

Felles-lab ST2:  
Distillation, Oldershow perforated plate  
Draft

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**Gruppe B16**

November 1, 2013



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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

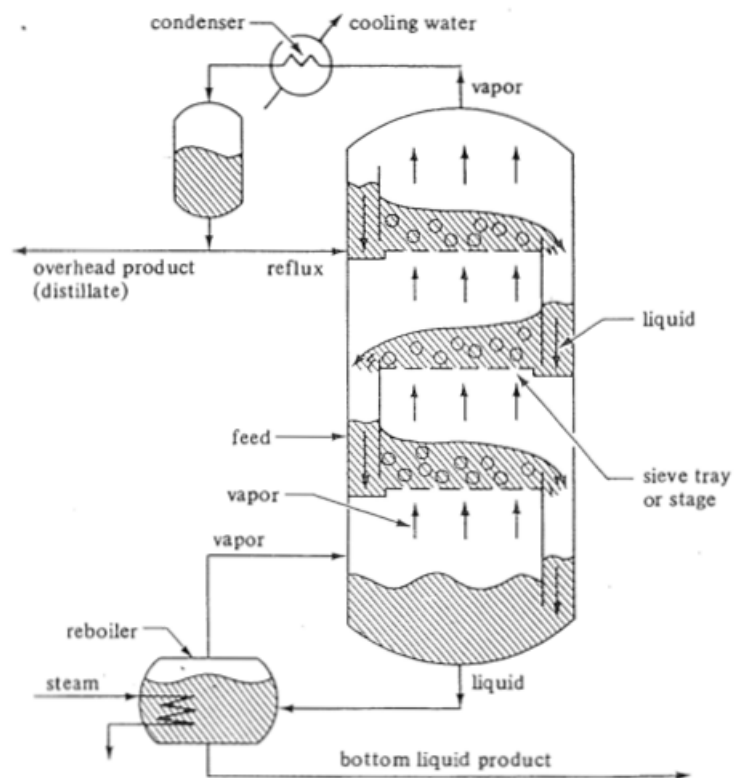
The theory in this experiment is gathered from the Task booklet [1] and Geankoplis [2].

### 2.1 Basic principles of distillation

Distillation is a technique of separating two liquids based on their differences in boiling temperature. Distillation takes place in a column which is designed to give a maximum vapour vs. liquid interaction in order to establish as many the vapour/liquid equilibriums (VLE) as possible. This is achieved by using trays that give a large liquid front. The trays are mounted on top of each other as can be seen in Figure 1, which is gathered from Geankoplis [2]. In general, the more trays, the better separation it is possible to achieve. The distillation column is driven by heat from a reboiler at the bottom of the column which evaporates the liquid. As the vapour progresses up through the column, it comes contact with liquid flowing downwards, establishing VLEs causing the component with the lowest boiling point to condensate. At the top of the column is a condenser which cools the vapour to make it condensate. This condensate flow can be collected into product (distillate) or be sent back into the column (reflux). If all condensate is directed back to the column, it is said that the column is operated in total reflux mode.

### 2.2 Weeping point

A weeping point is a condition in the distillation column which occurs when the vapour velocity is too low. The vapour pressure is not high enough to hold up the liquid in a tray, and instead the liquid flows through the perforated holes in the tray and drips ("weeps") out underneath. The vapour's contact with the liquid diminishes, and this causes both the pressure and the efficiency to drop.



*Figure 1: Fractionated distillation column containing sieve trays. The figure is gathered from chapter 11.4A in Geankoplis [2]*

## 2.3 Flooding point

The capacity of a countercurrent separation (liquid flows downwards and vapour flows upwards) 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.

## 2.4 Column efficiency

The efficiency of a distillation column is given by the ratio of the total number of ideal equilibrium stages,  $N_t$ , and the total number of actual trays in the column,  $N_a$ . A high efficiency means that for each stage, the upcoming vapour is close to or in equilibrium with the liquid flooding downwards. The equation for column efficiency ( $\eta$ ) is given by (2.1),

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

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

## 2.5 Reflux

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

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

where  $R_R$  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. If the column is operated in total reflux mode, this means that all condensate flows back into the column so that  $R_R \rightarrow \infty$ . This has an effect of the calculation of the operating lines. This means that the slope of the operation lines given in equation (2.3) and (2.4) becomes  $\frac{\infty}{\infty} = 1$  so that the operating line is in a  $45^\circ$  angle.

## 2.6 McCabe-Thiele method

The McCabe-Thiele method is a graphical method for calculating the total number of theoretical trays in a given binary (two-component) 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 [2]) for both components, a McCabe-Thiele diagram can be generated and the number of theoretical stages can be determined graphically (see Figure 2). 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 given by Equation (2.3),

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

where  $R_R$  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 given by Equation (2.4),

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

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 determines 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 [2])

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

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 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

**Figure 2:** The figure shows the McCabe-Thiele diagram for ethanol/water mixture, plotted for ethanol.

Table 1. The number of theoretical stages is then determined using the following procedure:

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 A the McCabe-Thiele diagrams in Section 4 were generated.

## 2.7 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 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 travels through the column. The components are then detected and represented as peaks on a chromatogram, as described in Perry's [3].

## 3 Experimental

The following section describes the set up and procedure of the experiment as it was performed.



### 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%<sub>wt</sub> ethanol and 89%<sub>wt</sub> 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 solution can achieve. 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  $t = t_0$ , 12 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 3.)

