The purpose of the Distillation Tray Efficiency at Total Reflux experiment is to determine the dependence of the Murphree tray efficiency on the vapor flow rate for a bubble cap and for a sieve tray. Distillation is a process by which two compounds are separated due to differences in their physical properties; primarily their boiling points. Water and ethanol are the two compounds used in this experiment. Based on their volatilities, the two compounds are designated as light key (ethanol) and heavy key (water), and as heat is applied through the distillation column’s reboiler, the light key is more easily vaporized. The light key then travels up the column, condenses, and is collected as distillate, whilst the heavy key travels down the column and is collected as ‘bottoms’. The distillation column is equipped with three sieve trays consisting of multiple holes, which allows for interaction between the upward flow of vapor and downward flow of liquid. A bubble cap tray is also equipped, which allows for trapping of vapor via a riser, initiating a forced interaction between the vapor and liquid. The column used operates at total reflux, therefore, no collection of the distillate or bottoms occurs, as all vapor condensed at the top of the column is fed back into the top tray and returned to the reboiler. Theoretically, the number of stages necessary for separation should decrease if the interaction between the liquid and vapor at each tray is maximized.

The characterization of this interaction can be defined by the Murphree efficiency (Eq. 1) for each stage, once it is assumed that the compositions of each tray are completely mixed. This shows the separation achieved on each tray.

\[ E_M = \frac{y_i - y_{i,n+1}}{y^* - y_{n+1}} \quad (Equation \ 1) \]

\( y_i \) is the dimensionless vapor composition at tray n, \( y_{i,n+1} \) is the dimensionless composition of the vapor at tray n+1, and \( y^* \) is the dimensionless vapor composition in equilibrium at tray n.

An energy balance around the condenser (Eq. 2) was used to determine the varied vapor flow rates and heat losses, since the power input was varied. This can then be used to completely establish the Murphree tray efficiencies for the sieve and bubble cap tray.

\[ m_{\text{vapor}} = \frac{\dot{Q}}{(H_{\text{vapor}} - H_{\text{liquid}})} \quad (Equation \ 2) \]

\( m_{\text{vapor}} \) is the mass flow rate of the vapor in lbs/hr, \( \dot{Q} \) is the heat duty of the condenser in J/hr, and \( H \) is the enthalpy of the vapor and liquid, respectively, measured in J/g.
Determination of the enthalpies of the liquid and vapor involves the creation of a hypothetical path, solved by a thermodynamic approach using the following equation. This allows for the use of vapor flow rate as a parameter, which can then be utilized for a more thorough understanding of the Murphree tray efficiency.

\[
\dot{Q} = \dot{m} c_{p,\text{liquid}} \Delta T \quad (\text{Equation 3})
\]

\(\dot{m}\) is the mass flow rate of the cooling water in g/min, \(c_{p,\text{liquid}}\) is the specific heat of liquid water in J/g*K, and \(\Delta T [\^\circ\text{C}]\) is the change in cooling water temperature between the inlet and outlet.

Finally, the overall column efficiency can be calculated using Eq. 4 by first using the graphical McCabe Thiele method\(^1\) to obtain a value for \(N_{\text{min}}\).

\[
\text{Column Efficiency} = \frac{N_{\text{min}}}{N_{\text{actual}}} \quad (\text{Equation 4})
\]

\(N_{\text{min}}\) is the minimum number of trays needed for the separation, and \(N_{\text{actual}}\) is the actual number of trays used in the column.

The distillation column’s reboiler was heated using wattages of 3860, 4960, 6070, 7170, and 8280 watts, and specific gravity and temperature measurements of each tray were acquired via hydrometers and thermocouples, respectively. 8280W was found to be the maximum power because above this, the column could not operate at total reflux. Four more data points were used to provide an even distribution of power so that an observable trend might be seen. Thermocouples were also located at the inlet and outlet of the condenser, and the input system water flow rate was controlled using a rotameter valve. Lab manual requirements and safety procedures were followed, so the reflux temperature was kept constant and was not allowed to exceed 60\(^\circ\text{C}\).\(^2\) After the system reached steady state, that is, after the reflux and thermocouple readings remained constant, all nine thermocouple readings and each hydrometer measurement was recorded. Then the wattage was changed to the next aforementioned value, water flow rate was adjusted to avoid changes in reflux temperature, and measurements were recorded once more.

