Oldershaw perforated plate distillation

Felleslab, TKP 4105 and TKP 4110

Title: Oldershaw perforated plates
Location: Trondheim

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Abstract
An oldershaw perforated plates distillation was performed on a water-ethanol mixture using complete reflux to study steady state, theoretical trays, column efficiency, flooding point and weeping point. The column efficiency was found to be highest with a heating duty of 60%. The weeping point was reached at a boiler power duty of 10%, while the column proved unable to reach the flooding point.

Jeg erklærer at arbeidet er utført selvstendig og i samsvar med NTNUs eksamensreglement.

Date and signatures:
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1 Purpose
The purpose of the experiment was to be introduced to the basic principles of steady state, column efficiency, vapor velocity and theoretical trays, by performing an oldershaw perforated plate distillation.

2 Theory

2.1 Flooding point
In a distillation column the vapor flows upwards and the liquid flows downwards. The flooding point is referred to as the vapor velocity when liquid accumulates in the column [1]. This happens when the vapor velocity gets too high, and the vapor drags the liquid flow up the column. The flooding point causes a pressure drop in the column, as well as reduced efficiency [2].

2.2 Weeping point
At steady state conditions the flow of liquid through the perforations is stopped by vapor velocity through the perforations [1]. Low vapor velocity causes the liquid to “weep” down through the perforations, giving less vapor-liquid contact, resulting in a lower efficiency [2].

2.3 McCabe-Thiele method
The McCabe-Thiele method is a graphical method for analysis of distillation by determination of the number of theoretical trays needed for a given separation [3]. It bases around the assumption of constant molar flows. Using mass balances and vapor-liquid equilibrium (VLE) data for components, a McCabe-Thiele diagram can be constructed, and theoretical number of trays can be determined [3]. An example is shown in Figure 8. The horizontal axis represents mole fraction of light component in liquid phase, while vertical axis represents mole fraction of light component in gas phase. Plotting VLE data gives the equilibrium line, while mass balance gives the enriching and stripping operating lines.

For the enriching section of the column, the mole fraction of light component in gas phase at tray \( n + 1 \), \( y_{n+1} \), is given by [3]:

\[
y_{n+1} = \frac{R}{R+1} x_n + \frac{x_D}{R+1}
\]  

(2.1)

Where \( R \) is the reflux ratio, \( x_n \) is the mole fraction of light component in liquid phase at step \( n \) and \( x_D \) is the mole fraction of light component in the distillate. For the stripping section of the column, the mole fraction of light component in gas phase at tray \( m + 1 \), \( y_{m+1} \), is given by [3]:

\[
y_{m+1} = \frac{L_m}{V_{m+1}} x_m - \frac{W x_W}{V_{m+1}}
\]  

(2.2)

Where \( L_m \) is the liquid flow from tray \( m \), \( V_{m+1} \) is vapor flow from tray \( m + 1 \), \( W \) is bottom flow, \( x_W \) is mole fraction of light component in bottom flow and \( x_m \) is mole fraction of light component in liquid phase at tray \( m \). In this experiment it is assumed constant molar flows, e.g. \( L_m = L_{m+1} \) and \( V_m = V_{m+1} \).
The parameter \( q \) represents the condition of the feed and is defined as [3]:

\[
q = \frac{\text{heat needed to vaporize 1 mole of feed at entering conditions}}{\text{molar latent heat of vaporization of feed}}
\]  

(2.3)

This is used in the diagram to plot the \( q \)-line, with slope \( q/(q - 1) \), crossing the crossing-point of the operating lines and the \( x = y \) line.

To determine number of theoretical stages required in a distillation, steps are drawn in the diagram, starting at the top tray where \( x = x_D \). The steps are made by drawing horizontal and vertical alternating lines between the equilibrium line and the operating line, until \( x = x_B \) is reached. The number of steps in the diagram represent the number of theoretical trays required in the distillation [3].

2.3.1 Total reflux

With total reflux the reflux ratio is infinitely large, hence the operating line can be found by taking the limit of (2.2) as \( R \) approaches infinity:

\[
y = \lim_{R \to \infty} \frac{R}{R + 1} x + \frac{x_D}{R + 1}
\]

(2.4)

This result can be used as the operating in the McCabe-Thiele method with total reflux, consequently the operating line at total reflux is given as the line \( y = x \).

