

Sieve Tray Distillation Column

Objective

The aim of this experiment was to determine the value for stage efficiency for sieve tray column by use of x-y diagram or McCabe-Thiele plot.

Construction:

The sieve tray distillation column consisted of two long sections of glass tube (one for the rectifying section and one for the stripping section) joined together at the feed plate. It was equipped with a set of 14 sieve trays which have been fabricated of light alloy. These were joined together as a "cartridge" by means of screwed stringers so that the down comers had the right clearances. The individual plates were sealed to the column wall by means of silicone rubber "O" rings and the cartridge was inserted into the column as a unit. This method of construction is common with sieve tray column of less than 0.5 meter diameter.

The sieve trays were arranged so that their temperatures were continuously monitored by thermocouples and so that liquid samples could be taken using narrow diameter tubes which were led from the column in two bundles, one at the feed tray and one from the base of the column.

The column was equipped with an electrically heated reboiler and a glass surface condenser. The liquid draining from the condenser flew into a vertical down comer and from this into a divider. Some flew into the product tank (controlled by a valve) and the remainder was returned as reflux to the top plate of the column. The flow rates both reflux and distillate product streams were measured by rotameters.

Liquid was taken from the reboiler through a double pipe heat exchanger cooler and was pumped by a metering pump into a bottoms product tank. Below these two product tanks was a feed tank whence feed is pumped by another metering pump through a pre-heater to the column's feed plate.

The products could run back into the feed tank and mixed up so as to regenerate the feed. There were points in both the condensate line and the base plate of the column where samples of the distillate and bottoms respectively could be withdrawn for analysis through septa by means of a hypodermic needle. The advantages of this column were that it was vacuum jacketed, thus ensured negligible heat losses and constant molar overflow. As well the liquid hold-up was very small which ensured that the time taken to reach steady state be quite small, allowing useful quantities of data to be obtained in small periods of operation.

Another advantage was that the column was monitored and controlled by a flexible computer based system. This allowed the operation conditions to be altered and controlled through the keyboard, as well as the collection of temperature profile data through this medium.

The system used in this experiment was Forane-P or Arklone P [1,1,2 trichloro 2,2,1 trifluoro ethane] as more volatile component and n-hexane as less volatile component.

Theory

Hold-up in this reboiler was quite large compared to that on the trays and in the condenser/reflux divider unit. Since a reasonable range of composition in the column was needed, the initial charge should have been richer in the less volatile component: a mole fraction of around 0.75 was suitable. When the temperature profile was stable over a period of time, it was recorded and samples of the distillate and the bottoms were taken. The sample point at the bottom of the sieve plate column gave direct access to the liquid in the reboiler; the condensate from the top of the column was sampled at a point adjacent to the reflux and distillate rota meters.

The liquid samples were analyzed by the means of the gas Chromatograph.

Compositions calculated from the recorded temperature using Raoult's law according to the equation $x = (P - P^*_{lv}) / (P^*_{mv} - P^*_{lv})$

Where ,

P^*_{mv} = vapour pressures of the more volatile component ,Arklone

P^*_{lv} = vapour pressures of the less volatile components ,hexane

P is the total daily atmospheric pressure

The Antoine constants are for use in the equation $\ln P^* = A - B/(t + C)$

Where P^* is the vapour pressure of the liquid in atmospheres (1.013 bar) and t is the temperature in degrees Celsius.

Vapour pressure data and other necessary data on Arklone P (Forane-P) and n-hexane were given:

Property	n-hexane	Arklone P
Molecular weight	86	186.5
Density (kg/m ³) at 25 C°	659	1565
ΔH_v (kJ/mol)	28.9	26.4
Antoine A	9.2342	0.26479
Antoine B	2714.3	2562.45
Antoine C	225.2	229.031

Operation at Total Reflux

The vapour rate can be estimated from the heater power setting. The maximum heater power was 750W and 80% of this was set in the control program.

The Ideal gas Law gave the volumetric flow rate of the vapour and the velocity in the column was found knowing its cross-sectional area.

