Batch Distillation

Gavin Duffy
School of Electrical Engineering Systems
DIT Kevin St.

Learning Outcomes

After this lecture you should be able to….

- Describe batch distillation
- Define and use the Rayleigh equation
- Explain batch distillation with constant product
- Explain batch distillation with constant reflux

Introduction

In batch distillation a fixed amount of charge is added to the still.

Top product composition varies with time. It depends on bottom product composition, number of trays and reflux ratio.

There is no steady state – compositions are changing with time

At start, top product is rich in MVC.

After time, top product becomes less rich in MVC.

A batch column is like the top half of a continuous column – it has a rectifying section only

The Batch Column

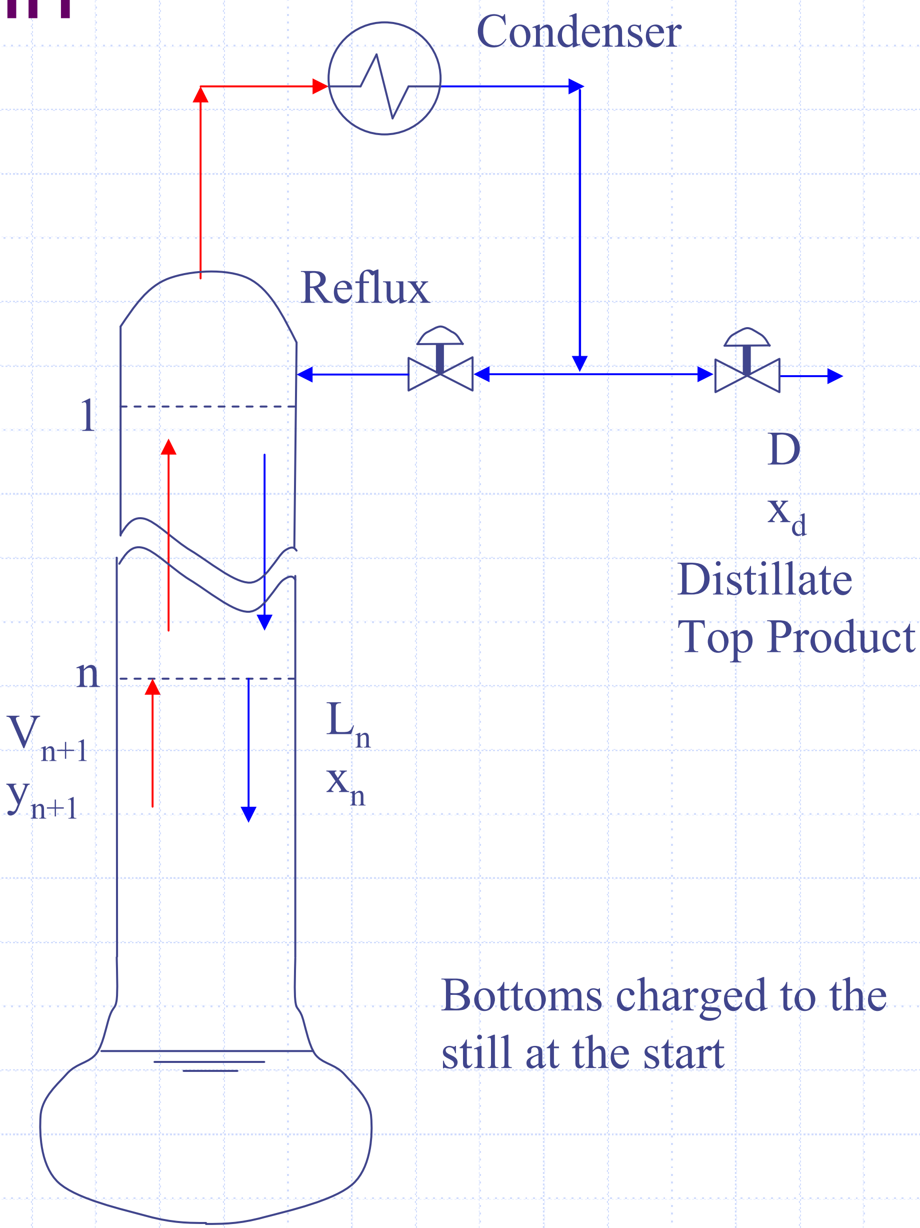
A portion is returned to the column as reflux