2.4 Gas chromatography

Gas chromatography (GC) is used for separation and detection of gasses and volatile components in mainly organic solutions. This is an analytical solution used to test the purity of the sample or the relative amount of each component of the test sample. [4]

In gas chromatography there is a moving phase, usually an inert gas, and a stationary phase consisting of a polymer or glass, called column. The sample will elute at different times on the column, called the retention time of the component. Using this, the weight fraction of each component can be calculated. [4]

3 Experimental

3.1 Column startup

The computer and cooling water were turned on, and the bottom and distillate taps were closed. The oldershow perforated plates column was filled with ethanol (11%, 5800 mL). The column was set to total reflux, and the boilers power duty was set to 90%, with a control box temperature of 105 °C. When the top temperature or pressure changed substantially the boiler was set to 50%. The time when the vapor stream started condensing was noted as zero time, \( t = 0 \).

In case of flooding the heated was turned off, and in case of any emergency the heater was turned off and the cooling left running.
3.2 Time required to reach steady state condition
Samples were taken from the top of the column every 5 minutes until 12 samples had been taken. At
the time of the 12\textsuperscript{th} sample, a sample was also taken from the bottom of the column. Samples were
analyzed using GC.

3.3 Efficiency vs. vapor velocity
The power was set to 40\%, and the column was left alone until it had reached steady state. Samples
(20 mL) were taken from top and bottom, to be analyzed. This was performed for 5 different boiler
duties. After the last sample, weeping and flooding points of the boiler were noted.

3.4 Column shutdown
After all samples were taken, the heater was turned off, and the column was left to cool. The column
was emptied, cooling water and computer were turned off.

4 Results
The mole fraction of ethanol in distillate at a boiler duty of 40\% was plotted against time in Figure 1.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Mole fraction of ethanol in distillate, $x_{et}$, plotted against time, $t$. It appears as if the data point at 15 min should have been higher, which would give a steady state after ~15 min, while the point at 35 min is higher than it should be.}
\end{figure}

In Figure 2, Figure 3, Figure 4, Figure 5 and Figure 6 the McCabe-Thiele diagrams are plotted for
boiler duties of 40\%, 50\%, 60\%, 70\% and 80\%, respectively.
Figure 2: McCabe-Thiele diagram for a power duty of 40%. Here, $x$ is the mole fraction of ethanol in the liquid phase and $y$ is the mole fraction of ethanol in the gas phase. The plot is made with Program code 1 in Appendix C - Matlab scripts.

Figure 3: McCabe-Thiele diagram for a power duty of 50%. Here, $x$ is the mole fraction of ethanol in the liquid phase and $y$ is the mole fraction of ethanol in the gas phase. The plot is made with Program code 1 in Appendix C - Matlab scripts.
Figure 4: McCabe-Thiele diagram for a power duty of 60%. Here, \( x \) is the mole fraction of ethanol in the liquid phase and \( y \) is the mole fraction of ethanol in the gas phase. The plot is made with Program code 1 in Appendix C - Matlab scripts.

The number of stages required is: 9.09
With a heating duty of: 60 %

Figure 5: McCabe-Thiele diagram for a power duty of 70%. Here, \( x \) is the mole fraction of ethanol in the liquid phase and \( y \) is the mole fraction of ethanol in the gas phase. The plot is made with Program code 1 in Appendix C - Matlab scripts.

The number of stages required is: 4.47
With a heating duty of: 70 %
Figure 6: McCabe-Thiele diagram for a power duty of 80%. Here, x is the mole fraction of ethanol in the liquid phase and y is the mole fraction of ethanol in the gas phase. The plot is made with Program code 1 in Appendix C - Matlab scripts.

In Table 1 the calculated theoretical steps for each boiler duty is shown.

