

Felleslab ST-2  
Distillation: Oldershaw perforated plate  
**Group B-16**

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## 1 Introduction

The purpose of this experiment has been to acquire knowledge of the different principles of distillation by examining an Oldershaw perforated plate distillation column. Column efficiency, theoretical trays and vapour velocity at steady states for different reboiler power outputs have been determined, and the properties of the weeping and the flooding point have also been explored.

## 2 Theory

The theory in this experiment is gathered from [?] and [?].

### 2.1 Weeping point

A weeping point is a condition in the distillation column which occur when the vapour velocity is too low. The vapour pressure is not sufficient to hold up the liquid in a tray. Instead the liquid will flow through the perforated holes in the tray and will "weep" out underneath. The vapour loses contact with the liquid, and this causes both the pressure and the efficiency to drop.

### 2.2 Flooding point

The capacity of a counter current (liquid flows downwards and vapour flows upwards) separation is called a flooding point. At the flooding point liquid will accumulate in the column as a result of the high vapour velocity which causes the pressure from the vapour to hold back the liquid from the downcomers. This increases the drop in pressure, resulting in a lower column efficiency.

### 2.3 Column efficiency

The efficiency of a distillation column is given by the ratio of the total number of ideal equilibrium stages and the total number of actual trays in the column. A high efficiency means that for each stage, the upcoming vapour is in equilibrium with the liquid which flows downwards. The equation for this is given in (2.1),

$$\eta = \frac{N_t - 1}{N_a} \quad (2.1)$$

where the  $-1$  indicates that the reboiler is regarded as one tray.

## 2.4 Reflux

Reflux is the portion of the vapour that condenses and is returned to the distillation column. The reflux ratio is given by (2.2),

$$R = L_n/D \quad (2.2)$$

where  $R$  is the reflux ratio,  $L_n$  is the condensed liquid flow back into the column and  $D$  is the distillate flow out of the column.

## 2.5 McCabe-Thiele method

The McCabe Thiele method is a graphical method for calculating the total number of theoretical trays in a given binary separation process. The method uses an assumption of constant molar overflow, which means that the vapour flow rate and molar liquid flow rate both are constant for all stages in a section of the column. Because of this the operating line is considered to be a straight line. Using mass balances and vapour-liquid equilibrium data (VLE) (section A.3-23 in Geankoplis [?]) for both components a McCabe-Thiele diagram (Figure 1) can be generated and the number of theoretical stages can be determined graphically. In a McCabe-Thiele diagram, the x-axis shows the mole fraction of the light component (the component with the lowest boiling temperature) in liquid phase, the y-axis shows the mole fraction of the light component in the gas phase. The VLE-data gives an equilibrium line which should lie above the operating lines which is generated from mass-balances and reflux ratios.

There are two operating lines. One for the enriching section (section of the column where the mole fraction of light component is higher than the feed) and one for the stripping section (section of the column where mole fraction of light component is lower than the feed). The enriching operating line is solved in equation (2.3),

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{x_D}{R+1} \quad (2.3)$$

where  $R$  is the reflux ratio,  $x_n$  is the mole fraction of light component in liquid phase at tray  $n$ ,  $x_D$  is the mole fraction of light component in the distillate. The stripping operating line is solved in equation (2.4),

$$y_{m+1} = \frac{L_m}{V_{m+1}}x_m - \frac{W_{x_W}}{V_{m+1}} \quad (2.4)$$

where  $L_m$  is the liquid flow from tray  $m$ ,  $V_{m+1}$  is vapour flow from tray  $m+1$ ,  $x_m$  is the mole fraction of light component in the liquid phase at tray  $m$ ,  $W$  is the flow out of the column at the bottom and  $x_W$  is the mole fraction of light component in the out flow.

