University of Baghdad

College of Engineering

Department of
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Experiment of Packed Distillation Column

Batch Distillation Column

for 4th Class
Department of Chemical Engineering

by

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1. Introdution

Distillation is the process of separating mixtures into their components by selective boiling and condensation. In the chemical process industry, distillation is widely accepted for product separation, purification, and waste removal. "Distillation" itself comes from the Latin word "distillare," which means to trickle down or drip. As a result, vapor is created by heating a mixture, then cooled and condensed back into liquid form, allowing the components to be separated based on their differences in volatility. Thousands of years ago, distillation was one of the earliest and most fundamental separation techniques. Historically, simple distillation techniques were used to purify water and produce alcoholic beverages in ancient civilizations such as Mesopotamia, Egypt, and China.

There is evidence of distillation dating back to ancient Mesopotamia (circa 3500 BC), where pottery vessels containing distilled alcohol were discovered. Historically, primitive stills were used to produce alcoholic beverages such as beer and wine. In addition, archaeological findings suggest that distillation was used for medicinal and perfume purposes. Distillation, a centuries-old process, has morphed from a simple method of purifying water and producing alcoholic beverages into a versatile separation technique indispensable across myriad industries. Its enduring legacy continues to shape the modern world and contribute to scientific and technological advancements. In the 20th century, further advancements in distillation technology, including the introduction of continuous distillation processes and advanced control systems, transformed the field of separation science. Today, distillation remains a cornerstone of various industries, including oil and gas, pharmaceuticals, food and beverages, and more.

Depending on the type and quantity of chemicals involved, the distillation process can be classified into two types: (1) batch distillation, primarily used in industries like specialty chemicals, biochemicals, and pharmaceuticals; and (2) continuous distillation, widely used in petrochemical and bulk chemical industries (see Figure.1).
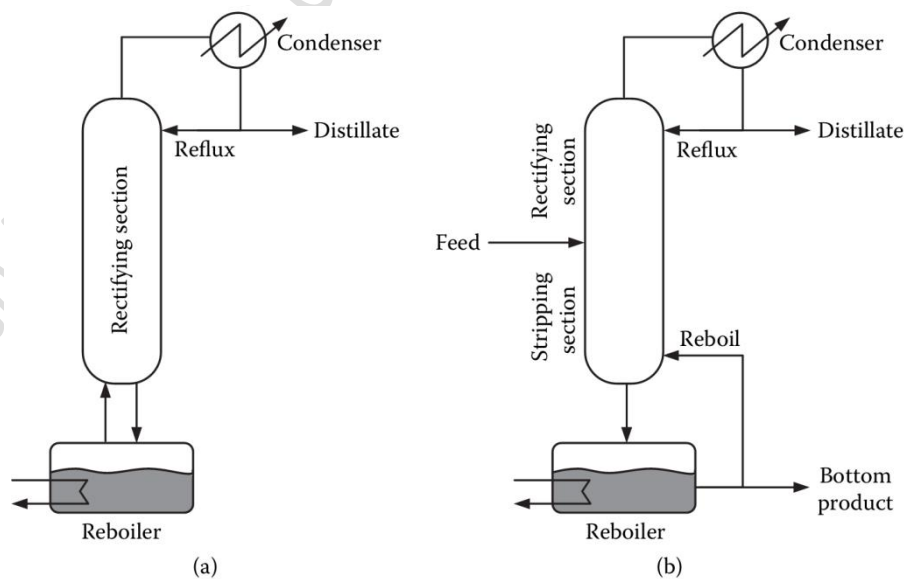
Figure.1: Types of Distillation Processes **(a)** Batch Distillation, and **(b)** Continuous Distillation

The process of batch distillation involves separating mixtures by taking advantage of differences in volatility. The separation is done in batches in batch distillation columns, which allow the distillation of a finite quantity of feed mixture in a batch-wise manner, as opposed to continuous distillation in which feed is continuously incorporated and products are continuously removed. Batch distillation is a common unit activity in small-scale manufacturing. When separating tiny amounts of high-technology/high-value-added chemicals and biochemicals, batch distillation is preferred to continuous distillation. The most distinguishing aspect of batch distillation is its versatility. This adaptability enables you to deal with uncertainties in feed stock or product specification. In addition, adjusting the column's operating parameters is a straightforward technique for dealing with many combinations. Batch distillation uses the least amount of capital to separate relatively pure components. Continuous distillation often needs a separate column for each component.