Remainder is removed as Distillate or Top Product

Reflux Ratio = Reflux/Distillate

Same operating line

$$y = \frac{R}{R+1}x + \frac{1}{R+1}x_d$$



Advantages

Separation of small quantities of mixtures, i.e. capacity too small to justify continuous separation

Flexibility to handle different feedstocks to produce different products

More than one product may be obtained – light components are removed first.

Different purities of the same component can also be obtained.

Upstream is batch operated and composition of feed varies with time

Fouling is a serious concern

Seader, J.D. & Henley, E.J. (2006), *Separation Process Principles*, Wiley, p466

Mass Balance

An overall mass balance for the batch distillation is as follows:

$$n_0 = n + D$$

Where n_0 = no. of moles in still at start

n = no. of moles in still at end

D = no. of moles in distillate

In all cases, the no. of moles is of all components.

Rayleigh Equation

Imagine a batch is heated and the vapour formed is removed immediately from the system without any reflux – a very simple distillation.

This was analysed by Rayleigh who developed the following equation:

$$\ln\left(\frac{n}{n_0}\right) = \int_{x_w}^{x} \frac{dx}{y-x}$$

Where n_o = initial no. of moles in still
n = no. of moles left in still at time t
x = liquid mole fraction of MVC at time t
y = vapour mole fraction of MVC at time t
x_w = liquid mole fraction of MVC in feed (t=0)
x_f = vapour mole fraction of MVC at time t

Rayleigh Equation contd.

Input to the equation

- Initial number of moles in the still (of both components n_0)
- Initial mole fraction of the MVC (x_w) in the still
- Final mole fraction of MVC in the still (x)

The results we get are:

- The total number of moles left in still (n)
- The number of moles of each component left in still
- The number of moles of each component in distillate

To solve Rayleigh

1. We can use a graphical method of integration

2. We can also use the relationship between y and x from relative volatility as follows:

$$y = \frac{\alpha x}{1 + x(\alpha - 1)}$$

Add this to the equation and integrate to give:

$$\ln \frac{n_o}{n} = \frac{1}{\alpha - 1}\left(\ln \frac{x_o}{x} + \alpha \ln \frac{1 - x}{1 - x_o} \right)$$

Activity – Use Rayleigh

A batch of crude pentane contains 15 mole percent butane and 85 mole percent pentane. It is added to a still and heated at atmospheric pressure. How many moles are left in the still when the remaining charge in the still is 97% pentane?

Initial conditions – work on the basis of 1 mole, i.e. n_o = 1 mole. x_o = 15% = 0.15

At the end, x = 3% = 0.03. What is n, the number of moles left in the still?

Alternative to Rayleigh

An alternative to the Rayleigh equation is described in McCabe Smith, 6th Ed., pp 700 to 701. The following equation is derived:

$$\frac{n_B}{n_{0B}} = \left(\frac{n_A}{n_{0A}}\right)^{1/\alpha_{AB}}$$

Where n_B = no. of moles of B (LVC) at end
n_{0B} = no. of moles of B (LVC) at start
n_A = no. of moles of A (MVC) at end
n_{0A} = no. of moles of A (MVC) at start
α_{AB} = relative volatility

See example 21.9 in McCabe Smith. It is similar to the previous problem. Instead of specifying a final mol fraction, a final no. of moles of A are given.

Reflux in Batch Distillation

In batch distillation the top product composition changes with time. What do we do if we want a constant top product composition?