### 3.3 Efficiency vs. vapour velocity

After the first thirteen samples had been collected, the boiler's power output was adjusted to 50% and left 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 power outputs of 60%, 70%, 80% and 90% respectively.

### 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 Ethanol-water mixture

In this experiment a solution with 11% ethanol was needed for distillation. By using section B 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

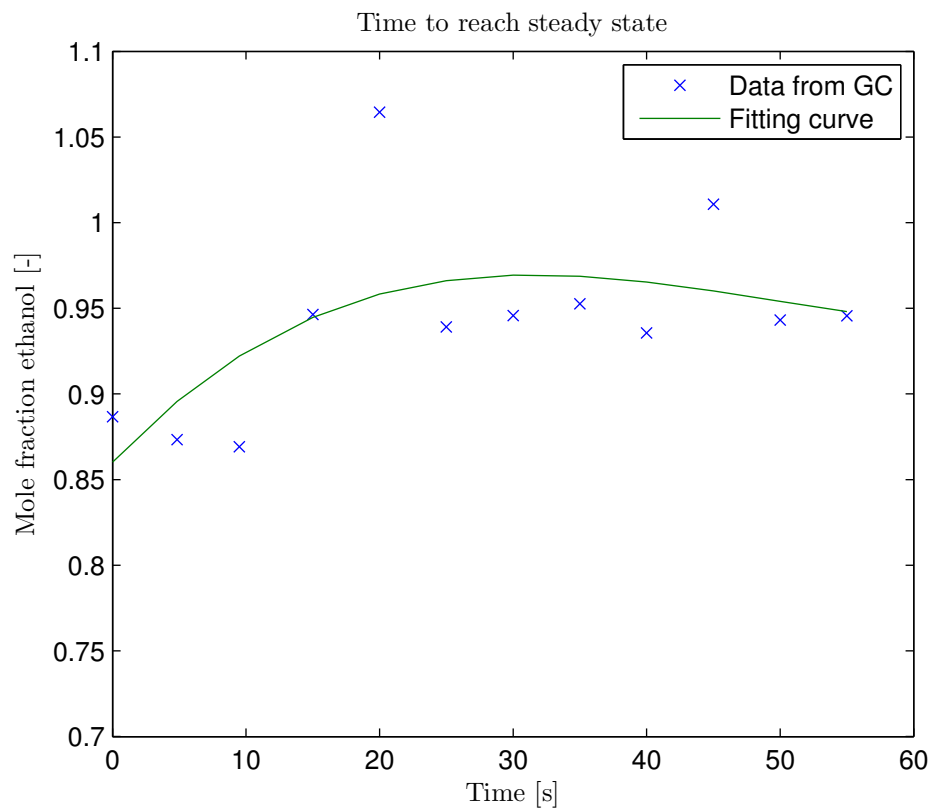
All samples were analysed using gas chromatography, however something seems to have gone wrong after the 12th sample. The upshot is that the samples after the 55 min sample have been rendered unsuitable for analytical purposes. In order to complete the exercise, a set of constructed top and bottom samples was constructed, displayed in Figure 3. The actual results are found in Table 2. Further discussion regarding the actual results is given in section 5.

### 4.1 Time required to reach steady state

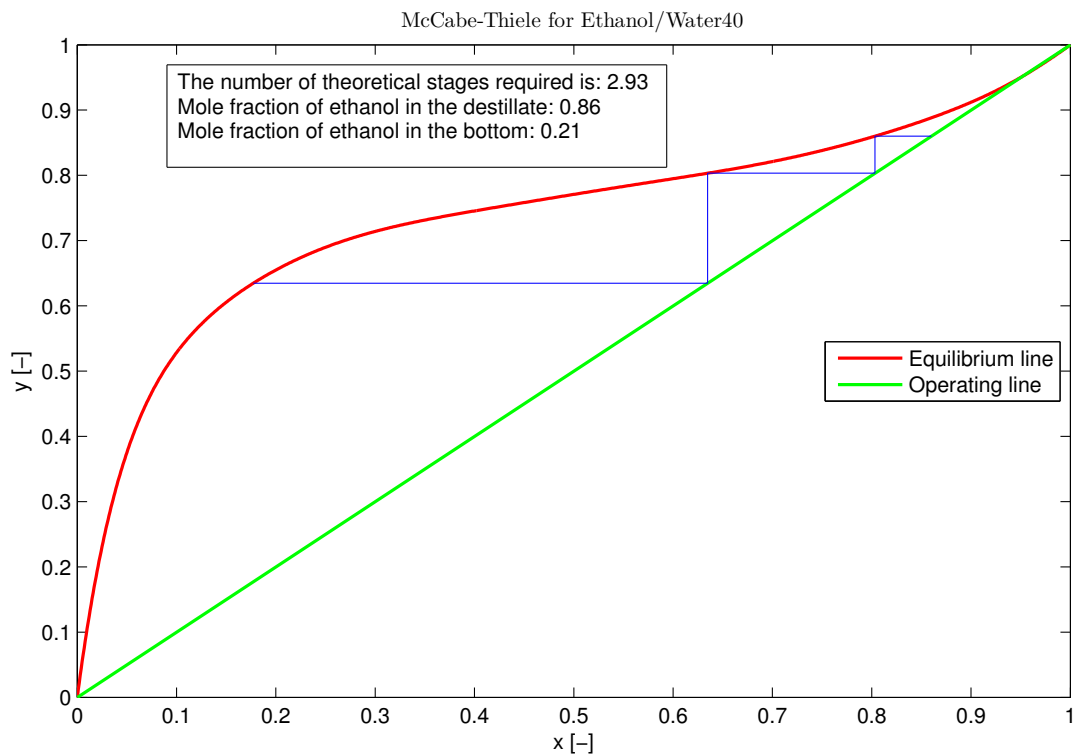
Figure 3 shows the mole fractions of ethanol in the distillate plotted against time in seconds, the values taken from Table 2. Some of the samples show a mole fraction higher than 1 (which is impossible). From Figure 3 it seems that steady state is reached after about 12 minutes, but it is important to be aware that as two samples got a higher mole fraction than 1, it is likely that the samples have a high level of uncertainty.