2. The Principle of Batch Distillation

The principle of batch distillation relies around separating components in a liquid mixture depending on their boiling points. This procedure is carried out in batches, which means that only a finite amount of the mixture is fed into the distillation equipment for each run. A traditional batch distillation column's operation may be divided into three phases: (1) startup, (2) production, and (3) shutdown. Before starting, the feed is typically charged to the reboiler and heated. During the starting time, the system is run in total reflux mode, which means no product is withdrawn from the top, until either a steady state is reached or the distillation composition product reaches the necessary purity. The production phase is often the longest, although for challenging separations such as high purity or azeotropic separations, the starting time might be important. For batch distillation operations to be optimized, it is important to understand these distinct phases and their specific tasks. The distillation column can be operated efficiently and safely if operators carefully control each phase and its parameters.

In operation, chemicals are charged into the reboiler vessel and evaporated in the thermosyphon reboiler through the charge port, controlled by the valve. With temperature indicators, the boiling liquid can be measured, and a valve allows it to drain. A series of bubble-cap plates at the bottom of the distillation column are used to capture vapours from the reboiler. Temperature indicators are used to measure the liquid and vapour temperatures across each plate. As vapors leave the top bubble-cap plate, they are completely condensed to liquid distillate in an overhead condenser. Samples of distillate can also be taken through a valve, which controls the reflux ratio. The reflux ratio is determined by how much reflux is present in the product. Upon cooling the product distillate in the product cooler, it can either be collected in the distillate receiver for batch rectifying or returned to the reboiler as an enricher with a constant reboiler composition for enrichment. Volumetric flow rate can be measured using a graduated cylinder positioned below the product cooler in the distillate line. Flow control valves control the flow rate of chilled water to the condenser and product cooler, and flow meters measure its flow rate. A pressure indicator measures the water pressure, while temperature indicators measure the inlet and outlet temperatures. The operating pressure under vacuum is controlled by the pressure control valve and monitored by the pressure indicator. The distillate receiver is designed to remove product when operating under vacuum.

3. Batch Distillation Apparatus

Figure.2 represents the batch distillation apparatus (QVF teaching systems-CST1 distillation packed column) manufactured by De Dietrich Process Systems Ltd, and Figure.3 shows a schematic diagram of the components of this apparatus.
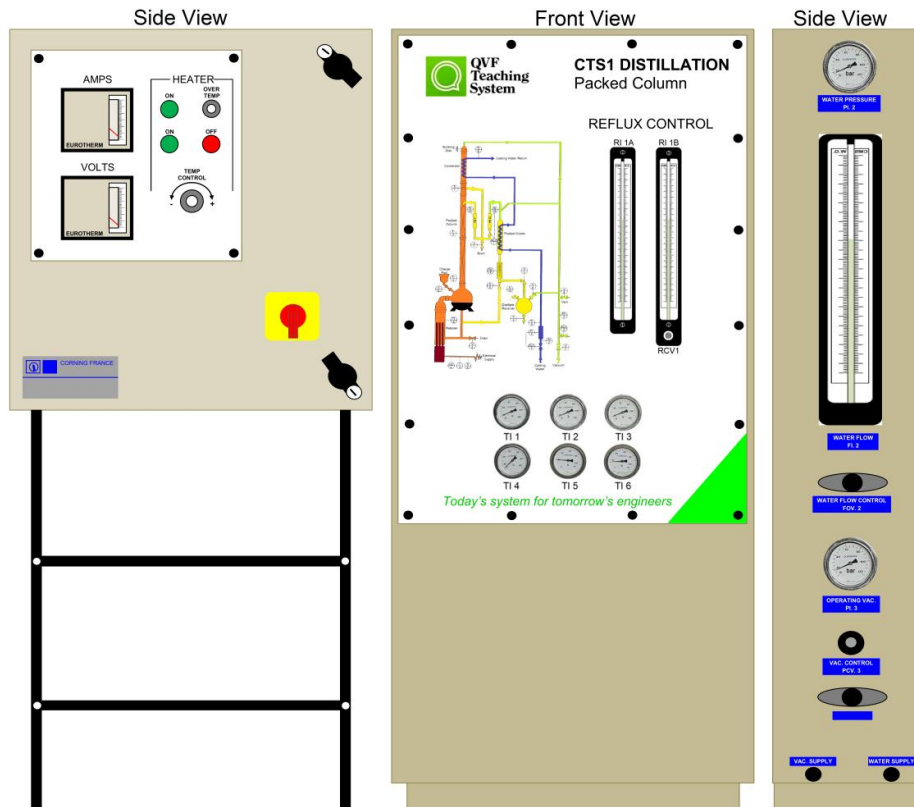
Figure.2: Batch Distillation Apparatus