<table>
<thead>
<tr>
<th>Boiler duty</th>
<th># of theoretical steps</th>
<th>Column efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 %</td>
<td>4.63</td>
<td>0.463</td>
</tr>
<tr>
<td>50 %</td>
<td>6.24</td>
<td>0.624</td>
</tr>
<tr>
<td>60 %</td>
<td>9.09</td>
<td>0.909</td>
</tr>
<tr>
<td>70 %</td>
<td>4.47</td>
<td>0.447</td>
</tr>
<tr>
<td>80 %</td>
<td>4.98</td>
<td>0.498</td>
</tr>
</tbody>
</table>

The column efficiency was plotted against vapor velocity in Figure 7.
Figure 7: Column efficiency plotted against vapor velocity.
The weeping point was observed using a boiler duty of 10%, while maximum power duty was not enough to reach the flooding point.

5 Discussion
In Figure 1, it is observed that the column reached an approximate steady state after about 20 minutes. After 20 minutes the composition in the distillate is relatively constant, which is an indication that equilibrium has been attained in each stage, and therefore a steady state, has been obtained. While the system is operating at total reflux and constant power duty, the equilibriums will remain constant, hence also the composition of the distillate.

From data obtained from the GC, all samples were found to have a molar fraction lower than the azeotrope of ethanol-water mixtures. The McCabe-Thiele diagrams show that to get closer to the azeotrope would require more significantly more stages in the column, since the column efficiency is already high. The column efficiency seen in Figure 7 shows no trend; therefore it is difficult to draw any conclusion if column efficiency is influenced by vapor velocity. However if the 4th point is assumed to be too low, an expected trend appears. The efficiency as a function of vapor velocity should indeed have a maximum where maximum contact between the phases is achieved. It looks like the column is most effective at 50-60% boiling power.

The weeping point was observed at 10% boiler heat duty, after which the column gave no distillate. Even at 100% boiler heat duty the flooding point was not observed, though the vapor velocity did increase substantially.
6 Conclusion
The time required to reach steady state conditions was approximately 20 minutes. The column efficiency was found to be highest with a heating duty of 60%. The weeping point was observed at 10% boiler heat duty, while the flooding point was not reached, even at 100% boiler heat duty.

References
    http://www.nt.ntnu.no/users/preisig/Repository/TKP_4110_Felles_Lab/experiment%20descriptions/DistInstruct.pdf

Symbols and abbreviations

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Unit</th>
<th>Description</th>
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<tbody>
<tr>
<td>c_i</td>
<td>mol L(^{-1})</td>
<td>Concentration of component i</td>
</tr>
<tr>
<td>D</td>
<td>m(^2)</td>
<td>Inner diameter of column</td>
</tr>
<tr>
<td>GC</td>
<td></td>
<td>Gas chromatography</td>
</tr>
<tr>
<td>IS</td>
<td></td>
<td>Internal standard</td>
</tr>
<tr>
<td>L_i</td>
<td>L s(^{-1})</td>
<td>Liquid flow at tray i</td>
</tr>
<tr>
<td>M_i</td>
<td>g mol(^{-1})</td>
<td>Molecular weight of component i</td>
</tr>
<tr>
<td>m_i</td>
<td>g</td>
<td>Mass of component i</td>
</tr>
<tr>
<td>n_i</td>
<td></td>
<td>Tray number</td>
</tr>
<tr>
<td>n</td>
<td>mol</td>
<td>The number of moles of component i</td>
</tr>
<tr>
<td>q</td>
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<td>Parameter for q-line</td>
</tr>
<tr>
<td>R</td>
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<td>Reflux ratio</td>
</tr>
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<td>L</td>
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<td>V_{tot}</td>
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</tr>
<tr>
<td>(\hat{V}_{gas})</td>
<td>L s(^{-1})</td>
<td>Volume of sample in gas phase</td>
</tr>
<tr>
<td>v</td>
<td>m s(^{-1})</td>
<td>Vapor velocity</td>
</tr>
<tr>
<td>W_i</td>
<td>L s(^{-1})</td>
<td>Bottom flow at tray i</td>
</tr>
<tr>
<td>x_i</td>
<td></td>
<td>Molefraction of component i in liquid phase</td>
</tr>
<tr>
<td>y_i</td>
<td></td>
<td>Molefraction of component i in gas phase</td>
</tr>
<tr>
<td>(\rho_i)</td>
<td>g cm(^{-3})</td>
<td>Density of component i</td>
</tr>
</tbody>
</table>
Appendix A - Calculations

A.1 Water-ethanol mixture

The mole fraction of component $i$, $x_i$, is given by:

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

where $n_i$ is moles of component $i$ and $n_{tot}$ is the total number of moles. Moles of component $i$ is given by:

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

Where $V_i$ is volume of component $i$ and $M_i$ is the molar mass of component $i$. In a mixture of only water and ethanol, moles of water is given by:

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

Where index w is for water, et is for ethanol and $V_{tot}$ is the total volume of the mixture. By applying (A.3) and (A.2) to (A.1):

$$x_{et} = \frac{V_{et} \rho_{et}}{V_{et} \rho_{et} + \rho_w(V_{tot} - V_{et})} \quad (A.4)$$

Inserting values $x_{et} = 0.11$, $\rho_{et} = 0.791 \, \text{g/cm}^3$, $M_w = 18.02 \, \text{g/mol}$ and $V_{tot} = 5800 \, \text{mL}$ the volume of ethanol is obtained as $V_{et} = 1655 \, \text{mL}$. This is the amount of pure ethanol needed. In 96% ethanol this is equal to:

$$V_{et96\%} = \frac{V_{et}}{0.96} \quad (A.5)$$

Giving $V_{et96\%} = 1724 \, \text{mL}$. The amount of water is then:

$$V_w = V_{tot} - V_{et} \quad (A.6)$$

Making the water required 4076 mL.

A.2 Conversion between mole fraction, volume fraction and mass fraction

The mole fraction of component A in a binary mixture is given as:

$$x_A = \frac{n_A}{n_{tot}} = \frac{n_A}{n_A + n_B} \quad (A.7)$$

Where $n_A$ and $n_B$ is the number of moles of component A and B respectively and $n_{tot}$ is the total number of moles. The mass fraction is of component A in a binary mixture given by:

$$\bar{x}_A = \frac{m_A}{m_{tot}} = \frac{m_A}{m_A + m_B} \quad (A.8)$$

Where $m_A$ and $m_B$ is the mass of component A and B respectively and $m_{tot}$ is the total mass of the mixture. Similarly the volume fraction of component A can be found by:
\[ \bar{\bar{x}}_A = \frac{V_A}{V_{\text{tot}}} = \frac{V_A}{V_A + V_B} \]  

(A.9)

Where \( V_A \) and \( V_B \) is the volume of component A and B respectively and \( V_{\text{tot}} \) is the total volume of the mixture. By utilizing that the mass of component A is the number of moles of component A multiplied with the molecular weight, (A.8) can be rewritten as

\[ \bar{x}_A = \frac{n_A M_A}{m_{\text{tot}}} \]  

(A.10)

Where \( M_A \) is the molecular weight of component A. Solving for \( n_A \), doing the exact same operations with respect to component B, and inserting into (A.7) yields:

\[ x_A = \frac{\bar{x}_A}{\bar{x}_A + (1 - \bar{x}_A) \frac{M_A}{M_B}} \]  

(A.11)

Here the fact that the sum of the mass fractions is equal to one. (A.11) provides the means to convert from mass fraction to mole fraction.

Rewriting (A.9) with respect to \( V_A \):

\[ V_A = \bar{x}_A V_{\text{tot}} \]  

(A.12)

By noting that the mass of component A is equal to its volume times its density (A.13) is obtained:

\[ m_A = \bar{x}_A \rho_A V_{\text{tot}} \]

\[ \Rightarrow n_A = \frac{\bar{x}_A \rho_A V_{\text{tot}}}{M_A} \]  

(A.13)

In the last step the number of moles of component A is found by dividing its mass by its molecular weight. A similar derivation can be performed with respect to B, and inserting these results into (A.7) yields:

\[ x_A = \frac{\bar{x}_A}{\bar{x}_A + (1 - \bar{x}_A) \frac{M_A \rho_B}{M_B \rho_A}} \]  

(A.14)

(A.14) enables calculation of the mole fraction when the volume fraction is known.
Appendix B - Data

The data obtained during the experiment with different power duties are summarized in Table 2.