### 4.2 McCabe-Thiele diagrams

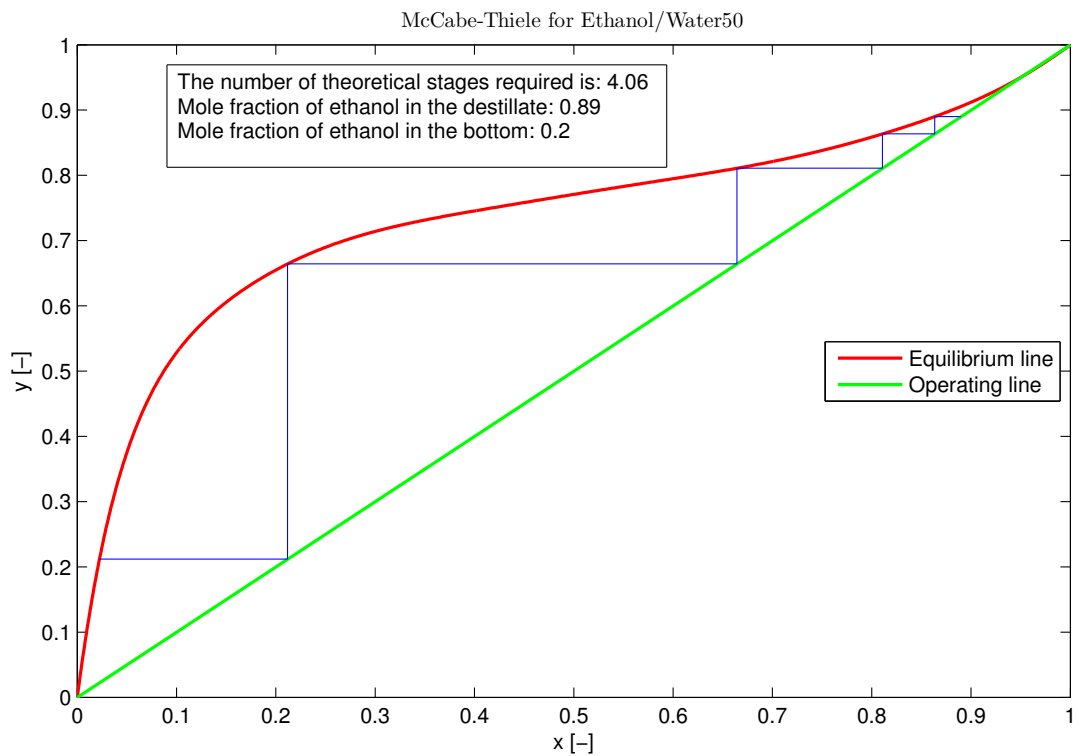
*The following diagrams have been generated from the made-up top and bottom fractions given in Table 3*



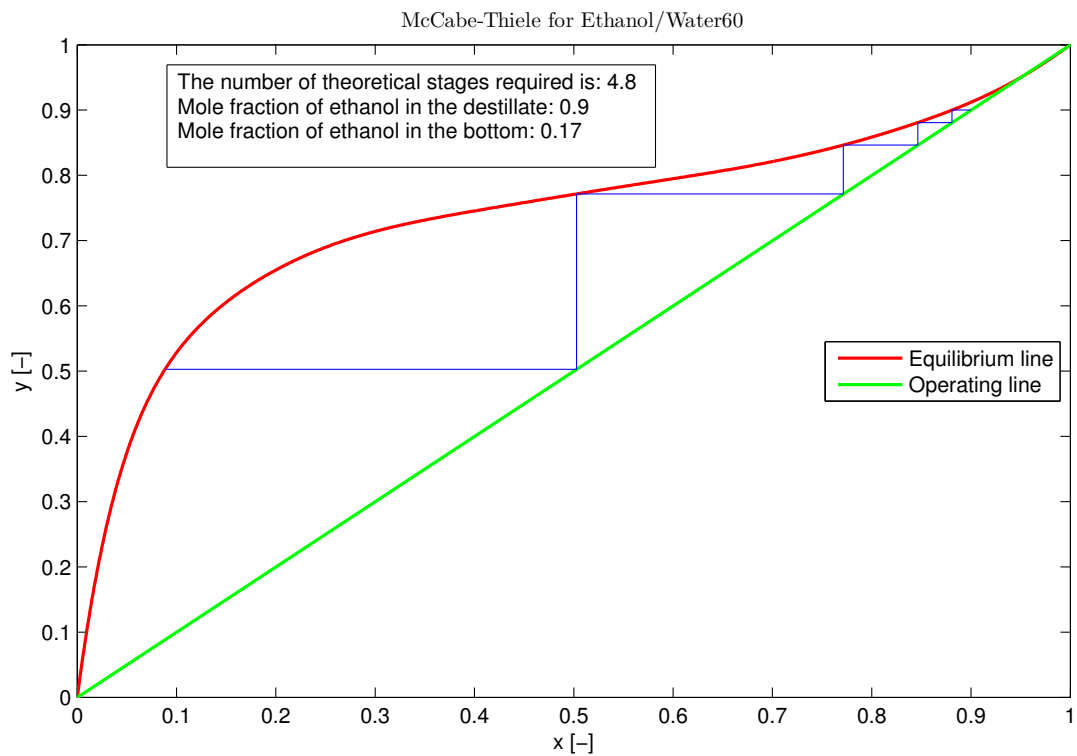
*Figure 3: Plot of the mole fractions of the sample tests after a given time. The fitted curve was found using MATLAB's polyfit tool with a 3rd degree polynomial.*



