

## Continuous rectification on a pilot-scale equipment

### 1. Introduction

Expressions: equilibrium stage = theoretical stage = theoretical plate

In case of flash distillation (distillation without reflux rate) the separation takes place on one equilibrium stage. The separation is limited by the equilibrium between the components.

The concentration of the volatile compound can be increased by increasing the number of the equilibrium stages. The vapor leaving the first stage is lead into the second stage, so the volatile compound enriches. If more stages are connected, higher concentration of the volatile compound can be reached. In practice it means that the vapor leaving the first stage is partly condensed and on the second stage the liquid-vapor equilibrium develops.

The volatile compound content of the liquid leaving the first stage can be reduced by partial evaporation. The vapor formed due to the partial evaporation has lower volatile compound content than the vapor leaving the first stage. Figure 1 shows the principle of partial-evaporation and -condensation. The initial concentration is  $x_{F1}$ , which is fed onto the first stage. After the heat transfer, the liquid evaporates and equilibrium between the vapor and liquid forms. The liquid phase on the first stage has a concentration of  $x_1$ . This phase is led onto the second stage. After the heat transfer, equilibrium forms on the second stage which provides a reduced volatile compound content in the liquid phase. On the other hand, the vapor leaving the first stage has a concentration of  $y_1$ . This vapor is partly condensed, i.e. it is the feed of the next stage ( $x_{F2}$ ). Therefore a vapor-liquid equilibrium forms, which provides a vapor ( $y_2$ ) enriched in volatile compound.

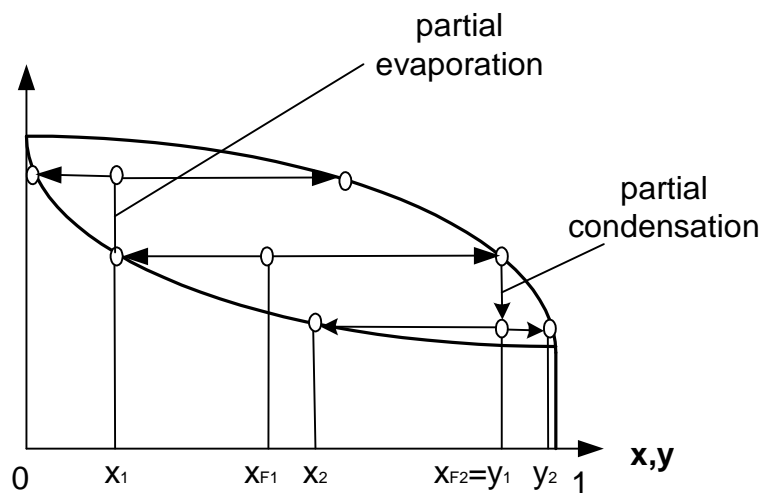


Figure 1: Principle of partial-evaporation and partial-condensation

The limiting factor of this process is heat transfer. Every single stage claims cooling or heating and they produce intermediate products. In order to reduce the total heat transfer, the intermediate products are recycled back onto the previous or the following stage. This is the principle of multistage distillation. (See Figure 2.) It can be seen in Figure 2 that the whole equipment constitutes a cascade system, which claims cooling and heating at one stage. The cascade produces two products: a vapor-phase product at the top, which has a high volatile compound content; and a liquid-phase product at the bottom, which has low volatile

compound content. The vapor-phase product is usually condensed, and sometimes overcooled. The recycled and enriched liquid-phase is called the reflux. The head product is called distillate; the liquid-phase product at the bottom is called the bottom product. The heat transfer is located at the boiler/vaporizer.