The feed condition determine the relationship between the the vapour in the stripping section  $V_m$  and vapour in the enriching section  $V_n$ , as well as the liquid phases,  $L_m$  and  $L_n$ . If the feed is dual phase, the vapour will add to  $V_m$  to give  $V_n$ . This condition is defined by  $q$  (equation 11.4-12 in Geankoplis [?])

$$q = \frac{\text{heat needed to vaporize 1 mol of feed at entring condiction}}{\text{molar latent heat of vaporization of feed}}$$

In the McCabe-Thiele diagram,  $q$  is used to create a  $q$ -line with slope  $\frac{q}{q-1}$ , going through the point  $(x_f, y_f)$ , where  $x_f$  is the molar fraction of light component in liquid phase in feed.  $q$  gives properties for the  $q$ -line given in table 1. The number of theoretical stages is then determined using the following

Table 1: The table shows the properties of the  $q$ -line for different values of  $q$

$q$ value	$q$ -line	Properties of feed
$q = 1$	vertical	Feed enters at boiling point
$q = 0$	horizontal	Feed enters at dew point
$q > 1$	Positive slope	Feed is cold liquid
$q < 0$	Negative slope	Feed is superheated vapour

steps:

1. Start at top tray where  $x = x_D$ .
2. Draw a horizontal line until you hit the equilibrium line. This point is tray 1.
3. Draw a vertical line until you hit the operating line.
4. Count and repeat step 2 until you hit  $x = x_B$ .

By using the MATLAB-scripts in Appendix ?? the following McCabe Thiele diagram was generated.

*Figure 1: The figure shows the McCabe-Thiele diagram for ethanol/water mixture, plotted for ethanol.*

## 2.6 Gas chromatography (GC)

Gas chromatography was used in this experiment to calculate the composition in the samples gathered from the apparatus. Gas chromatography is often used for separation of small quantities. The sample in question is heated and injected into a glass column where inert gas transports the mobile phase (the test sample) forward. Sample components are separated based on their boiling points and relative affinity for the stationary phase. The higher a component's affinity for the stationary phase, the slower it comes off the column. The components are then detected and represented as peaks on a chromatogram.[?]

## 3 Experimental

The following section describes the set up and procedure of the experiment.

### 3.1 Startup

The computer and the cooling water was turned on and the bottom and distillate taps were closed, and 5.8 L of a solution containing 11% ethanol and 89% water was poured into the boiler. The column was then started with total reflux, meaning that all the distillate was led back into the top of the column by a pump. In order to prevent the thermostat from interfering with the power supply the target temperature was set to 105 °C, which is a higher temperature than the boiling temperature of the solution. The boiler was set to have a power output of 40% of its maximum 4 kW, and the timer was started when the vapour stream started to condensate inside the cooler.

### 3.2 Time required to reach steady state

The first experimental task was to measure how long time it would take for the column to reach steady state. Therefore, starting at  $t = t_0$ , 13 distillate samples were collected with intervals of approximately 5 min in order to examine the composition of the distillate over time. (The data collected in this procedure are plotted in the diagram shown in Figure ??). **REFER- ANSE TIL PLOTTET MED DE FAKTISKE RESULTATENE** )  
<— resultater

### 3.3 Efficiency vs. vapour velocity

After the first thirteen samples had been collected, the boiler's power output was adjusted to 50% and let to stand for 30 min to ensure that steady state had been reached. Samples were then taken from both the distillate and the batch in the reboiler, and the reflux ratio was logged so that the vapour speed could be calculated. This procedure was then repeated with the power output at 60%, 70%, 80% and 90%.

### 3.4 Flooding and weeping point

To find the weeping point of the column, the reboiler's power output was lowered until liquid started seeping through the perforations in the plates. This occurred when the power output was set to somewhere between 5% and 10%. To find the flooding point, the power output was set to 100% (i.e. 4 kW), but this was still not sufficient to flood the column. The supervisor estimated that it would probably be necessary with a power source of 8 kW in order to properly flood the column.

### 3.5 Gas chromatography (GC)

500  $\mu\text{L}$  of each sample taken during the experimental procedure was mixed with 250  $\mu\text{L}$  isopropanol and analysed by gas chromatography to establish their composition.

### 3.6 Calculations

All the samples should be analysed in order to calculate efficiency and the theoretical number of stages will be calculated using McCabe Thiele method. The composition will be measured using pycnometers which uses the density difference in order to calculate the composition. The values are compared to tables found in literature. The pycnometers will be calibrated using distilled water at room temperature to obtain precise weight and volume. The samples are cooled down to room temperature (samples should be covered to avoid evaporation). Pour the samples and measure them in the pycnometer one-by-one, avoid air-bubbles. Dry the pycnometers between each sample test.