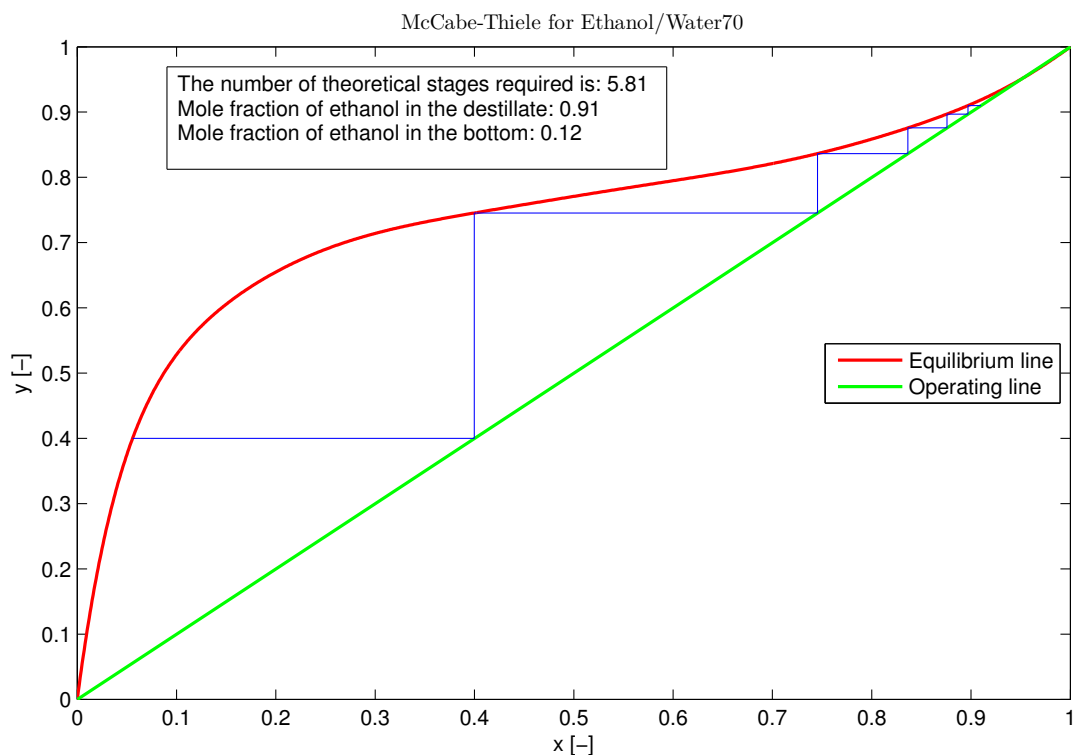
**Figure 4:** The figure shows the McCabe-Thiele diagram for column with a power output of 40%.  $x$  is the mole fraction of ethanol in liquid phase and  $y$  is the mole fraction of ethanol in the vapour phase. The plot calculating the number of theoretical steps is made from the MATLAB-script in A.2. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.