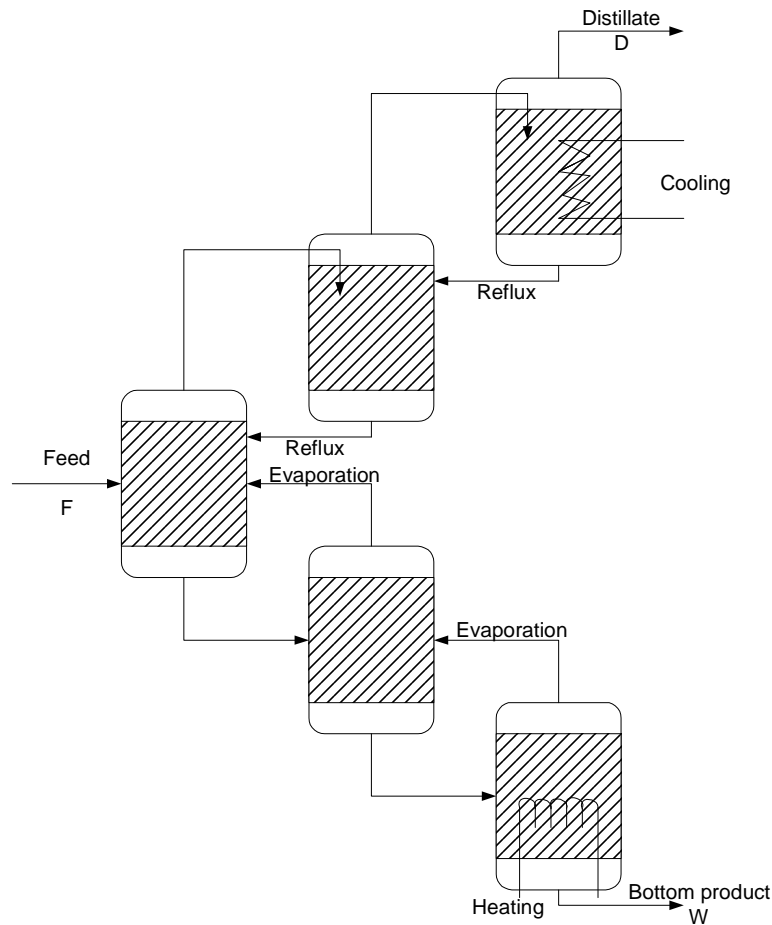


Figure 2: Principle of multistage distillation

In exploitation, the process is carried out in one vertical, cylindrical-shaped apparatus, called the multistage distillation tower. In order to reach better intensification of the vapor and liquid phases, horizontal plates or packages with high porosity are built into the tower. The apparatus is connected with a boiler/vaporizer and a condenser.

## 2. Determination of the number of the theoretical plates in case of rectification

The quality of separation can be characterized by the number of the theoretical plates, in other words the theoretical stages. A theoretical plate is a functioning plate, where the vapor rising from the stage and liquid leaving downpipe the same stage are in equilibrium. Several methods exist to determine the number of the theoretical plates, but in most cases the analytic calculation from plate to plate, or the McCabe-Thiele diagram is used.

During the calculation the term of Lewis is applied. According to the term of Lewis, the mole rate of the vapor-phase upstream and the mole rate of the liquid-phase downstream is constant along the length of the distillation tower. This means that at a given plate one mole condensing vapor results in vapor product, which is enriched in the volatile component. The term of Lewis assumes that:

1. the heats of evaporation in case of the two component are equal
2. the sensible changes in heat can be neglected compared to the heat of evaporation
3. no mixing heat occur
4. the loss of heat is negligible small

### 3. Balances of the multistage distillation tower

The schematic drawing of a continuous multistage distillation tower can be seen in Figure 3.

For the determination of the number of theoretical plates in case of the separation of  $x_D$ - $x_W$ , the equilibrium data of the mixture, the mass rate and concentration values are needed. The principle of determination as follows:

The concentration of the liquid-phase downstream (leaving a given plate) and the concentration of the vapor-phase upstream (leaving the same plate) are located on the equilibrium curve. The operating line shows the relationship between  $y_{n+1}$ - $x_n$  i.e. the vapor-phase upstream leaving the theoretical plate no.  $n+1$  and the liquid-phase downstream leaving the theoretical plate no.  $n$ .