The parts of the apparatus can be explained as follows:

Part.1: Reflux control flow meter (**RI**), where the vapours that leave the top of either column and are totally condensed to liquid distillate in the overhead condenser are passed to this meter. A reflux control flow meter is divided into two types:

1. **RI.1A:** Reflux flow meter, indicates the quantity of distillate returned to the column as reflux. Variable area type, range 0-7.5 l/min.
2. **RI.1B:** Product distillate flow meter, indicates the quantity of distillate removed as product. Variable area type, range 0-7.5 l/min.
3. **RI.1C:** Product distillate flow meter. It is a graduated tube calibrated in millimetres.

Part.2: Cooling water flow meter (**FI**), variable area type, range 0-15 l/min.

Part.3: Pressure indicator (**PI**), which is divided into:

1. **PI.2:** Water pressure indicator. Bourdon type, range 0-4 bar gauge.
2. **PI.3:** Vacuum pressure indicator. Bourdon type, range -1-0 bar gauge.
3. **PI.4:** Steam pressure gauge. Bourdon type, range 0-1 bar gauge.
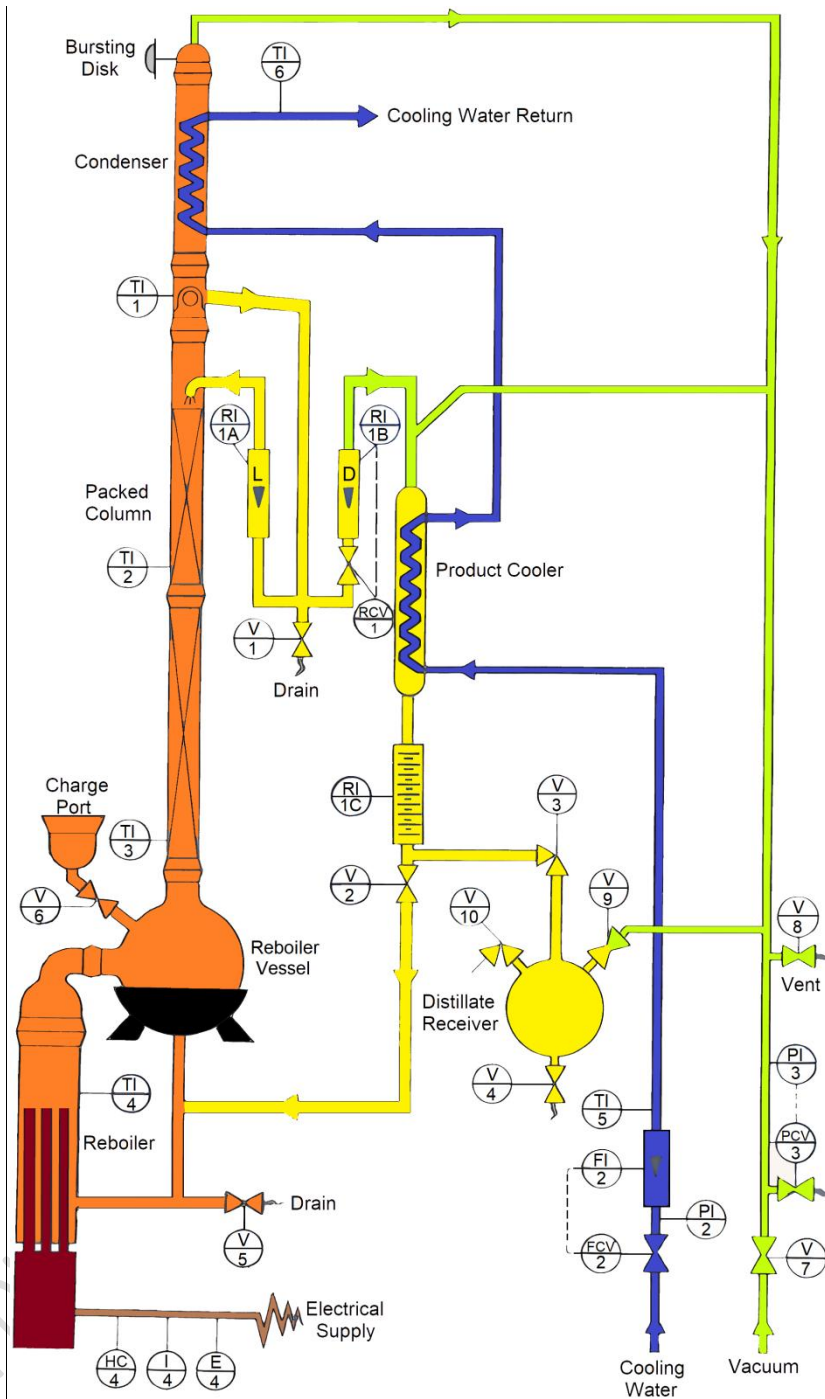
Figure.3: Schematic Diagram of the Components of the Batch Distillation Apparatus

