***LABORATORY OF SEPARATION PROCESSES***

**Work No. 11**

**Differential rectification in tray column**

**IN A TUTSHELL**



Distillation is a diffusion separation process through which it is possible to separate individual components of a homogeneous liquid mixture based on their different boiling points, or varying volatility. The oldest distillation processes include batch, differential distillation, and simple continuous distillation. In both cases, only limited separation can be achieved. By introducing the distillate into another distillation stage, it is possible to achieve higher purity of the resulting distillate (Fig. 1 A)). A downside of such multi-stage distillation is the relatively low yield and high energy demands, as the cooled liquid distillate must be brought to a boil again and the mixture partially evaporated. The yield has been successfully increased by reusing the residue (Fig. 1 B)) and the energy demand was reduced by utilizing the energy (enthalpy) from the previous stage (Fig. 1 C)).

Obrázok, na ktorom je náčrt, diagram, kresba, technický výkres

Automaticky generovaný popis

Fig 1 A) Multistage distillation without the use of residues. B) Multistage distillation with the use of residues. C) Multistage distillation with the use of residues and vapor enthalpy = tray rectification.

This type of multi-stage distillation with counter-current flow of liquid and vapors is called rectification, and the device in which it is carried out is known as a rectification column. The energy required to generate vapors is supplied to the boiler, at the bottom of the rectification column, and vapors from the top stage condense in the condenser, with a portion of them being returned as reflux to the top stage.

During the contact between upward flowing vapors on a stage and the liquid descending to the bottom of the column, into the boiler, bidirectional mass transfer occurs, accompanied by heat exchange and partial condensation of the less volatile component from the vapors into the liquid and partial evaporation of the more volatile component from the liquid into the vapors. Through this process, the vapors become enriched with the more volatile component as they pass through the column.

Efficient enrichment of the vapors with the more volatile component requires intensive contact between the vapor and liquid phases. To achieve sufficiently intensive mixing of the phases within the column, various types of internals or packings are used in rectification columns. If internals are placed within the column, intensive phase contact occurs only at certain sites within the column. In this case, we refer to tray rectification columns. Individual trays in the rectification column retain a portion of the liquid phase (liquid holdup), through which the vapor phase passes (vapor holdup is often considered negligible). The liquid flows to the lower tray through overflow pipes, whose height determines the liquid holdup on the tray. In practice, the most widespread types are bubble cap, sieve, grid, and valve trays (Fig. 2).



Fig 2 Tray column with various types of internals.

While in tray-based systems the composition of vapors and liquid changes "abruptly" from one tray to another, in packed columns, the composition of vapors and liquid changes continuously.

By increasing the number of trays, or by increasing the height of the packing in the rectification column, we also increase its separation efficiency. In rectification columns, the efficiency of separation can also be influenced by changing the reflux ratio, which represents the ratio of the molar flow of condensed vapors that are returned to the column (reflux) to the molar flow of the distillate that is taken from the column.

Rectification columns can be designed for both continuous and semi-continuous processes. In continuous rectification columns, the feed is continuously introduced into the column. The distillate is taken from the top of the column (the topmost tray) and the residue from the boiler, with their composition and molar flow remaining constant over time (they are in a steady state). In semi-continuous rectifications, the feed is added into the boiler, the distillate is taken from the top of the column, and the residue remains in the boiler. In such cases, it is differential distillation, because either the composition of the distillate or its flow changes over time. If we have a constant reflux ratio during differential rectification, then the composition of the distillate will change over time. An example of differential rectification with a constant reflux ratio is the distillation of alcohols from natural sources (e.g., agave). During the process, fractions of the distillates are taken off at specified intervals of distillation temperatures, corresponding to certain proportions of distilled components. Individual fractions are then mixed in appropriate ratios to create distillates with the desired sensory properties. If a constant composition of the distillate is required in differential rectification, the reflux ratio must be adjusted over time, which from a process control perspective is not the simplest requirement.

# THEORY

# Vapors from the first stage of the rectification column are led to the condenser. The condensate is then divided into two parts: one is taken off as the product, and the other is returned to the column as liquid reflux (Fig. 3). The ratio of the molar amount of reflux to the molar amount of distillate is called the reflux ratio:

|  |  |
| --- | --- |
|  | (1) |

# where *R* is the reflux ratio, is liquid reflux returned to the column and is molar amount of distillate. The value of the reflux ratio in in the interval *R ϵ< Rmin ; ∞ > .*

The minimum reflux ratio, *Rmin ,* is the theoretical value at which it is still possible to obtain a distillate with the desired content of the more volatile component from the feed. However, this value is theoretical because achieving the desired separation of the feed would require an infinitely large rectification column. This implies that only a minor enrichment of the vapors with the more volatile component occurs at each stage of the rectification column, and thus, an infinite number of stages would be required to achieve the desired separation.