#### 3.1. Mass balance

The total mass balance of distillation is as follows:

$$F = D + W \quad (1)$$

where  $F$  is the feed,  $D$  is the distillate and  $W$  is the bottom product. All of them can be expressed in kg, kg/s, mole, or mole/s.

Equation 2 expresses the component balance:

$$F \cdot x_F = D \cdot x_D + W \cdot x_w \quad (2)$$

where  $x$  is the concentration in mole/mole.

#### 3.2. Mass- and component balance of the enrichment zone

If the enrichment zone of the multistage distillation tower is considered and the term of Lewis is taken into account, equations are as follows:

$$V_{n+1} = V \quad \text{and} \quad L_{n+1} = L \quad (3)$$

$$V = D + L \quad (\text{mass balance}) \quad (4-a)$$

$$V \cdot y_{n+1} = D \cdot x_D + L \cdot x_n \quad (\text{component balance}) \quad (5-a)$$

The reflux ratio is defined as follows:

$$R = \frac{L}{D} \quad (6)$$

where  $L$  is the flow rate of liquid downstream,  $D$  is the flow rate of distillate.

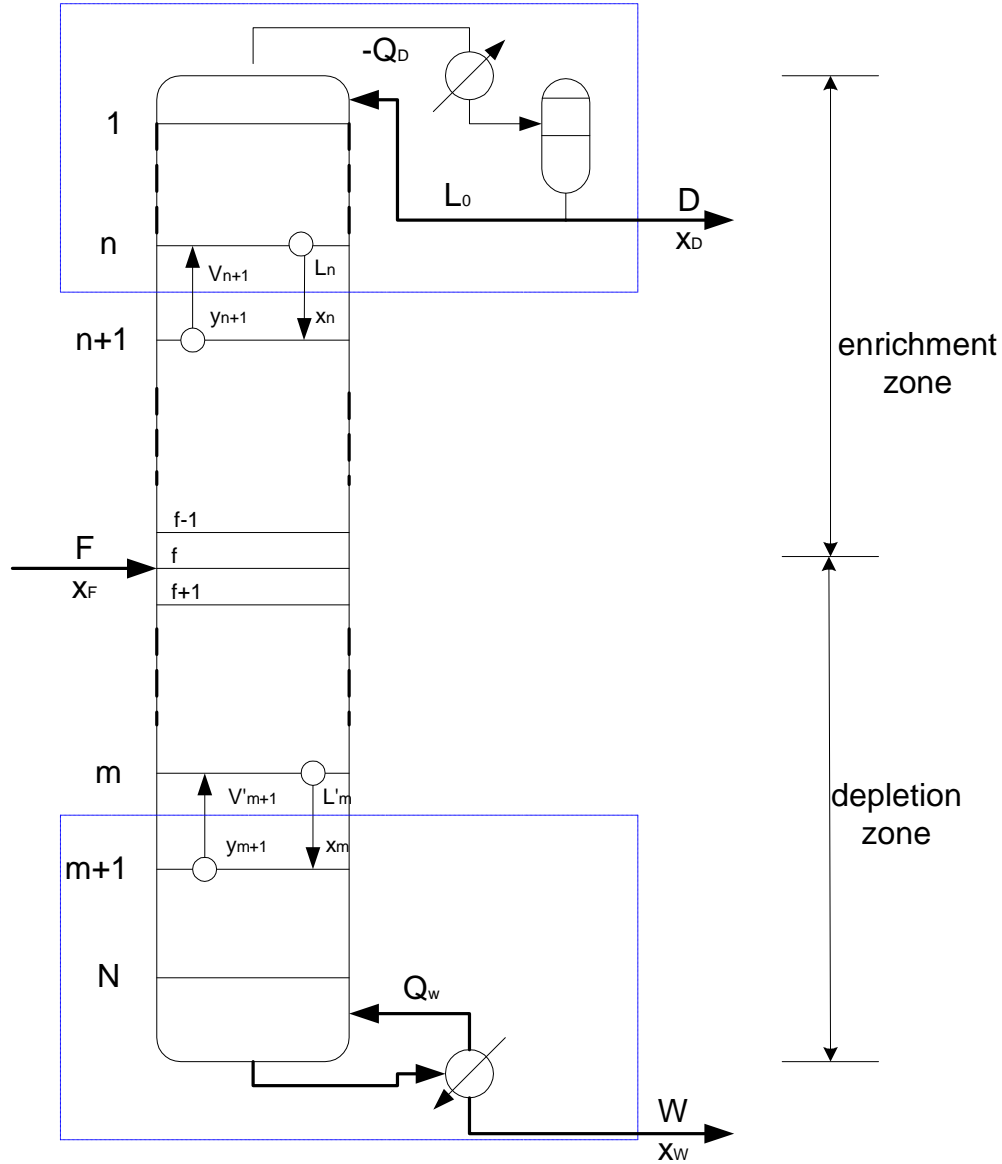


Figure 3: A schematic drawing of the multistage distillation tower

If Eq.(6) is implemented into Eq. (5-a), the operating line of the enrichment zone forms:

$$y_{n+1} = \frac{R}{R+1} \cdot x_n + \frac{x_D}{R+1} \quad (7)$$

The operating line expresses the relationship between the concentrations of the liquid-phase downstream from plate no.  $n$  and the vapor-phase upstream from plate no.  $n-1$ .

### 3.3. Mass- and component balance of the depletion zone

Similar equations can be applied for the depletion zone of the multistage distillation tower. The liquid and vapor rates are different from the rates in the enrichment zone. Represent  $L'$  and  $V'$  the liquid-phase downstream and the vapor-phase upstream, respectively in case of the depletion zone! The reason of the difference is the feed, because the heat of the feed influences the mole rates of the liquid-phase downstream and the vapor-phase upstream.

$$L' = V' + W \quad (4-b)$$

$$L' \cdot x_m = V' \cdot y_{m+1} + W \cdot x_w \quad (5-b)$$

If  $y_{m+1}$  is expressed from Eq. (5-b), the operating line of the depletion zone forms:

$$y_{m+1} = \frac{L'}{V'} \cdot x_m - \frac{W}{V'} \cdot x_w \quad (8)$$

### 3.4. Determination of the number of theoretical plates using McCabe-Thiele diagram

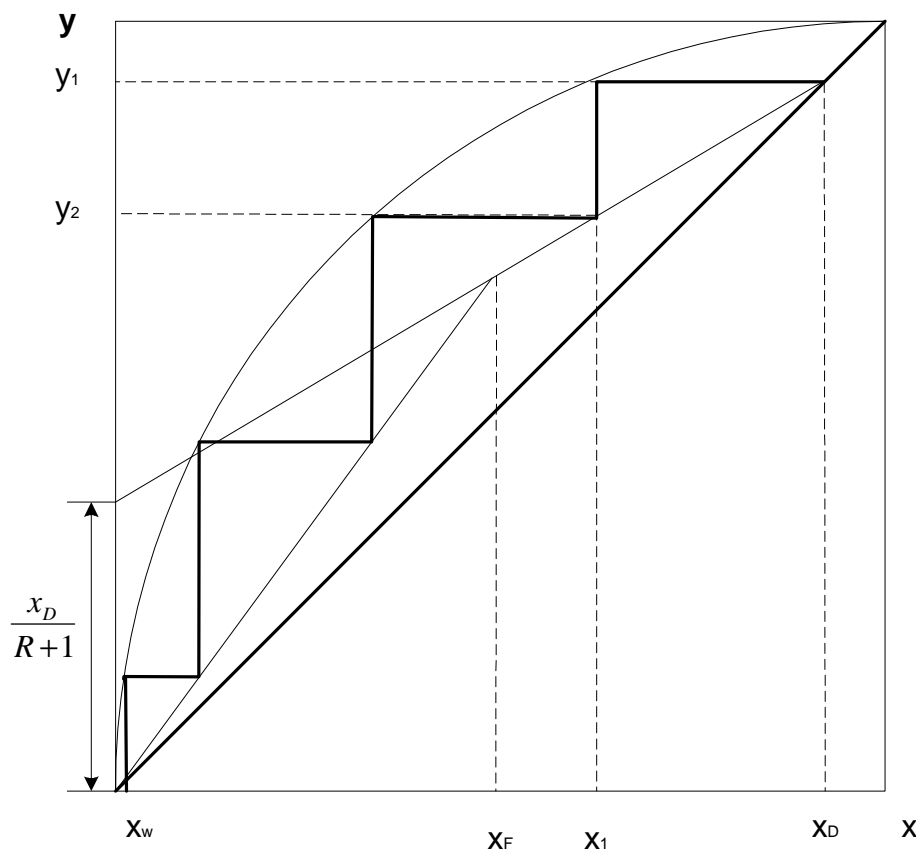


Figure 4: McCabe-Thiele diagram

The first step of editing the diagram is to draw the equilibrium line for the system on the diagram. The operating lines of the enrichment zone and the depletion zone are drawn on the diagram. Eq. 7, namely the operating line of the enrichment zone can be represented by a straight line of slope  $\frac{R}{R+1}$  (which is equal to  $\frac{L}{V}$ ), passing through the point  $x = x_D$  and  $y_1 = x_D$ . The intercept of the straight line is  $\frac{x_D}{R+1}$ . Eq. 8, i.e. the operating line of the depletion zone can be represented by a straight line of slope  $\frac{L'}{V'}$ , passing through the point  $x = x_w$ . It can be shown that under the conditions assumed above the operating lines intersect

at a point where  $x = x_F$ . The number of the theoretical plates can be obtained by drawing vertical and horizontal steps between the equilibrium and operating lines. As shown in Figure 4, this graphical construction is known as the McCabe-Thiele Diagram. The number of the steps between  $x_F$  and  $x_D$  gives the number of the theoretical plates above the feed, and the steps between  $x_W$  and  $x_F$  represent the plates below the feed plus the boiler, which is treated as one theoretical plate. The  $x$  co-ordinate of any vertical step representing a plate is the liquid composition on that plate; the  $y$  co-ordinate of the point of intersection with the operating line is the composition of the vapor rising from the plate below and the intersection with the equilibrium line gives the vapor composition leaving the plate in equilibrium with the liquid.

Note: In order to study the multistage distillation, total reflux is often used. Total reflux means  $D=0$ ;  $W=0$  and  $F=0$ . In this case the reflux ratio is infinite. The two operating lines form one line, which is the diagonal of the square.