Part.4: Temperature indicator (**TI**), which is divided into:

1. **TI.1:** Measure the vapour temperature leaving the packed column.
2. **TI.2:** Measure the vapour temperature inside the packed column.
3. **TI.3:** Measure the vapour temperature entering the packed column.
4. **TI.4:** Measure the temperature of the boiling liquid.
5. **TI.5:** Measure the inlet temperature.
6. **TI.6:** Measure the outlet temperature.

Part.5: Valves (V), which includes:

1. **RCV.1:** Reflux control valve, which used to control the ratio of reflux to product, and the reflux ratio.
2. **V.1:** Rotameter drain valve.
3. **V.2:** Distillate flow valve.
4. **V.3:** Distillate flow valve (distillate receiver).
5. **V.4:** Distillate receiver drain valve.
6. **V.5:** Reboiler drain valve.
7. **V.6:** Charge port valve.
8. **V.7:** Vacuum isolate valve.
9. **V.8:** Vent valve
10. **V.9:** Vacuum isolate valve (distillate receiver).
11. **V.10:** Vent valve.
12. **V.11:** Steam trap by-pass valve.
13. **PCV.3:** Steam pressure control valve.
14. **FCV.2:** Water flow control valve.

Part.7: Heater controller (HC.4).

Part.8: Ammeter (I).

Part.9: Voltmeter (E).

The distillation packed column has four ideal plates with an 80 mm diameter and a 1500mm packed height, and the size of the glass raschig rings (packing) is 10mm.

4. Working Procedure

The working procedure includes the following steps:

1. It is important to ensure that the feed concentration in the reboiler vessel is correct, and that the liquid level is at the equator of the vessel (the parallel lines as appeared in Figure. 4). The total working capacity of the reboiler system is (28 litres), which is divided between the vessel and the heater, where the working capacity of the spherical vessel is (13 Litres). The ratio of water to ethanol is (70/30).
2. Before starting the operation, you must ensure that:

   a. The following parts are must be closed: V.1, RCV.1, V.3, V.4, V.5, V.6, V.7, V.9, FCV.2, and PCV.3.
   b. Also, before starting operation, V.3 valve and the cooling water supply port must be open, where the system must contain the required amount of water. Water flow is 4-6 litres per minute.

Figure. 4: Equator of Reboiler Vessel

3. After we have made sure that all the necessary steps that must be taken before operation have been completed, we turn on the system and wait until the liquid begins to boil.

4. Over time, the liquid continues to boil, and the ethanol vapor begins to rise. Meanwhile, we continue to monitor the temperature gauge (TI.1) until it stabilizes.

5. When the temperature gauge (TI.1) stabilizes, where it will record a temperature higher than 80 °C, we open the valve (RCV.1).

6. We balance the valve (RCV.1) that controls the reflux flow (RI.1A) with the product distillate flow (RI.1B) until we get the (L/D) ratio equal to one.