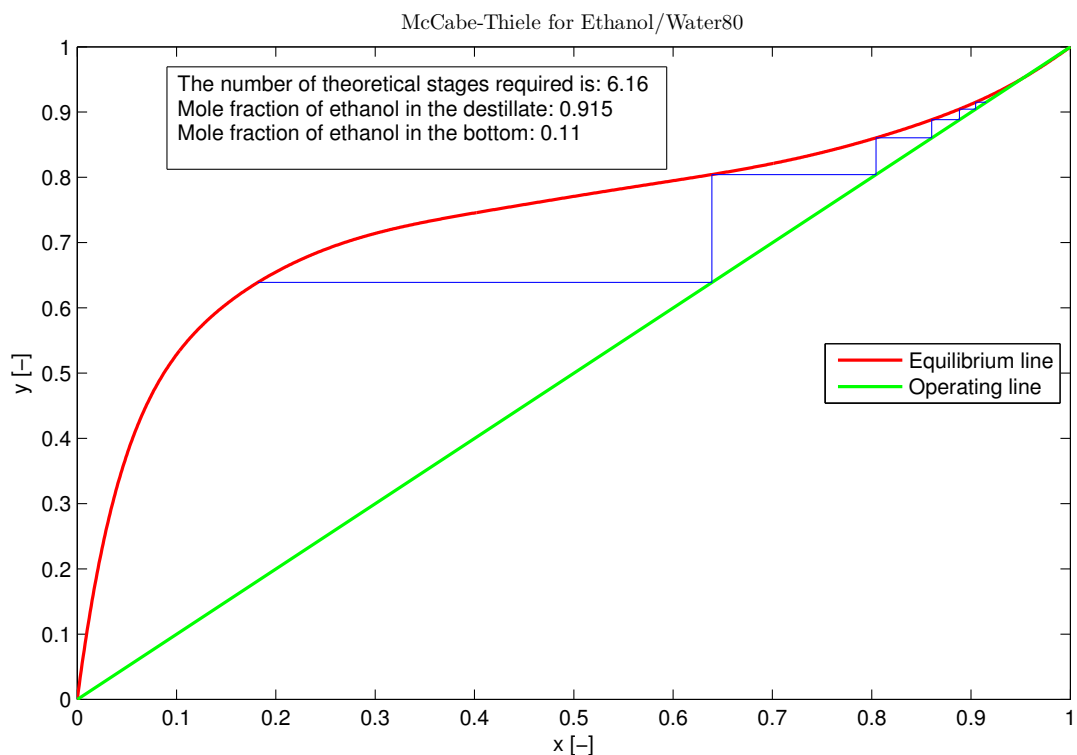
**Figure 5:** The figure shows the McCabe-Thiele diagram for column with a power output of 50%.  $x$  is the mole fraction of ethanol in liquid phase and  $y$  is the mole fraction of ethanol in the vapour phase. The plot calculating the number of theoretical steps is made from the MATLAB-script in A.2. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.



**Figure 6:** The figure shows the McCabe-Thiele diagram for column with a power output of 60%.  $x$  is the mole fraction of ethanol in liquid phase and  $y$  is the mole fraction of ethanol in the vapour phase. The plot calculating the number of theoretical steps is made from the MATLAB-script in A.2. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.

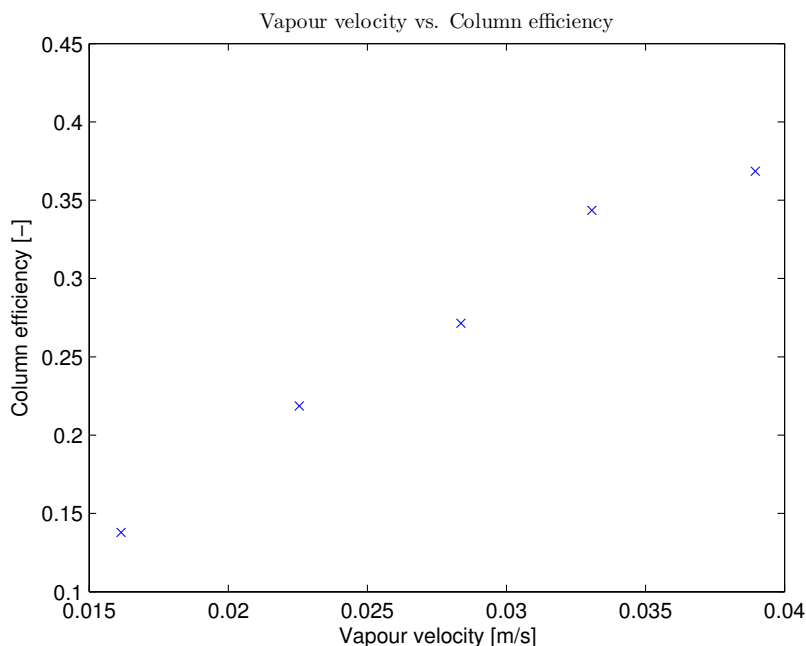


**Figure 7:** The figure shows the McCabe-Thiele diagram for column with a power output of 70%.  $x$  is the mole fraction of ethanol in liquid phase and  $y$  is the mole fraction of ethanol in the vapour phase. The plot calculating the number of theoretical steps is made from the MATLAB-script in A.2. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.



**Figure 8:** The figure shows the McCabe-Thiele diagram for column with a power output of 80%.  $x$  is the mole fraction of ethanol in liquid phase and  $y$  is the mole fraction of ethanol in the vapour phase. The plot calculating the number of theoretical steps is made from the MATLAB-script in A.2. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.





**Figure 9:** The figure is showing a plot of column efficiency vs. the vapour velocity for the power duties 40% ,50% ,60% ,70% and 80%. The plot has been created using made-up data from Table 3 as the empirical data from the lab were unusable.

### 4.3 Column efficiency vs. vapour velocity

The column efficiency was calculated from equation (2.1), with  $N_a = 14$ . The vapour velocity was calculated from (E.7). It was assumed that  $y_i = V_i/V_{tot}$  could be calculated directly from the VLE-data using  $x_D$  from the sampling. Figure 9 shows the plot of column efficiency vs. the vapour velocity for each power duty using the constructed set of data in Table 3.

