Expt. MT 303

Distillation with Total Reflux

Aim
To determine the overall efficiency of the plate column, operating under total reflux for a binary system and number of transfer units for a packed column.

Theory

Overall efficiency = \frac{\text{Number of theoretical stages}}{\text{Number of actual stages}} (1)

Fenske’s equation is

\[ N_M + 1 = \frac{\log\left(\frac{x_D}{1-x_W} \frac{1-x_D}{x_W}\right)}{\log \alpha_{av}} \] (2)

\[ \alpha_{av} = \sqrt{\alpha_{top} \times \alpha_{bottom}} \] (3)

\( N_M \) = number of theoretical stages at total reflux, and \( \alpha_{av} \) = average relative volatility.

Procedure

1. Prepare a calibration chart of refractive index (RI) vs. mole fractions of components A and B.
2. Fill approximately 2/3rd volume of the distillation flask with a mixture of A and B.
3. Start water circulation through the condenser.
4. Close the bottom sample "draw out" cock to prevent the overflow of liquid through the sample line during the experiment. Open the top sample "draw out" cock to allow the air to escape when the heating is started. Switch on the heating mantle and allow the flask temperature to rise.
5. When the vapours rise to the top of the column, close the top "draw out" cock and increase the heating rate if necessary. After about 30 min, note the top and the bottom temperatures. If some milky liquid appears near the top "draw out" or the sample line, drain it in a small conical flask and discard. This could be due to the formation of emulsion of the organic liquid with moisture.
6. When the top and the bottom temperatures become steady (based on two successive readings taken at 10 min time interval) it indicates that the system has attained steady state and the samples may be withdrawn.
7. 2 to 4 ml of top sample is collected in a test-tube after purging off some quantity. The test-tube should be immediately closed to avoid losses. Determine the RI of the sample.

8. Count the number of actual plates in the column.

**Apparatus**

![Diagram of Packed Column and Tray Tower]

**Observations**

**Data from the literature**

1. Molecular weights of $A$ and $B$.

2. Refractive indices of $A$ and $B$ at ambient temperature.

3. Densities of $A$ and $B$ at ambient temperature.

MT 303-2
Calibration data for mole fraction vs. RI

<table>
<thead>
<tr>
<th>S.N.</th>
<th>Mole fraction of A</th>
<th>RI</th>
</tr>
</thead>
</table>

Observations

1. Number of actual stages in the column.
2. Top temperature (°C)
3. Bottom temperature (°C)
4. RI of top and bottom samples
5. Mole fraction of top sample ($X_D$)
6. Mole fraction of residue ($X_B$)

Calculations

1. Calculate the number of stages (theoretical) by McCabe Thiele method and calculate the overall efficiency by equation [1].
2. Calculate the number of stages by Enthalpy-Composition method and calculate the efficiency by equation [1].
3. Calculate the number of stages by Fenske’s equation (equations [2] and [3]) and calculate the efficiency by equation [1].
4. Calculate number of transfer units (NTU) for a packed column.

$$NTU = \int_{y_B}^{y_D} \frac{dy}{y^* - y}$$

5. Comment on the precaution and the sources of error.