7. When we obtain the exact balance ratio of (L/D), we close valve (V.2) and open valve (V.3).

8. Distilled ethanol begins to flow into the distillate receiver vessel, and when we obtain the appropriate amount of it, we close valve (V.3) and open valve (V.2) again.

9. Steps 7 and 8 are repeated three additional times every quarter hour, while monitoring the temperature gauge (TI.1) and maintaining the set balancing ratio (L/D).

10. We measure the voltage and current of the distillation process.

11. Finally, we close the system after completing all four measurements.

6. Results Analysis

After collecting the four distilled batches,, we calculate the mass of each batch using a pycnometer. The calculation steps can be summarized as follows:

1. We record the mass of the pycnometer (M_p) while it is empty, which will be constant and used in the calculations of all batches.

2. The next step is to fill the pycnometer with the distilled liquid (ethanol) and record the mass (M_{pL}).

3. To calculate the mass of the liquid (M_L), we subtract the mass of the empty pyrometer from the mass of the pyrometer filled with liquid.

$$M_L = M_{PL} - M_P \qquad (1)$$

4. Since the volume of the pycnometer (V_P) is known (written on it), the density (ρ) of each batch can be calculated.

$$\rho = \frac{M_L}{V_P} \qquad (2)$$

We can list the results obtained in a table as shown below in the Table.1.

Table.1: Calculations of Batches Mass

Batch No.	Mass of pycnometer, g	Mass of pycnometer + Liquid, g	Mass of Liquid, g	Density, g/cm^3	Current, Amp.	Voltage, Volts
1	M_p	M_{pL1}	M_{L1}	ρ_1		
2	M_p	M_{pL2}	M_{L2}	ρ_2	I	V
3	M_p	M_{pL3}	M_{L3}	ρ_3		
4	M_p	M_{PL4}	M_{L4}	ρ_4		

4. Calculation of Batch Distillation Experiment

The case of a column with four ideal plates used to separate a mixture of ethanol alcohol and water may be considered. Initially there are S_1 moles of alcohol of mole fraction xs_1 with respect to the more volatile component (MVC), alcohol, in the still. The top product is to contain a mole fraction x_d, and this necessitates a reflux ratio R_1. If the distillation is to be continued until there are S_2 moles in the still, of mole fraction xs_2, then, for the same number of plates the reflux ratio will have been increased to R_2. If this column is operated at a constant reflux ratio R, the concentration of the more volatile component in the top product will continuously fall. Over a small interval of time dt, the top-product composition with respect to the more volatile component will change from mole fraction x_d to x_d+dx_d, where dxd is negative for the more volatile component. If in this time the amount of product obtained is dD_b, then a material balance on the more volatile component gives:

$$ln\frac{S_1}{S_2} = \int_{x_{s2}}^{x_{s1}} \frac{dx_s}{x_d - x_s} \tag{3}$$

Where:
S_1: initial mole fraction of water plus ethanol
S_2: remaining mol fraction at time t.
x_d: mole fraction of MVC in vapour (distilled).
x_s: mole fraction of MVC in liquid (still).
1/xd-xs: area under curve.

This formula is known as the Rayleigh equation.

The mole fraction (x_d) is calculated from the masses of the batches obtained from the experiment. The below equation can be used to convert the weight fraction (wt.%) to the mole fraction of ethanol:

$$x_d = \frac{\dfrac{M_{Lethanol}}{mw_{ethanol}}}{\dfrac{M_{Lethanol}}{mw_{ethanol}} + \dfrac{M_{Lwater}}{mw_{water}}} \tag{4}$$

Where:
$mw_{ethanol}$: Molecular weight of ethanol.
mw_{water}: Molecular weight of water.
$M_{Lethanol}$: Calculated mass of ethanol.
M_{Lwater}: Calculated mass of water, which is equal to:

$$M_{Lwater} = 1 - M_{Lethanol} \tag{5}$$

The Table.2 represents the physical properties of water and ethanol.