There are two options for reflux in batch distillation.

1. Increase the reflux ratio with time to keep the product concentration constant. Low reflux initially; high reflux towards the end.
2. Use a fixed reflux ratio. Operate the still until the top concentration falls below a setpoint.

Temperature can be used to determine when the top concentration has reached the setpoint.

Constant Product *Variable Reflux*

To maintain a constant product composition, the reflux ratio is increased from a low value initially to a large value at the end.

High reflux means that a lot of heat is needed. The distillation should be stopped once a chosen reflux ratio is exceeded.

The amount of distillate removed and material left behind is given by the following:

$$D = n_0\left(\frac{x_0 - x_B}{x_D - x_B}\right)$$

Where

x_0 = mol fraction of MVC in still at the start

x_B = mol fraction of MVC in still at the end

x_D = mol fraction of MVC in distillate (constant!)

n_0 = no. of moles in still at start

D = no. of moles of distillate removed

$$n = n_0\left(\frac{x_D - x_0}{x_D - x_B}\right)$$

Where

x_0 = mol fraction of MVC in still at the start

x_B = mol fraction of MVC in still at the end

x_D = mol fraction of MVC in distillate (constant!)

n_0 = no. of moles in still at start

n = no. of moles left in still

Design approach Constant Product

Choose the top product composition, x_D, and the still composition x_0. Get the x-y graph ready.

If you don't have a number of ideal stages (i.e. new design) then choose the initial reflux ratio, draw the rectifying operating line and step from x_D down to meet x_0 to give no. of stages, N.

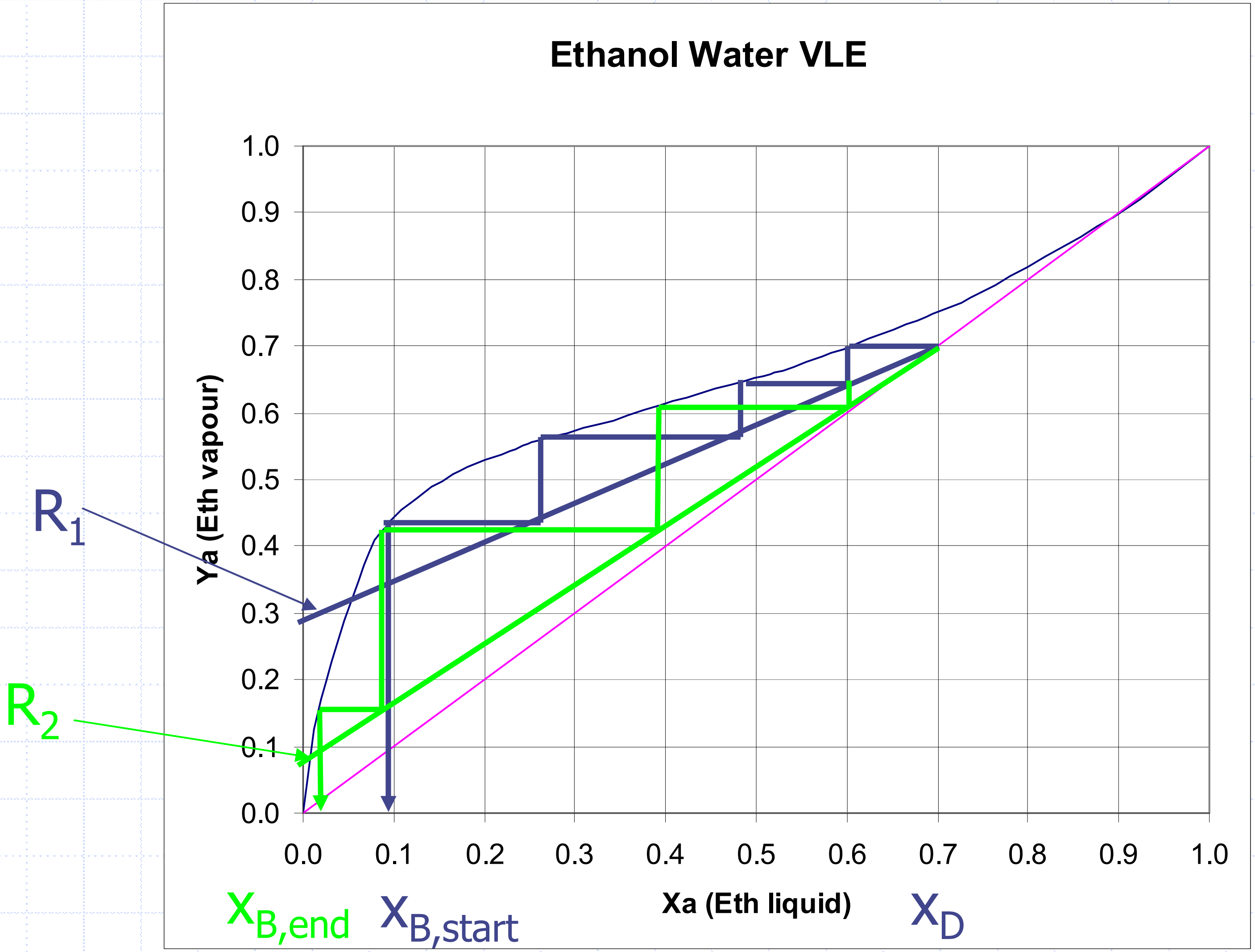
If you have N already, then use it to determine $R_{initial}$.