## 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), but after the 12 first samples something seems to have gone wrong, as can be seen from the data in Table 4. Firstly, all top samples for the power duty of 50%, 60%, 70% and 80% apparently had a mole fraction of zero, although the result for the top samples should be around or above 90%<sub>wt</sub>. The test results for the ethanol mole fractions in the bottom samples are too high to make any sense. Typically, a bottom sample should contain 5%<sub>wt</sub> to 15%<sub>wt</sub> ethanol, but the GC test results show figures far greater 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 as a distillation process would extract ethanol from the solution, lowering the ethanol mole fraction in the batch. What might have caused this is difficult to point out, but it is quite certain that the error does not lie in the preparation 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 isopropanol added to them in the correct amount (250  $\mu$ L) and were thoroughly shaken by both members of the experiment group, so it is difficult to see what might have gone wrong there. One possibility is that someone not involved in the experiment moved the prepared samples around in or completely out of the GC carousel causing the top sample zeros. This does however not explain the high concentrations in the bottom samples.

Steady state for the column is reached when the the graph in Figure 3 flattens out. As seen in the figure, the graph looks flat after 15 minutes. However, there is probably a high uncertainty from the GC, as described in the previous paragraph and seen in Figure 3: The graph actually turns near the end because of the outliers (points where  $x_D > 1$ ).

The best setting for the column is the peak of the column efficiency. The column efficiency tends to increase with higher vapour velocity, meaning that the best setting for the column is when the power duty is above 80%. It is important to note that Figure 9 was calculated from constructed data, so for the real data the trend might not have been the same.

## 6 Conclusion

The time required to reach steady state was estimated to approximately 15 min. Even at 4 kW (full power) flooding was not observed, so it was concluded that it was not possible to flood the column with the power supply on hand. The weeping point was observed to be at below 10% power duty (i.e. 400 W). This means that the minimum power required in order to distillate was 400 W. The theoretical number of trays required at different power duties was calculated using MATLAB and was found to 2.93, 4.06, 4.8, 5.81 and 6.1 for power duties of 40%, 50%, 60%, 70% and 80% respectively. These figures were calculated using constructed data due to unapplicable results from the GC analysis. The column efficiency increased with higher vapour velocity (higher power output) and was 0.369 at a power duty of 80% (for the constructed data set).

## List of symbols and constants

Symbol	Unit	Description
$A$		Cross section area of column
$D$	$\text{mL s}^{-1}$	Distillate flow out of column
$L_m$	$\text{mL s}^{-1}$	Liquid flow from tray $m$
$L_n$	$\text{mL s}^{-1}$	Condensate flow back into column
$M_{EtOH}$	$\text{g mol}^{-1}$	Molar mass of ethanol
$M_{H_2O}$	$\text{g mol}^{-1}$	Molar mass of $\text{H}_2\text{O}$
$M_i$	$\text{g mol}^{-1}$	Molar mass of component $i$
$\dot{m}_i$		Mass flow of component $i$
$N_a$	[-]	Actual number of equilibrium stages
$N_t$	[-]	Theoretical number of equilibrium stages
$\dot{n}_i$	$\text{mol s}^{-1}$	Mole flow of component $i$ in gas phase through column
$\dot{n}$	$\text{mol s}^{-1}$	Total mole flow of gas through column
$\dot{n}_{EtOH}$	$\text{mol s}^{-1}$	Mole flow of ethanol in gas phase through column
$\dot{n}_{H_2O}$	$\text{mol s}^{-1}$	Mole flow of $\text{H}_2\text{O}$ in gas phase through column
$p$	bar	Pressure
$q$	[-]	The feed condition used to define the $q$ -line in a McCabe-Thiele diagram, defined by Equation (11.4-12) in Geankoplis [2]
$R$	$\text{J K}^{-1} \text{mol}^{-1}$	The universal gas constant
$R_R$	[-]	Reflux ratio
$T$	K	Temperature
$V_{EtOH}$	mL	Volume of ethanol in distillate
$V_{H_2O}$	mL	Volume of $\text{H}_2\text{O}$ in distillate
$V_i$	mL	Volume of component $i$ in distillate
$V_m$	$\text{mL s}^{-1}$	Vapour in stripping section $m$
$V_{m+1}$	$\text{mL s}^{-1}$	Vapour flow from tray $m+1$
$V_n$	$\text{mL s}^{-1}$	Vapour in stripping section $n$
$V_{tot}$	mL	Total volume of distillate
$\dot{V}_{gas}$	$\text{m}^3 \text{s}^{-1}$	Gas flow through column

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$\dot{V}_i$	$\text{m}^3 \text{s}^{-1}$	Gas flow of component $i$ through column
$v$	$\text{m s}^{-1}$	Average vapour velocity inside column
$W$	$\text{mL s}^{-1}$	Flow out of the bottom of the column
$x_B$	[-]	Mole fraction of light component in the stream flowing out from the reboiler
$x_D$	[-]	Mole fraction of light component in distillate
$x_f$	[-]	Mole fraction of light component in liquid phase in the feed
$x_m$	[-]	Mole fraction of light component in the liquid phase at tray $m$
$x_n$	[-]	Mole fraction of light component in liquid phase at tray $n$
$x_W$	[-]	Mole fraction of light component in the out flow
$y_f$	[-]	Y-value at feed point
$y_{m+1}$	[-]	Mole fraction of light component in gas phase at tray $m+1$
$y_{n+1}$	[-]	Mole fraction of light component in gas phase at tray $n+1$
$\eta$	[-]	Column efficiency
$\rho_{EtOH}$	$\text{g cm}^{-3}$	Density of ethanol
$\rho_{H_2O}$	$\text{g cm}^{-3}$	Density of $\text{H}_2\text{O}$
$\rho_i$	$\text{g cm}^{-3}$	Density of component $i$