Table.2: Physical Properties of Water and Ethanol

| Property	Water	Ethanol
Chemical formula	H_2O	C_2H_6O
Molecular weight	18	46
Boiling temperature, T_n	100 ºC, 373.15 K	78.35 ºC, 351.5 K
Critical temperature, T_c	647	514 K
Critical Pressure, P_c	220.64 bar	63 bar
Density, ρ	1000 kg/m^3	789 kg/m^3
Latent heat of vaporization	40650 J/mole, 2256 J/g	42300 J/mole, 846 J/g

The McCabe-Thiele graphical construction method (see Figure.5) can be used to calculate mole fraction of MVC in liquid (x_s) as follows:

1. We plot the intercept point on the y-axis. The intercept can be calculated from the following equation:

$$\text{Intercept} = \frac{x_d}{R + 1} \qquad (6)$$

2. We draw the xd1 point on the inclined line at an angle of 45º.
3. We connect the two points with a straight line.
4. From the point of x_{d1}, we draw straight lines that are equal in number to the ideal plates (4).
5. We project the last line onto the x-axis, and the intersection point represents the x_{s1}.

These steps are repeated for all batches (x_{d1} to x_{d4}), as shown in Figure.5,b.

(a)

(b)
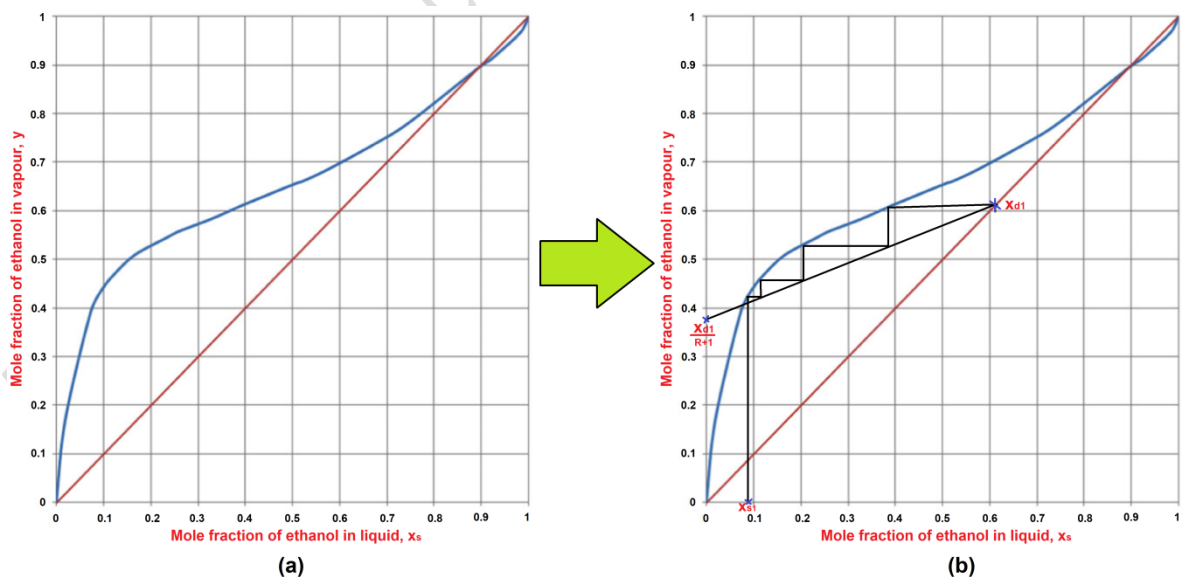
Figure.5: McCabe-Thiele Equilibrium Diagram for Ethanol-Water (y is vapor-phase mole fraction)

We list all the data we obtained for each xd, xs, and 1/(xd-xs) in the Table.3.

<p align="center">**Table.3:** xd, xs, and 1/(xd-xs) values</p>

xd	xs	1/(xd-xs)
xd_1	xs_1	$1/(xd_1-xs_1)$
xd_2	xs_2	$1/(xd_2-xs_2)$
xd_3	xs_3	$1/(xd_3-xs_3)$
xd_4	xs_4	$1/(xd_4-xs_4)$

