{C}_{p, liq} / \mathbf{H}_{vap, liq} \right]$$

where,

$$\begin{split} R_i &= \text{internal reflux rate (gmol/min)} \\ R_e &= \text{measured external reflux rate (gmol/min)} \\ T_i &= \text{temperature of overhead vapor (°C)} \\ T_e &= \text{temperature of external reflux (°C)} \\ C_{p,liq} &= \text{heat capacity of reflux (cal/gmol°C)} \\ H_{vap,liq} &= \text{heat of vaporization (cal/gmol)} \end{split}$$

An energy balance around the condenser may also yield the internal vapor rate according to the following relationship:

$$R_i = (m_{cw} C_{P,cw} \Delta T_{cw}) / H_{vap,cond}$$

where,

$$\begin{split} R_i &= \text{internal reflux rate (gmol/min)} \\ m_{cw} &= \text{mass flow of the cooling water (g/min)} \\ C_{P,cw} &= \text{heat capacity of cooling water (cal/g^oC)} \\ \Delta T_{cw} &= \text{change in cooling water temperature (}^oC) \\ H_{vap,cond} &= \text{heat of vaporization of the liquid condensate} \end{split}$$

(cal/gmol)

An energy balance around the reboiler yields the following relationship:

 $V_{reb} = (1/_{Hvap,reb}) (m_{steam}H_{vap,steam} - q_{btms}\rho_{btms}C_{p,btms}\Delta T_{btms})$

where,

 V_{reb} = process vapor flow from reboiler (gmol/min) $H_{vap,reb}$ = heat of vaporization of the liquid in reboiler

(cal/gmol)

$$\begin{split} m_{steam} &= mass \ flow \ of \ the \ steam \ (g/min) \\ H_{vap,steam} &= heat \ of \ vaporization \ of \ the \ steam \ (cal/g) \\ q_{btms} &= volumetric \ flow \ in \ the \ sample \ loop \ (liters/min) \\ \rho_{btms} &= density \ of \ liquid \ in \ the \ sample \ loop \ (g/liter) \\ C_{p,btms} &= heat \ capacity \ of \ bottoms \ liquid \ (cal/g^{o}C) \\ \Delta T_{btms} &= change \ in \ temperature \ through \ bottoms \ loop \ (^{o}C) \end{split}$$

High internal vapor rates can result in the liquid becoming unable to flow down the column. At these conditions, flooding can occur. Characteristics of flooding include very large pressure drops and temperature gradients across the flooded trays. As a result, the column becomes inefficient and difficult to control (Simpson, 2005). Empirical flooding correlations are given in McCabe, et al., 2001, and fluid physical properties are obtained from Green, 1984.

Results

The predicted flood point of the column was determined to be 0.87 kg/min (See Appendix); however, the experimentally determined flood point of the column was found to be 0.82 kg/min (Table 1). This discrepancy shows a 6.1% difference between the predicted and experimentally determined flood point of the column.

In the experiment, the values of the steam flow rates were calculated using an energy balance around the reboiler. The values of the steam flow rates at 50%, 75%, 100%, and 125% were determined to be 0.44 kg/min, 0.65 kg/min, 0.87 kg/min, and 1.09 kg/min, respectively. A plot of column differential pressure against the column steam flow rate was used to determine the point at which flooding occurred in the distillation column.

In Fig. 2, the values of the column differential pressure start to deviate from linearity at a steam flow rate of 0.87 kg/min. The column differential pressure increases drastically at the onset of flooding. Therefore, from Fig. 2, flooding was determined to occur between 0.87 kg/min and 1.09 kg/min.



Figure 2: Column Differential Pressure vs. Steam Flow Rate

In order to further define the flood point of the column, plots of reboiler level versus steam flow rate, condenser (ethanol wt%) versus steam flow rate, reboiler (ethanol wt%) versus steam flow rate, and tray temperature versus steam flow rate were used.

In Fig. 3, the values of the reboiler level start to drop significantly at a steam flow rate of 0.87 kg/min. The reboiler level should decrease at the onset of flooding

The authors begin with the key results, then elaborate on the detailed findings. (The Appendix is not included with this sample.)

Introduce the Figures and Tables in the paragraphs above the illustration. because more of the liquid from the reboiler is being vaporized. This increased vapor flow rate is the cause of flooding because the liquid cannot overcome the vapor flow rate coming up the distillation column.