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## References

- [1] Felles lab: Distillation Columns, [http://www.nt.ntnu.no/users/preisig/Repository/TKP\\_4110\\_Felles\\_Lab/experiment%20descriptions/DistInstruct.pdf](http://www.nt.ntnu.no/users/preisig/Repository/TKP_4110_Felles_Lab/experiment%20descriptions/DistInstruct.pdf), visited: 1.10.2013
- [2] Geankoplis, Christie John *Transport Processes and Separation Process Principles (includes unit operations)*, 4. ed; Prentice Hall, 2003
- [3] Perry, R.H and Green, D.W, *Perry's Chemical engineers' Handbook*, 8. ed; McGraw-Hill, 2008

## A MATLAB

### A.1 Steady state plot

```
function p = steadystate(inputfile)
    data = load('steadystate.txt');
    time = data(:,2);
    frac = data(:,1);
    p = polyfit(time,frac,3);
    f = polyval(p,time);
    figname = ['Time to reach steady state'];
    hFig = figure('Name',figname,'Visible','on');
    set(hFig,'NumberTitle','off');
    plot(time,frac,'x',time,f,'-');
    axis([0 60 0.7 1.1])
    legend('Data from GC', 'Fitting curve')
    xlabel('Time [s]','Interpreter','LaTeX');
    ylabel('Mole fraction ethanol [-]','Interpreter','LaTeX');
    title(figname,'Interpreter','LaTeX');
    exportname = ['steadystate'];
    export_fig(exportname, '-pdf', '-transparent')
end
```

### A.2 Number of theoretical steps

```
%\begin{minted}[frame=lines]{matlab}
%This script plots the equilibrium line for a binary mixture of ethanol and
%water, and calculates the number of steps needed to reach a certain
%concentration with total reflux.
%PART 1 - Plotting the equilibrium line and the operating line
xy = load('vle2.txt');
x = xy(:,1);
y = xy(:,2);
%Distillate composition, xd and bottom composition, xb
xd = [0.86 0.89 0.90 0.91 0.915];
xb = [0.21 0.20 0.17 0.12 0.11];
pow = [40 50 60 70 80];
for i=1:length(xd)
    %x is the mole fraction of ethanol in the liquid phase, and y is ...
```

```
%the mole
%fraction of ethanol in the gas phase.
%Fit a polynom of tenth degree, p, to fit the data
p = polyfit(x,y,10);
%plot p and the operating line y = x

hFig = figure('Name','McCabe-Thiele','Position',[50 150, 800, 500]);
eq = plot(0:0.001:1,polyval(p,0:0.001:1));
set(eq, 'Color','red','LineWidth',1.5)
op = line([0 1],[0 1]);
set(op, 'Color','green','Linewidth',1.5)
axis([0 1 0 1]);
xlabel('x [-]');
ylabel('y [-]');
legend('Equilibrium line', 'Operating line','Location','East');

%Calculating and plotting theoretical stages
% gets input from the function molefraction.m
                                %puts in wanted values

xs=[];
ys=[];
% Starts at the top at the operatingline y=x
xs(1)=xd(i);
ys(1)=xd(i);
f =@(x,y)(p(1)*x^10+p(2)*x^9+p(3)*x^8+p(4)*x^7+p(5)*x^6+p(6)*x^5+...
p(7)*x^4+p(8)*x^3+p(9)*x^2+p(10)*x^1+p(11) - y);%Solve the equation
j=1; %making a counter
    while xs(j)>xb
        xs(j+1)=fzero(f,0.5,[],ys(j));
        line([xs(j) xs(j+1)],[ys(j) ys(j)],'color','b');
        ys(j+1)=xs(j+1);
        if xs(j+1)>xb
            line([xs(j+1) xs(j+1)],[ys(j) ys(j+1)],'color','b');
        end
        j=j+1;
    end

%Calculating number of theoretical steps
N=j-2+(xb(i)-xs(j-1))/(xs(j)-xs(j-1));
N=round(N*100)/100;
str={['The number of theoretical stages required is: ' num2str(N)],...
```

```

        ['Mole fraction of ethanol in the destillate: ' ...
        num2str(round(xd(i)*1000)/1000)],...
        ['Mole fraction of ethanol in the bottom: ' ...
        num2str(round(xb(i)*1000)/1000)]];
    stbox = annotation('textbox',[0.2 0.8 0.1 0.1],'String',str);
    figname = ['McCabe-Thiele for Ethanol/Water', num2str(pow(i))];
    title(figname,'Interpreter', 'LaTeX')
    set(hFig,'NumberTitle','off','Visible','off');
    exportname = num2str(pow(i));
    export_fig(exportname, '-pdf', '-transparent');
    pow(i)

end
% Create the table of constructed data for the experiment in LaTeX code
fileID =fopen('xdxb.txt','w');
fprintf(fileID,'%s \r\n','\toprule')
fprintf(fileID,'%s & %s & %s %s \r\n ', 'Reboiler power', '$x_d$ [-]' ...
    , '$x_b$ [-]', '\\');
fprintf(fileID,'%s \r\n','\midrule');
for i=1:length(xb)
    fprintf(fileID,'% -3.f & %5.3f & %5.3f %s \r\n',pow(i), ...
        xd(i),xb(i), '\\');
end
fprintf(fileID,'%s','\bottomrule')
fclose(fileID);