**Table 2: Data from the experiment with different power duties, the compositions are given as volume fractions and were obtained from a GC analysis.**

<table>
<thead>
<tr>
<th>Power duty</th>
<th>Reflux rate [mL/min]</th>
<th>Temperature [°C]</th>
<th>Top composition</th>
<th>Bottom composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 %</td>
<td>55</td>
<td>78.2</td>
<td>0.912</td>
<td>0.1899</td>
</tr>
<tr>
<td>50 %</td>
<td>77.5</td>
<td>77.3</td>
<td>0.9297</td>
<td>0.1678</td>
</tr>
<tr>
<td>60 %</td>
<td>100</td>
<td>77.4</td>
<td>0.9462</td>
<td>0.1611</td>
</tr>
<tr>
<td>70 %</td>
<td>120</td>
<td>77</td>
<td>0.9073</td>
<td>0.1415</td>
</tr>
<tr>
<td>80 %</td>
<td>143</td>
<td>77.2</td>
<td>0.9167</td>
<td>0.1186</td>
</tr>
</tbody>
</table>

The data used to determine the time it took the column to reach steady state are displayed in Table 3.

**Table 3: Data obtained to determine the time it took for the column to reach steady state. The top composition is given as a volume fraction and were obtained from a GC analysis.**

<table>
<thead>
<tr>
<th>Time [min: sec]</th>
<th>Top composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>00:00</td>
<td>0.3729</td>
</tr>
<tr>
<td>05:11</td>
<td>0.8106</td>
</tr>
<tr>
<td>10:22</td>
<td>0.8552</td>
</tr>
<tr>
<td>15:13</td>
<td>0.8507</td>
</tr>
<tr>
<td>20:15</td>
<td>0.8931</td>
</tr>
<tr>
<td>25:11</td>
<td>0.9161</td>
</tr>
<tr>
<td>30:17</td>
<td>0.9116</td>
</tr>
<tr>
<td>35:20</td>
<td>0.9435</td>
</tr>
<tr>
<td>40:17</td>
<td>0.9144</td>
</tr>
<tr>
<td>45:17</td>
<td>0.9138</td>
</tr>
<tr>
<td>50:17</td>
<td>0.9241</td>
</tr>
<tr>
<td>55:10</td>
<td>0.912</td>
</tr>
</tbody>
</table>
Appendix C - MATLAB scripts

Program code 1: McCabeThiele.m

% 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 = importdata(['C:\Users\Kasper Linnestad\Dropbox\KasperTL\Felleslab\','...
    'Oldershaw\Data\VLE.txt']);
x = xy(:,1);
y = xy(:,2);
% This is given as a mass fractions, and have to be converted to mole fractions
x = x./(x+(1-x).*(46.0684/18.0153));
y = y./(y+(1-y).*(46.0684/18.0153));
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 polynomial of tenth degree, p, to fit the data
p = polyfit(x,y,10);

% Distillate composition, xd and bottom composition, xb

xd = [0.761781626 0.803175239 0.844401545 0.751246925 0.772503324];
xb = [0.067452835 0.058572555 0.055940675 0.048396637 0.039864581];

% PART 2 - Calculating and plotting each theoretical stage

for k=1:length(xd)
    xs = [];
    ys = [];
    i = 1;
    xs(i) = xd(k);
    ys(i) = xd(k);
    % A counter, i; The composition on each stage, xs(i) and ys(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 above equation for each step, and plot the stages in the McCabe-Thiele diagram
    % plot p and the operating line y = x
    fig=figure(k);
    set(fig,'Name','McCabe-Thiele','Position',[50 150, 800, 500]);
    hold on
    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');
while xs(i)>xb(k)
  xs(i+1) = fzero(f,0.5,[],ys(i));
  if xs(i+1)>xb(k)
    line([xs(i) xs(i+1)],[ys(i) ys(i)],'Color','blue','LineWidth',1.5);
  else
    line([xs(i) xb(k)],[ys(i) ys(i)],'Color','blue','LineWidth',1.5);
  end
  ys(i+1) = xs(i+1);
  if xs(i+1)>xb(k)
    line([xs(i+1) xs(i+1)],[ys(i) ys(i+1)],'Color','blue',
         'LineWidth',1.5);
  end
  i       = i+1;
end
for j=1:i-1
  if xs(j+1)>xb(k)
    plot(xs(j+1),ys(j),'-.kx','MarkerSize',15,'LineWidth',1.5)
  else
    plot(xb(k),ys(j),'-.kx','MarkerSize',15,'LineWidth',1.5)
  end
end