Choose an upper reflux ratio. Draw another operating line using the same x_D. Step off N stages to give x_B, the final still composition.

The still composition is changing – less and less of the MVC. R is increased with time but no. of stages is fixed so distillation must be stopped when bottoms composition drops to a minimum value

Figure out how to measure composition real time!

Constant Product



Must stop at $x_{B,end}$ or x_D will start decreasing

Constant Reflux *Variable Product*

The other option is to leave reflux fixed and work around a variable product composition, x_D. Simple solution.

x_D varies from x_{D1} at the start to x_{D2} at the end.

An equation very similar to Rayleigh's can be used:

$$\ln\left(\frac{n_o}{n}\right) = \int_{x_B}^{x_0} \frac{dx}{x_D - x}$$

Where n_0 = no. of moles in still at start

n = no. of moles in still at end

x_0 = mol fraction of MVC in still at the start

x_B = mol fraction of MVC in still at the end

x_D = mol fraction of MVC in distillate (a variable)

Design approach Constant Reflux

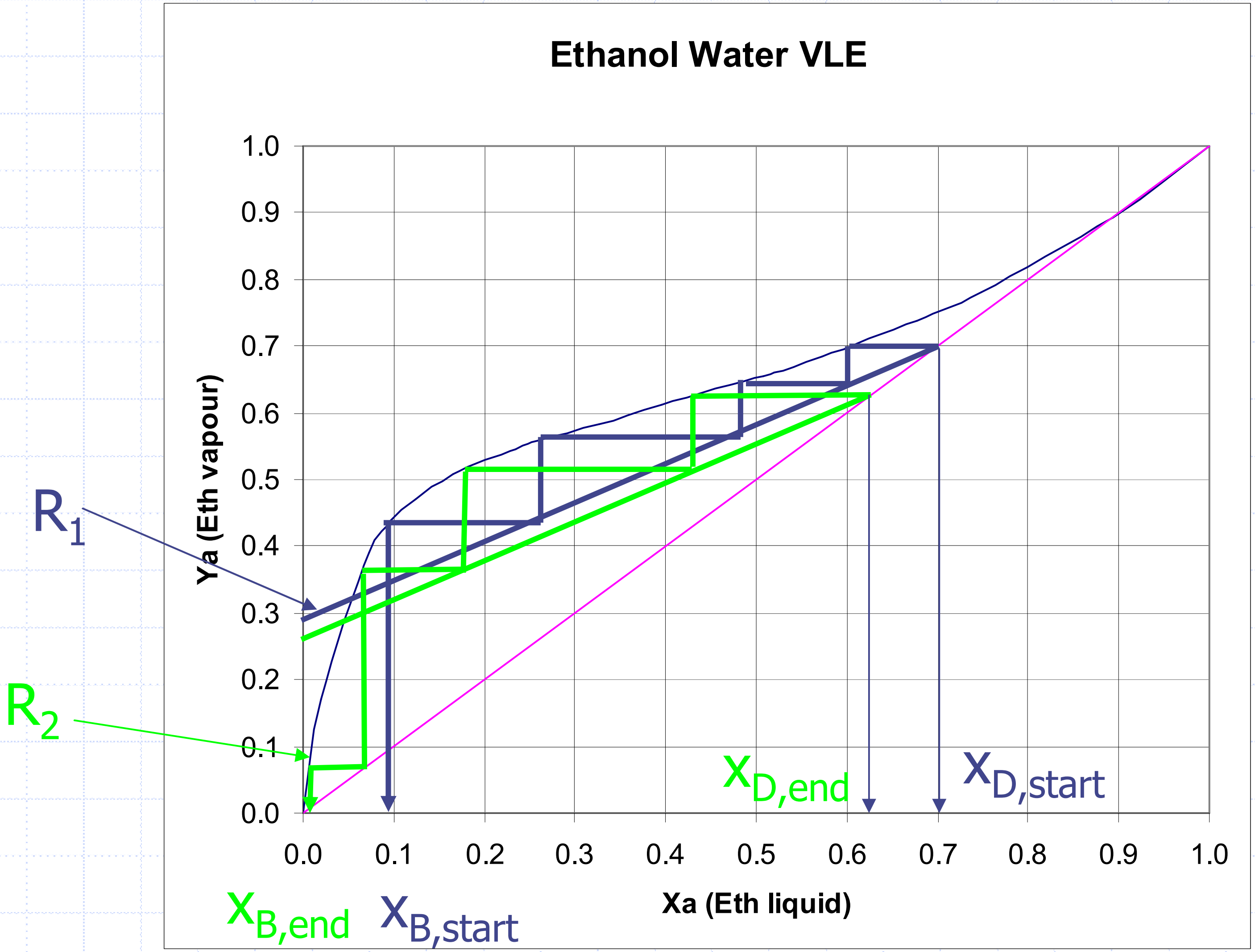
Choose the top product composition, x_D, the initial still composition x_0, and the final still composition x_B. Get the x-y graph ready.

If you don't have a number of ideal stages (i.e. new design) then choose the reflux ratio, draw the rectifying operating line and step from x_{D1} down to meet x_0 to give no. of stages, N.

If you have N already, then use it to determine R.

Redraw the operating line with the same slope. The objective is to draw the operating line in the right place such that when N stages are stepped off, the last step hits x_B exactly. Where the operating line started gives x_{D2} (where it crosses the x=y line).

Constant Reflux



Must stop at $x_{B,end}$ or x_D will start decreasing

Constant Reflux

The average mole fraction of the MVC in the distillate is given by:

$$x_{D,avg} = \frac{n_0 x_0 - n_t x_{B_t}}{n_0 - n_t}$$

Where n_0 = no. of moles in still at start
x_0 = mol fraction of MVC in still at start
n_t = no. of moles in still at time t
x_{Bt} = mol fraction of MVC in still at time t

Constant Reflux

Distillation time can be calculated if boilup rate is known:

$$time = \frac{R+1}{V}\left(n_0 - n_t\right)$$

Where R = reflux ratio

V = boilup rate (if kmol/hr then time in hr)

n0 = no. of moles in still at start

n_t = no. of moles in still at time t