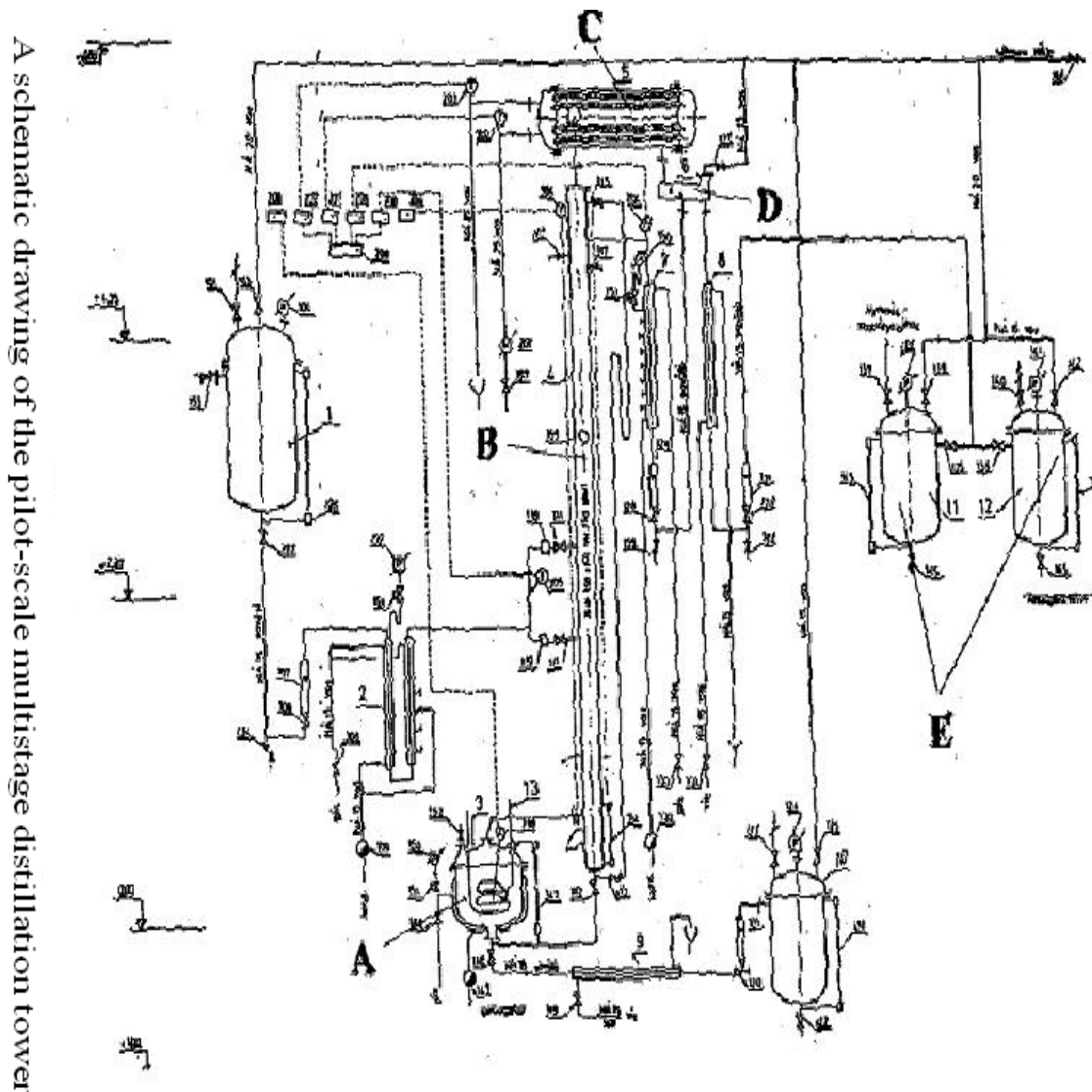


Figure 5: A schematic drawing of the pilot-scale multistage distillation tower

#### 4. The pilot-scale multistage distillation tower

The theoretical flow chart of the apparatus can be seen in Figure 5. The main elements of the multistage distillation tower are as follows:

- A. boiler with a volume of 100 dm<sup>3</sup>. The casing and the coil pipe of the boiler can be heated by steam
- B. multistage distillation tower with an inside diameter of 150 mm, and a height of 1.5 m. It is filled with VM-350 package.
- C. Condenser
- D. Reflux-distributor
- E. Distillate collector

The tower can be applied for either batch or continuous distillation, and also at atmospheric pressure or at vacuum. During the laboratory practice atmospheric pressure and total reflux are used for the separation of methanol-water mixture.

To be determined:

- a) Number of the theoretical plates considering 1 m package
- b) Load factor of the tower at the head and at the bottom of the tower
- c) Heat balance of the condenser

The measurement:

Open the cooling water of the condenser. Adjust it so, that the temperature difference between the inflow and outflow be between 10°C and 40°C. The flow rate of the cooling water can be measured by a flow meter.

Open the steam valve, which provides the heating of the bottom of the tower and the coil pipe. Wait until the stationary state.

At the stationary state (when the temperatures are constant), read the values of the parameters as follows:

- Pressure of the heating steam:.....bar
- Flow rate reflux:.....L/h
- Temperature of the inflowing cooling water:.....°C
- Temperature of the outflowing cooling water:.....°C
- Temperature of the reflux:.....°C
- Time of 0,1 m<sup>3</sup> cooling water:.....s
- Water content of head product:.....mole/mole
- Methanol content of the bottom product:.....mole/mole

The water content of the bottom product is determined by Karl-Fischer titration. The methanol content of the head product is determined by a gas chromatograph.