%Add a textbox with the number of stages required
N=i-2+(xb(k)-xs(i-1))/(xs(i)-xs(i-1));

str   ={'The number of stages required is: ', num2str(N,3)};
stbox = annotation('textbox',[0.2 0.7 0.1 0.1],
                    'String',str,...
                    'LineStyle','none');
name  = [num2str(30+10*k) '.emf'];
print(fig,name,'-dmeta');
end
Appendix D - Assignment calculations

D.1 Pycnometer

The volume of pycnometer \( i \) can be calculated by the following:

\[
V_i = \frac{W_{i,H_2O} - W_i}{\rho_{H_2O}} \tag{D.1}
\]

Where \( V_i \) is the volume of pycnometer \( i \); \( W_{i,H_2O} \) is the weight of pycnometer \( i \) filled with pure water; \( W_i \) is the weight of pycnometer \( i \) and \( \rho_{H_2O} \) is the density of pure water at 25 °C. The density of each sample can be determined by:

\[
\rho_{\text{sample,}i} = \frac{W_{i,\text{sample}} - W_i}{V_i} \tag{D.2}
\]

Where \( W_{i,\text{sample}} \) is the weight of pycnometer \( i \) filled with the sample and \( \rho_{\text{sample,}i} \) is the density sample \( i \). The composition can then be found by comparing the calculated density with values from a table from the literature. The values given in the assignment is summarized in Table 4 together with values calculated by utilization of (D.1) and (D.2). The density of water was found in Geankoplis [3].

The weight percentage of each sample was found by the data from Green and Perry [2]. The results are listed in Table 4.

The mole fraction of a binary mixture is given as:

\[
x_i = \frac{n_i}{n_{\text{tot}}} = \frac{n_i}{n_i + n_j} \tag{D.3}
\]

Where \( n_i \) is the number of moles of component \( i \); \( n_j \) is the number of moles of component \( j \); \( x_i \) is the mole fraction of component \( i \) and \( n_{\text{tot}} \) is the total number of moles. This can be rewritten in terms of mass and molecular weight which leads to:

\[
x_{\text{EtOH}} = \frac{m_{\text{EtOH}}}{m_{\text{EtOH}} + m_{\text{H}_2\text{O}}} = \frac{w\%_{\text{EtOH}}}{w\%_{\text{EtOH}} + w\%_{\text{H}_2\text{O}}} \tag{D.4}
\]

Where \( x_{\text{EtOH}} \) is the mole fraction of ethanol; \( m_{\text{EtOH}} \) is the mass of ethanol, \( M_{\text{EtOH}} \) is the molecular weight of ethanol; \( m_{\text{H}_2\text{O}} \) is the mass of water; \( M_{\text{H}_2\text{O}} \) is the molecular weight of water; \( w\%_{\text{EtOH}} \) is the weight percentage of ethanol and \( w\%_{\text{H}_2\text{O}} \) is the weight percentage of water. The molecular weights are obtained from [2]. This enables the calculation of the mole fraction of ethanol in each sample, and the result is presented in Table 4.

<table>
<thead>
<tr>
<th>Pycnometer #</th>
<th>( W_i ) [g]</th>
<th>( W_{i,H_2O} ) [g]</th>
<th>( W_{i,\text{sample}} ) [g]</th>
<th>( V_i ) [L]</th>
<th>( \rho_{\text{sample,}i} ) [gL^{-1}]</th>
<th>( w% \text{ EtOH} )</th>
<th>( x_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.9817</td>
<td>16.95955</td>
<td>16.0903</td>
<td>4.9928</td>
<td>0.8229</td>
<td>88.1 %</td>
<td>0.7433</td>
</tr>
<tr>
<td>2</td>
<td>11.77385</td>
<td>16.75745</td>
<td>16.6962</td>
<td>4.9986</td>
<td>0.9847</td>
<td>8.0 %</td>
<td>0.0329</td>
</tr>
</tbody>
</table>
Table 4 display that the mole fractions calculated by (D.4) is in great vicinity of the ones listed in the script [1]