Conversely, at an infinitely large value of the reflux ratio, *R = ∞* , the enrichment of the vapors with the more volatile component is maximized at each stage, and a column with the minimum number of stages suffices for the desired separation of the feed. To achieve a reflux ratio of *R = ∞*, the denominator in equation (1) must be zero, meaning no distillate is withdrawn from the rectification column.

Therefore, the value of the reflux ratio significantly affects the separation efficiency of the rectification column. The higher the value of (*R*), i.e., the larger the amount of condensate returned to the rectification column as reflux, the better the conditions within the column for enriching the vapors with the more volatile component.

Obrázok, na ktorom je text, diagram, rad, snímka obrazovky

Automaticky generovaný popis

Fig 3 Flow diagram of a differential tray column. The magnified view shows (N-1) tray.

During the startup of a differential rectification process, until the column warms up to the operating temperature and its regime stabilizes (temperatures, liquid holdup on stages), all the condensate is returned back to the column as reflux, i.e., the column operates at an infinite reflux ratio. After the conditions in the rectification column have stabilized, a portion of the condensate is withdrawn as distillate, the product of rectification.

Determining the number of stages required in the rectification column to achieve the desired separation of the feed is based on monitoring the changes in the composition of the vapor and liquid phases along the rectification column. To simplify the solution to this problem, the concept of a theoretical stage is used. This concept assumes equilibrium between the liquid and vapor phases leaving the stage (Fig. 3).

The correlation between the composition of the vapor stream leaving a stage and the liquid that flows onto this stage is derived from the material and enthalpy balance of the individual stages of the rectification column. If we simplify by assuming that the molar evaporative enthalpies and the molar heat capacities of the components of the mixture to be separated are equal, and if we neglect the value of the mixing enthalpy, under steady-state conditions, it holds that the molar flow rate of the liquid phase flowing onto a stage equals the molar flow rate of the liquid phase flowing from the stage. The same applies to the molar flow rate of the vapor phase. Based on this assumption, the relationship between the composition of the vapors flowing from the certain stage and the liquid flowing on to this certain stage of the enriching section of the rectification column can be expressed by the operating line equation:

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| --- | --- |
|  | (2) |

where *y* and *x* are the mole fractions of the more volatile component in the vapor and liquid phase, respectively, *R* is the reflux ratio, the index *N* refers to the stage number in the enriching section of the rectification column, and the lower index The stages in the column are numbered from the top (condenser) to the bottom (reboiler).

The assumptions we adopted when deriving the operating line for the enriching section of the rectification column allow us to determine the number of theoretical stages of the rectification column using the McCabe-Thiele method. First, it is necessary to construct a *y-x* diagram, into which the equilibrium dependence for the chosen binary mixture *y = f(x)* and the diagonal (Fig. 4) are plotted.

Obrázok, na ktorom je rad, diagram, rovnobežný

Automaticky generovaný popis

Fig 4 A graphical method for determining the number of theoretical stages of a rectification column using the McCabe-Thiele method. For: A.) Rmin < R < ∞; number of steps= 5 , B.) R = ∞; number of steps = 3,5.

On the diagonal, we mark the composition of the feed, distillate, and residue. Using equation (2), we construct the operating line for the enriching section of the rectification column. We assume that the stages in the column act as theoretical stages, meaning that the liquid phase and the vapor phase leaving this stage are in equilibrium. The composition of the vapor phase rising from the stage and the liquid phase coming to this stage is given by the operating line equation. These links between the compositions of the more volatile component in both phases on each stage can be drawn in the *y-x* diagram, resulting in right-angle steps. One right-angle step then represents the transition from one stage to another. The last right-angle step ends at the composition of the residue in the boiler. The number of theoretical stages (NTS) then corresponds to the number of steps minus one, which represents the boiler of the rectification column. For a column operating at an infinite reflux ratio, *R = ∞*, the operating line of the enriching section merges with the diagonal, and in batch rectification, no additional feed is introduced to the column, so the composition of the feed is not necessary (Fig. 4 B)).