Bottom: $0.5\text{cm}=0.005\text{m}$ in 93 s, $D=100\text{mm}=0.1\text{m} \Rightarrow A=0.0078$
Bottom flow rate = $0.005/93 \times (\pi \times 0.1^2)/4 = 4.2 \times 10^{-7} \text{ m}^3/\text{s}$
 $t=31.56^\circ\text{C}$

Distillate: $0.5\text{cm}=0.005\text{m}$ in 122s, $D=100\text{mm}=0.1\text{m} \Rightarrow A=0.0078$
Distillate flow rate = $0.005/122 \times (\pi \times 0.1^2)/4 = 3.2 \times 10^{-7} \text{ m}^3/\text{s}$
 $t=65.57^\circ\text{C}$

Atmospheric pressure from barometer in the laboratory = 29.62 inHg
Atmospheric Pressure = $29.62 \text{ inHg} \times 25.4 = 752.3 \text{ mmHg} = 0.99 \text{ atm}$

From the temperature profile supplemented by the GLC measurements and number of peaks, compositions on the trays were assigned as following:

Mole fraction = mole ratio / (1+ mole ratio)

Feed composition mole fraction = 0.5
Top composition 3 peaks mole fraction = 0.897 lvc
Bottom composition 2 peaks mole fraction = 0.935 mvc \Rightarrow Alclone = 0.103

A value of stage efficiency that reconciles the number of stages with the observed enrichment was found. From McCabe Thiele plot the number of theoretical stages found = 7 stages

$\eta = 7/14 = 50\%$ stage efficiency

Stages in rectifying section = $7 \times 0.5 = 3.5 = 4$

From plot: intercept = $x_D / (R+1) = 0.9/(R+1)$ $R=5$ reflux ratio

Reflux flow rate at 80% heater power = 120 ml/min

$120 = V = (R+1) D$

$L_a + D = 120 \text{ ml/min}$

$D = V/(R+1)$

$D = 20 \text{ ml/min}$

Reflux ration $L = 120 - 20 = 100$

$L_a / D = R = 5$

$\ln P^* = A - B/(t + C)$

using the above table P^* for Arklone P calculated :

at $t = 31.56^\circ\text{C}$, $\ln P^* = 9.26479 - 2562.45/(31.56 + 229.031) \Rightarrow P^* = 1.3 \text{ atm}$

$x = (P - P^*_{1v}) / (P^*_{mv} - P^*_{1v})$

$x = (0.99 - 0.4925) / (1.3 - 0.4925) = 0.61$

Continuous feed

From the temperature profile supplemented by the GLC measurements and number of peaks, compositions on the trays were assigned as following:

Set point 3 ,feed pump was set to 25 s, heater 80 % 600 W

Feed composition mole fraction = 0.5

Bottom composition 2 peaks mole fraction = $35.71/178.92=0.136$ mvc , $t= 64.84$ °C

Top composition mole fraction= 0.895 , $t=31.28$ °C

Set point 3 feed pump was set to 25 s, heater 85% 637.5 W

Feed composition 3 peaks mole fraction = $155.89/120.89= 0.44$ mvc

Bottom composition 2 peaks mole fraction = $1555.09/38.86= 0.175$ mvc , $t= 65.39$ °C

Top composition mole fraction= $204.79/23.8685= 0.903$ mvc , $t=31.06$ °C

Q-Which method do you think would be more accurate, using Raoult's Law or the experimental results.

Raoult's law would be more accurate.

Q-Was there any observable effect of vapour velocity in the column on the stage efficiency and if so explains.

Efficiency was calculated to be 50%. Vapour velocity will increase as the stage efficiency increases.

Conclusion

From McCabe Thiele plot a value of stage efficiency that reconciles the number of stages with the observed enrichment was found . Vapour velocity and stage efficiency had direct relationship and linearly were proportional. By recording the temperature and use of Raoult's law ,the compositions of components could be found. And Raoult's law calculations are more reliable.

Human error in taking measurements contributed in the inaccuracy of the results.