Evaluation:

- a. number of the theoretical plates

The number of the theoretical plates is edited from the concentrations measured using simulation program.

- b. The load factor of the tower

$$F = v \cdot \sqrt{\rho_G} \quad \left[ \sqrt{Pa} \right] \quad (9)$$

where  $v$  is the velocity of vapor in the tower (m/s) and  $\rho_G$  is the density of vapor (kg/m<sup>3</sup>).

The density of vapor can be calculated from the gas law:

$$\rho_G = \frac{M}{22.41} \cdot \frac{P}{P_N} \cdot \frac{T}{T_N} \quad (10)$$

$M$  is the average molar weight,  $P_N$  and  $T_N$  are the pressure and temperature at 20°C,  $T$  is the temperature of the head or bottom product, and  $P$  is the pressure of the heating steam.

Since the scale of rotameter is considering to water, the read value of rotameter has to be turned into l/h methanol. This can be carried out using Eq. (11):

$$W_{MeOH} \approx W_{water} \cdot \sqrt{\frac{\rho_{water}}{\rho_{MeOH}}} \left[ \frac{dm^3}{h} \right] \quad (11)$$

The volumetric rate of methanol is divided by the density of methanol results in the mass rate of methanol, which is equal to the mass rate of the rising vapor, because total reflux is applied. From the mass rate of the rising vapor, the volumetric rate of the rising vapor can be calculated since the density of the vapor is known from Eq.10. The velocity of the vapor can be calculated from the volumetric rate of the vapor, dividing it by the diameter of the tower. The load factor can be determined using Eq.9. The load factor has to be determined for both the head and bottom product. The term of Lewis can be applied. This can be carried out turning the mass rate of head product to mole rate because we know the composition of the head product. According to the term of Lewis the mole rate of the head product is equal to the mole rate of the bottom product. The composition of the bottom product is measured, so the mole rate of the bottom product can be easily turned into mass rate. From the vapor density belonging to the given composition, the volumetric rate of vapor can be calculated. From this value the velocity of vapor can be calculated, and after that the load factor of the depletion zone can be determined.

### c. Heat balance

The heat balance is considering the condenser:

$$V \cdot i_V'' + H \cdot c_{P,water} \cdot T_{in} = L \cdot c_{P,L} \cdot T_L + H \cdot c_{P,water} \cdot T_{out} \quad (12)$$

Reordering the Eq.11:

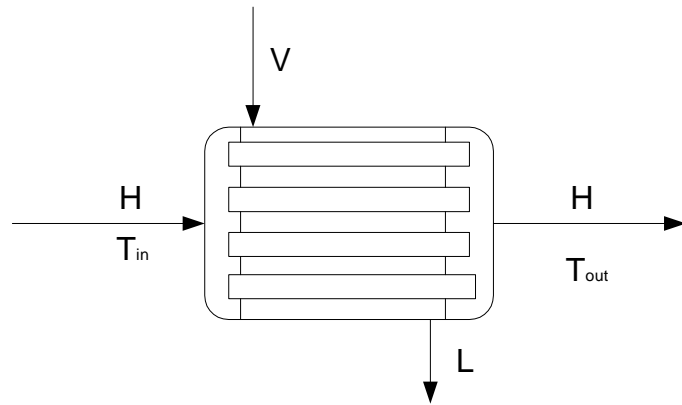
$$V \cdot (i_V'' - i') = V \cdot r = H \cdot c_{P,water} \cdot (T_{out} - T_{in}) \quad (13)$$

and  $V=L$  because of the total reflux.  $V$  is the quantity of steam (kg/h),  $L$  is the quantity of condensed steam (kg/h),  $H$  is the quantity of cooling water (kg/h).

The temperature of the liquid leaving the condenser can be treated as boiling temperature. ( $c_{P,L} = i'$ )

The evaporation heat of methanol:  $r = 1102$  kJ/kg, and density of methanol  $\rho = 787$  kg/m<sup>3</sup>.





*Figure 6: A schematic drawing of the condenser*