When running this experiment, each team member must take safety as a top priority. Appropriate lab clothing, close toed shoes, and proper eye protection must be worn at all times. The distillation column apparatus can become very hot, so take care to avoid direct skin contact with the machine, especially when taking measurements. The hydrometer for the reflux is placed on the wall above the column, so extra caution should be used when climbing the stairs. When adjusting the power to the heater, make sure the supplied power does not go over 9000 watts, as this could cause the ethanol and water mixture to overflow from the distillation column. Ethanol is a flammable substance, so sparking from any nearby source could potentially cause a fire.

The specific gravity readings were used to calculate the compositions of ethanol in terms of mole fraction and mass, and were shown to decrease from top to bottom of the column (i.e. 73.865 ± 0.002 to 46.388 ± 0.009 mol% ethanol from tray 4 to tray 1 for 3860 W). This coincides with theoretical expectation. There was an exponential increase of liquid to vapor mole fraction mole fraction of ethanol until a liquid mole fraction of 0.2, wherein a fairly linear trend was observed. The 6\(^{th}\) order polynomial for this relationship is:

\[
y = -95.619x^6 + 277.54x^5 - 315.15x^4 + 178.27x^3 - 52.662x^2 + 8.0868x + 0.0148 \quad \text{where } R^2 = 0.9986
\]
The adjustment of the heat settings, or the increase in power, increased the superficial flow rate of vapor from $13.811 \pm 3.542 \text{ kg/(hr*ft}^2\text{)}$ for 3860 W to $77.685 \pm 8.984 \text{ kg/(hr*ft}^2\text{)}$ for 8280 W. The temperature and specific gravity of the mixture also varied as the superficial flow rate changed. The temperature ranged from $79.250 \pm 0.480^\circ\text{C}$ to $78.633 \pm 0.153^\circ\text{C}$ in the distillation column from tray 1 to tray 4. The reflux temperature was kept at a constant $19.4 \pm 0.1^\circ\text{C}$ to eliminate another variable in the enthalpy calculations. From Eq. 1, the Murphree bubble tray efficiency ranged from $65.1 \pm 7.7$ to $69.4 \pm 0.6\%$ as the superficial flow rate increased from $13.811 \pm 3.542 \text{ kg/(hr*ft}^2\text{)}$ to $49.232 \pm 4.299 \text{ kg/(hr*ft}^2\text{)}$. For the same flow rate, the Murphree sieve tray efficiency increased from $58.2 \pm 5.3\%$ to $61.9 \pm 1.4\%$. Conversely, higher flow rates decreased these efficiencies, as entrainment flooding occurred. For example, the highest flow rate of $77.865 \pm 8.984 \text{ kg/(hr*ft}^2\text{)}$ had bubble and sieve tray efficiencies of $67.5 \pm 6.8\%$ and $58.3 \pm 5.6\%$, respectively. By forming a vapor liquid equilibrium diagram, and solving for the liquid and vapor mole fractions of ethanol, the number of ideal trays was found to be 4, whereas the theoretical amount was 5. Therefore, the overall column efficiency was determined to be 80.0\%.

Error considerations arose from fluctuations in experimental readings, as some readings, such as the hydrometer readings, affect the composition values and tray efficiencies. Other sources of error arose from an unstable rotameter level and from weeping present in the sieve tray.

In conclusion, for the majority of vapor flow rates, the bubble cap tray was more efficient, and sieve tray efficiencies increased with an increasing vapor flow rate. Furthermore, the temperature decreased up the distillation column, while the mole fraction of ethanol increased up the column. Recommendations for future trials include replacing the rotameter and hydrometer readings with digital readouts to reduce the experimental error. Also, a wider variety of wattages and more trials per wattage would be preferred to ensure correct data collection.

References