**D.2 Vapor velocity**

The vapor velocity, \( v \), is given by:

\[
v = \frac{\dot{V}_{\text{gas}}}{A} = \frac{\dot{V}_{\text{gas}}}{\frac{\pi}{4} D^2}
\]  

(D.5)

where \( \dot{V}_{\text{gas}} \) is the volumetric flow of the gas phase, \( A \) is the cross section area of the column and \( D \) is the inner diameter of the column. In this experiment the reflux rate is given as a volumetric liquid flow, and thus has to be converted to a volumetric gas flow. This is done by first converting the liquid flow to a mole flow, then the mole flow is converted to a vapor flow using the ideal gas law.

\[
\dot{n} = \dot{n}_{\text{Et}} + \dot{n}_{W} = \dot{V}_{\text{liquid}} \frac{x_{\text{Et}} \rho_{\text{Et}}}{M_{\text{Et}}} + \dot{V}_{\text{liquid}} \frac{(1 - x_{\text{Et}}) \rho_{W}}{M_{W}}
\]  

(D.6)

\( \dot{n} \) is the molar flow rate; \( \dot{V}_{\text{liquid}} \) is the volume flow of the reflux, e.g. the reflux rate; \( x_{\text{Et}} \) is the volume fraction of ethanol; \( \rho_{\text{Et}} \) is the density of ethanol; \( \rho_{W} \) is the density of water; \( M_{\text{Et}} \) is the molecular weight of ethanol and \( M_{W} \) is the molecular weight of water. The volumetric gas flow can then be found by the ideal gas law:

\[
\dot{V}_{\text{gas}} = \frac{\dot{n}RT}{p} = \frac{\left( \dot{V}_{\text{liquid}} \frac{x_{\text{Et}} \rho_{\text{Et}}}{M_{\text{Et}}} + \dot{V}_{\text{liquid}} \frac{(1 - x_{\text{Et}}) \rho_{W}}{M_{W}} \right) RT}{p}
\]  

(D.7)

Where \( R \) is the gas constant; \( T \) is the temperature and \( p \) is the pressure. Using the data from the script [1] and inserting (D.7) into (D.5) the vapor velocity was found to be

\[
v = 0.140 \text{ m/s}
\]
Program code 2: McCabeThiele1.m

% 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 = importdata([\'C:\Users\Kasper Linnestad\Dropbox\KasperTL\Felleslab\',...
       \'Oldershaw\Data\VLE.txt\']);
x  = xy(:,1);
y  = xy(:,2);
% This is given as a mass fractions, and have to be converted to mole fractions
x  = x./(x+(1-x).*(46.0684/18.0153));
y  = y./(y+(1-y).*(46.0684/18.0153));
% 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
figure('Name','McCabe-Thiele','Position',[50 150, 800, 500]);
hold on
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', 'EastOutside');

% Distillate composition, xd and bottom composition, xb

xd = 0.744;
xb = 0.033;
%PART2 - Calculating and plotting each theoretical stage

xs = [];
ys = []; i = 1;
xs(i) = xd;
ys(i) = xd;

%A counter, i; The composition on each stage, xs(i) and ys(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 above equation for each step, and plot the stages in the McCabe-Thiele diagram

while xs(i)>xb
    xs(i+1) = fzero(f,0.5,[],ys(i));
    line([xs(i) xs(i+1)],[ys(i) ys(i)],'Color','blue','LineWidth',1.5);
    ys(i+1) = xs(i+1);
    if xs(i+1)>xb
        line([xs(i+1) xs(i+1)],[ys(i) ys(i+1)],'Color','blue','LineWidth',1.5);
    end
    i = i+1;
end

for j=1:i-1
    plot(xs(j+1),[ys(j)],'-kx','MarkerSize',15,'LineWidth',1.5)
end

%Add a textbox with the number of stages required

str={'The number of stages required is:', num2str(i-1)};

stbox = annotation('textbox',[0.2 0.7 0.1 0.1],'String',str,'LineStyle','none');

Running Program code 2 yields Figure 8 and it is obtained that five theoretical stages is required to achieve the desired concentrations.
The number of stages required is: 5

Figure 8: A McCabe-Thiele diagram for a binary mixture of ethanol and water at 1 bar. The number of theoretical stages is calculated with Program code 2.