Now, and depending on the results from Table.3, we will calculate the area under the curve as shown in the Figure.6 to extract the two terms of integration (x_{s1} and x_{s2}) in Equation.3.
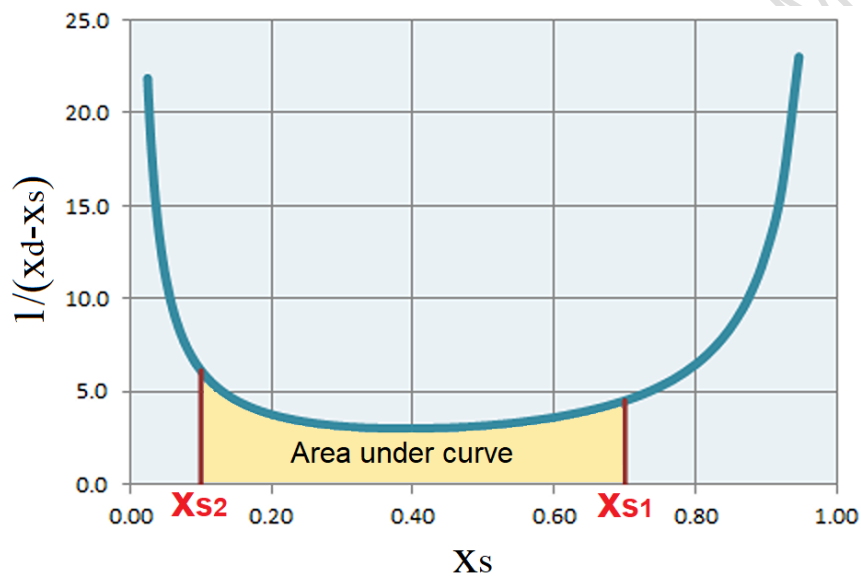
<p align="center">**Figure.6:** Calculation of the Area Under Curve</p>

The reflux ratio (R) can be calculated from the following equation:

$$R = \frac{L}{D} \tag{7}$$

Where:
L: Liquid return from the condenser to the batch reactor (refluxed liquid).
D: Distillate, which is calculated from the following equation:

$$D = S_1 - S_2 \tag{8}$$

The next step, using the Riedel equation to calculate the enthalpy of vaporization:

$$\frac{\Delta H_n}{\bar{R} T_n} = \frac{[1.092(lnP_c - 1.013)]}{(0.93 - T_{rn})} \tag{9}$$

Where:
ΔH_n: Molar enthalpy of vaporization, also called molar latent heat of vaporization-λ, (J/mol).
$\bar{R}$: Gas constant, which equal to (8.314 J·K^{-1}·mol^{-1}).
T_n: Boiling temperature, (K)
P_c: Critical pressure of the substance.
The constants 1.092, 1.013, and 0.93 are specific to the Riedel equation.
T_{rn}: Reduced temperature, (K) which can be calculated from the following equation:

$$T_{rn} = \frac{T_n}{T_c} \tag{10}$$

Where:
Tc: Critical temperature, (K).

Since we have a mixture of liquids (ethanol and water), the boiling temperature (T_n) and critical temperature (T_c) are taken for the mixture (T_{nmix}, T_{cmix}) and calculated from the following equation:

$$T_{nmix} = (T_{nethanol} \times xd_{Eavr}) + (T_{nwater} \times xd_{Wave}) \tag{11}$$

$$T_{cmix} = (T_{cethanol} \times xd_{Eavr}) + (T_{cwater} \times xd_{Wavr}) \tag{12}$$

By the same way, we can calculate the critical pressure for the mixture (P_{cmix}):

$$P_{cmix} = (P_{cethanol} \times xd_{Eavr}) + (P_{cwater} \times xd_{Wavr}) \tag{13}$$

The xd average (xd_{Eavr}) of ethanol can be calculated from the following equation:

$$xd_{Eavr} = \frac{xd_1 + xd_2 + xd_3 + xd_4}{4} \tag{14}$$

The xd average (xd_{Wavr}) of water is calculated from the equation below:

$$xd_{Wavr} = 1 - xd_{Eavr} \tag{15}$$

The heat to be supplied to provide the reflux (Q_R) during the total distillation (total reboiler heat duty) in joules is:

$$Q_R = Q_C + DH_D \tag{16}$$

Where:
Q_C: Heat removed from condenser (W).
H_D: Enthalpy of distillate (J/mol).

And:

$$Q_R = \lambda RD \tag{17}$$

Where:
λ: Molar latent heat of vaporization of the mixture.

$$Q_C = (L + D)\lambda \tag{18}$$

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