### 3.7 Ethanol-water mixture

In this experiment a solution with 11% ethanol is needed to distillate. By using section ?? with  $x_{et} = 0.11, \rho_{et} = 0.785 \text{ g cm}^{-3}, \rho_w = 1000 \text{ g cm}^{-3}, M_w =$

$18.02 \text{ g mol}^{-1}$ ,  $V_{tot} = 5.8 \text{ L}$ . The volume of ethanol needed is  $V_{et} = 1.655 \text{ L}$ . Only 96% ethanol is used so  $V_{et,96\%} = \frac{1.655}{0.96} = 1.724 \text{ L}$  is needed.

## 4 Results

## 5 Discussion

The gas chromatography did not work as expected. The first 12 samples gave reasonable results (except from two samples who had a mole fraction above 1). After the 12 first samples something went wrong, as can be seen in Table 4. First, all top samples for the power duty of 50%, 60%, 70% and 80% gave a mole fraction of zero, although the result for the top samples should be around 90%. The test indicates that the ethanol mole fractions of the bottom samples are too high to make any sense. Typically, a bottom sample should contain an ethanol mole fraction of 5 to 15 %<sub>wt</sub> ethanol, but the GC test show numbers that are far higher than this (although the numbers decrease from 62%<sub>wt</sub> to 26%<sub>wt</sub>). All of these figures are much higher than the starting concentration of 11%<sub>wt</sub> and therefore make little sense. What might have caused this is difficult to point out, but it is quite certain that the error doesn't lie in the mixing of the distillation solution as the amounts of water and ethanol were checked and double checked before mixing. All the sample tests prepared for GC had propanol added to them in the correct amount (250  $\mu\text{L}$ ) and were thoroughly shaken by both members of the experiment group, so it is difficult to see what might have happened. One possibility is that someone not involved in the experiment has moved the prepared samples around in or out of the GC machine carrousel causing the top sample zeros. This does however not explain the high concentrations in the bottom samples.

Trondheim, November 1, 2013

Signatures: \_\_\_\_\_



## References

## A Vapour velocity

The mean vapour velocity,  $v$ , is given as the volume of gas flowing through the column,  $\dot{V}$ , divided by the column's cross section area,  $A$ , as shown in (A.1):

$$v = \frac{\dot{V}}{A} \quad (\text{A.1})$$

Assuming ideal gas it is possible to calculate the volume flow of each component,  $\dot{V}_i$ , through the molar flow of each component,  $\dot{n}_i$ :

$$\dot{V}_i = \frac{RT\dot{n}_i}{p} \quad (\text{A.2})$$

where  $T$  is the temperature of the gas and  $R$  is the gas constant.

The molar flow of each component,  $\dot{n}_i$ , is found by dividing the mass flow of each component,  $\dot{m}_i$ , by the molar mass,  $M_i$ :

$$\dot{n}_i = \frac{\dot{m}_i}{M_i} \quad (\text{A.3})$$

The mass flow can be calculated by (A.4)

$$\dot{m}_i = \dot{V} \frac{V_i}{V_{tot}} \rho_i \quad (\text{A.4})$$

where  $\dot{V}$  is the total volume flow through the reflux column,  $V_i$  the volume flow of component  $i$ ,  $V_{tot}$  the **FIND WHICH TOTAL VOLUME THIS IS!**, and  $\rho_i$  the density of component  $i$ . Combining (A.1), (A.2), (A.3) and (A.4), a more usable expression for  $v$  is obtained:

$$v = \frac{RT}{Ap} \frac{\dot{V} \rho_i}{M_i} \frac{V_i}{V_{tot}} \quad (\text{A.5})$$

Using the the relation in (A.6)

$$\dot{n} = n_{etOH} + n_{H_2O} \quad (\text{A.6})$$

a final expression of  $v$  is achieved:

$$v = \frac{RT}{Ap} \left( \frac{\dot{V} \rho_{etOH}}{M_{etOH}} \frac{V_{etOH}}{V_{tot}} + \frac{\dot{V} \rho_{H_2O}}{M_{H_2O}} \frac{V_{H_2O}}{V_{H_2O}} \right) \quad (\text{A.7})$$