Figure 3: Reboiler Level vs. Steam Rate

The liquid that contains the least concentrated amount of ethanol usually remains at the bottom of the distillation column. This happens because ethanol, which has a lower boiling point than water, vaporizes more readily at lower temperatures. When flooding occurs, the vapor flow rate is so high that all the liquid is pushed towards the top of the column. Therefore, the condenser should contain more water, resulting in a lower overall ethanol concentration in the condenser, as indicated by Fig. 4.



Figure 4: Condenser (Ethanol Wt%) vs. Steam Rate

The increased steam flow rate adds such a large amount of heat to the reboiler that more ethanol is boiled off than usual. As indicated by Fig. 5, the increased amount of heat vaporized the ethanol more readily than the water, resulting in a lower amount of ethanol in the reboiler.



Figure 5: Reboiler (Ethanol Wt%) vs. Steam Flow Rate

As the steam flow rate is increased, the temperature of the trays also increased. This trend is indicated by both Fig. 6 and Fig. 7.



Figure 6: Tray Temperature vs. Steam Flow Rate for Trays 1 Through 5



Figure 7: Tray Temperature vs. Steam Flow Rate for Trays 6 through 10 and the Reboiler

Table 1 gives values taken from experimental data from Figs. 2 through 6 to arrive at an estimate of the average steam flow rate at the onset of flooding.

| Operating variable | Value at onset of Flooding | Value after Flooding | Steam Flow Rate at Onset of Flooding (kg/min): |
|------------------------------------|-------------------------------|-------------------------|---|
| Column differential pressure (kPa) | | | |
| (Fig. 2) : | 2.36 | 3.38 | 0.87 |
| Reboiler level (cm H2O) (Fig. 3): | 26.46 | 26.29 | 0.87 |
| Condenser ethanol wt% (Fig. 4): | 78.20 | 66.30 | 0.87 |
| Reboiler ethanol wt% (Fig. 5): | 21.30 | 20.40 | 0.81 |
| Tray 5 temperature (°C) (Fig. 6): | 80.74 | 81.65 | 0.75 |
| | Average Steam Flow Rate at | | |
| | | Onset of Flooding | 0.82 |

| Table 1: | Average | Steam | Flow | Rate at | Onset of | of Floodiı | ng |
|----------|---------|-------|------|---------|----------|------------|----------|
| | 0 | | | | | | <u> </u> |

The difference between the predicted and experimentally determined flood point of the column could have two causes. One cause could be a limitation in the procedure. An energy balance around the reboiler was used to determine the predicted flood point of the column. This equation, however, did not account for the heat lost to the surroundings. If the heat loss had been accounted for, the predicted vapor flow from the column would not have been as high because heat would have been lost to the surroundings. The transfer of heat from the reboiler to the bottoms is not perfectly efficient. The heat lost to the surroundings would result in a lower predicted process vapor flow from the reboiler.

Another cause for the difference between the predicted and the experimentally determined flood point could have been contamination of the apparatus, specifically tray fouling. When tray fouling occurs, a solid build-up forms on the trays and covers some of the holes, lowering the number of holes for liquid/vapor transfer. This blockage would result in the column being more likely to flood at lower vapor flow rates and, therefore, the column would require less heat from steam to cause flooding. Thus, tray fouling could account for the discrepancy between the predicted flooding steam flow rate and the experimental flooding steam flow rate, as observed during the experiment.

Conclusions and Recommendations

The experiment results showed a 6.1% difference between the predicted flood point of the column (0.87 kg/min), and the experimentally determined flood point of the column (0.82 kg/min). Experimenters traced the difference to two potential causes: a limitation in the procedure or contamination of the apparatus. The energy balance equation around the reboiler did not take into account the heat lost to the surroundings. The other potential cause, tray fouling, could have compromised the liquid/vapor transfer, causing the column to flood at lower vapor flow rates and, ultimately, with less heat from steam.

Why did the Results turn out as they did? The authors clearly set out two possible causes.

The first paragraph concisely summarizes the results and possible causes. The final paragraph makes recommendations for future experimenters. Experimenters recommend that changes in operating conditions at each steam rate be monitored carefully for observance of flooding. A plot of column differential pressure against the column steam flow rate provides a good indication of the point of the onset of flooding. The computer program running the distillation column should be monitored carefully as well. Should one of the pumps turn off, it should be restarted for proper operation of the system.

References

Green, D.W., ed. Perry's Chemical Engineering Handbook. Sixth Edition. McGraw-Hill Book Company, New York. 1984.

McCabe, W.L., et al. Unit Operations of Chemical Engineering. 6th Edition. New York: McGraw-Hill, 2001.

Simpson, Dale. "Distillation Experiment - Flooding," Lab Handout ChE264, The University of Texas at Austin, Summer 2005.