```

### A.3 Calculation and plot of vapour velocity vs. column efficiency

```

function v = effiplot(effi,xd,p)
%Plots the efficiency vs. vapour velocity
    reflow = [57.2 82.5 105 124 147]; %ml/min
    reflow = reflow*1e-6*(1/60); %m3/s
    R = 8.314; %J/Kmol
    rhoetoh = 785; % kg/m3
    rhowat = 997; %kg/m3
    A = 3.8E-3; %m2
    T = 363; %K
    P = 10e5; %kg/ms2
    metoh = 4.607e-2; %kg/mol

```



```
mwat = 1.802e-2; %kg/mol
f =@(x)(p(1)*x^10+p(2)*x^9+p(3)*x^8+p(4)*x^7+p(5)*x^6+p(6)*x^5+...
    p(7)*x^4+p(8)*x^3+p(9)*x^2+p(10)*x^1+p(11));
for i=1:length(reflow)
    v(i) = ((R*T)/(A*P))*(reflow(i)*((f(xd(i))*rhoetoh/metoh)+...
        ((1-f(xd(i)))*rhowat/mwat)));
end
hFig = figure('Name','Vapour velocity');
figname = ['Vapour velocity vs. Column efficiency'];
plot(v,effi,'x')
xlabel('Vapour velocity [m/s]')
ylabel('Column efficiency [-]')
title(figname,'Interpreter','LaTeX');
set(hFig,'NumberTitle','off','Visible','off');
export_fig('velocity','-pdf','-transparent');
end
```

## B Equations for Ethanol-water mixture

The mole fraction for a given component  $i$  is given by equation (B.1),

$$x_i = \frac{n_i}{n_{tot}} \quad (\text{B.1})$$

where  $n_i$  stands for the number of mole for component  $i$ , and  $n_{tot}$  for the total number for mole in the mixture. Number of moles when using pycnometers is given by equation (B.2),

$$n_i = \frac{V_i \rho_i}{M_i} \quad (\text{B.2})$$

where  $V_i$  is the volume of component  $i$ ,  $\rho$  the density and  $M$  the molar weight. For the mixture of water and ethanol, the number of moles of ethanol is given by the following equation,

$$n_w = \frac{\rho_w(V_{tot} - V_{et})}{M_w} \quad (\text{B.3})$$

where index  $_{et}$  is ethanol and  $_w$  is water. The mole fraction of ethanol can be found by combining (B.1), (B.2) and (B.3).

$$x_{et} = \frac{\frac{V_{et} \rho_{et}}{M_{et}}}{\frac{V_{et} \rho_{et}}{M_{et}} + \frac{\rho_w(V_{tot} - V_{et})}{M_w}} \quad (\text{B.4})$$

## C Results from GC

**Table 2:** The table is showing the results from the Gas Chromatography. The 5th column explains the sample content. So that 50 means sample taken after 50 minutes.

Sample name	Sampling time	Carousel #	$x_{EtOH}$	Sample content
B16_1	13:29	81	0,88675493	0
B16_2	13:34	82	0,8733395338	4,8333333333
B16_3	13:40	83	0,8692047	9,5
B16_4	13:45	84	0,9464284182	15
B16_5	13:50	85	1,0645126104	20
B16_6	13:56	86	0,9390891194	25
B16_7	14:01	87	0,9457674026	30
B16_8	14:11	88	0,9525999427	35
B16_9	14:22	89	0,9356185198	40
B16_10	14:27	90	1,0107297897	45
B16_11	16:46	91	0,9431585073	50
B16_12	16:51	92	0,9456739426	55
B16_13	16:57	93	0,664142549	40btm
B16_14	17:07	94	0	50top
B16_15	17:13	95	0,619551897	50btm
B16_16	17:18	96	0	60top
B16_17	17:23	97	0,5991532803	60btm
B16_18	17:29	98	0	70top
B16_19	17:34	99	0,3690922856	70btm
B16_20	17:41	100	0	80top
B16_21	17:46	101	0,2554974258	80btm

## D Constructed data

The data in Table 3 was constructed in order to complete the rapport as there was something terribly wrong with the results from the gas chromatography.

**Table 3:** *The table is showing the constructed output from the top and bottom. These data are used for all calculations regarding comparing different column settings.*

Reboiler power	$x_d$ [-]	$x_b$ [-]
40	0.860	0.210
50	0.890	0.200
60	0.900	0.170
70	0.910	0.120
80	0.915	0.110

Table 4 shows how the efficiency correlates to the number of theoretical steps for the constructed data.

**Table 4:** *The table is showing the number of theoretical steps and the column efficiency for different power duties.*

Reboiler power	Theoretical steps, N	Efficiency $\eta$
40	2.930	0.138
50	4.060	0.219
60	4.800	0.271
70	5.810	0.344
80	6.160	0.369

## E Vapour velocity

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

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

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

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

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,  $M_i$ :

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

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

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

where  $\dot{V}_{gas}$  is the total volume flow of gas through the reflux column,  $V_i$  the volume of component  $i$  in the distillate,  $V_{tot}$  the total volume of distillate, and  $\rho_i$  the density of component  $i$ . Combining (E.1), (E.2), (E.3) and (E.4), a more usable expression for  $v$  is obtained:

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

Using the the relation in (E.6)

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

a final expression of  $v$  is achieved:

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