The assumption of achieving equilibrium at each stage (theoretical stages) is rarely fulfilled under real conditions. An information on the rate to which the composition of the liquid and vapor stream leaving the real stage has approached equilibrium is given by the column efficiency.

|  |  |
| --- | --- |
|  | (3) |

where is the efficiency of the column, *NTS* and *NAS* represent the number of theoretical and actual stages of the rectification column, respectively. The efficiency of the rectification column is determined experimentally by comparing the achieved and theoretical sharpness of separation. It also expresses the average value of the vapor enrichment efficiency with more volatile component on the individual stages of the column.

The enrichment efficiency of the *n*-th stage, *EN*, is defined as the ratio of the degree of achieved and theoretical vapor enrichment with the more volatile component on that stage.

|  |  |
| --- | --- |
|  | (4) |

Where represents the composition of the vapors (mole fraction of the more volatile component in the vapor phase) leaving the *N* -th stage, and is the composition of vapors coming from the next lower stage. The symbol corresponds to the mole fraction of the more volatile component in the vapor phase, which would be in equilibrium with the liquid flowing down from the *N* -th stage.

In the case where the column operates at an infinite reflux ratio, under steady-state conditions, the composition of the liquid flowing down from a higher stage to the *n* -th stage is same as the composition of vapors rising from the *n* -th stage:

|  |  |
| --- | --- |
|  | (5) |

In this case, the enrichment efficiency of the *n* -th stage can be expressed by the equation:

|  |  |
| --- | --- |
|  | (6) |

In the literature, empirical correlations to determine the enrichment efficiency of the *N* -th stage are given by one of the following equations:

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| --- | --- |
|  | (7) |

where *α* represents the relative volatility of the components of the mixture being separated at the temperature on the stage, and (mPa.s) is the viscosity of the liquid phase on the stage.

Equation (7) includes only some of the conditions that affect the enrichment of the vapor with more volatile component. In addition to temperature, the composition of the liquid phase, and phase equilibrium, the enrichment efficiency of a stage is also influenced by other physicochemical properties of the mixture being separated, the hydrodynamic conditions in the column, and the geometric shape of the stage. The influence of these factors on the enrichment efficiency of the stage is quantified by introducing a correction, which is represented by the coefficient *K* in the following equation:

|  |  |
| --- | --- |
|  | (8) |

# OBJECTIVES OF THE WORK

1. Determine the separation efficiency of the stages and the boiler of a laboratory bubble cap rectification column, as well as the overall separation efficiency of the enrichment part of the column.

**ASSIGNMENT OF THE WORK**

Perform differential rectification of a binary methanol-isopropanol mixture Determine the enrichment efficiency values of the stages and the boiler of a laboratory column operating at an infinite reflux ratio. Calculate the value of the correction factor *K*. Calculate the efficiency *η* of the laboratory column without the boiler. Calculate the steam velocity in the free cross-section of the laboratory column *w*. Perform the experiments at 1200 W and 1800 W on the heating boiler's regulatory transformer of the column.

**EQUIPMENT DESCRIPTION**

The experiment is conducted in a laboratory rectification column:

A diagram of a machine

Description automatically generated

Fig 5 Schematic diagram of the differential rectification in bubble cup column.

The laboratory column consists of the five bubble cup stages (4). The lower part of the column is connected to the boiler (1). The vapor from the top (first) stage of the column is fed to the condenser (7). The boiler is filled with the feed with volume of 6 liters. The heating of the feed and its partial evaporation is provided by induction heating (2). The vapor passes through a droplet separator and an isolated section of pipe (1d) before entering the column. The reverse flow of the liquid returning to the column flows from stage to stage through bubble caps, and the liquid phase is sampled using sampling valves (5a-5c). The temperature profile in the column is indicated on the control panel display (10g).

The rectification column operates at atmospheric pressure at an infinite reflux ratio, with all of the vapor condensates being directed as liquid to the rectification column. From the last stage, the liquid phase reflux flows through a measuring cylinder (3) with a shut-off valve (3a) into the boiler. On the control panel there is a main switch (10a), a wattmeter (10b), a heating controller (10c - 10f) and a temperature display (10g).

**WORKING PROCEDURE**

**SAFETY PRECAUTIONS**

1. When sampling, it is important to be aware that we are working with a boiling liquid. Additionally, we are working with flammable substances, whose vapors are harmful to health.

**PREPARATION FOR THE MEASUREMENT**

1. Firstly, we will inspect the equipment and check it for completeness. Then, we will prepare the digital refractometer for measurement.
2. Supply cooling water to the condenser (7).
3. Request permission to start the measurement.

**MEASUREMENT**

Note: Record all parameter values highlighted in RED in *Table 1* and *Table 2*, respectively.

1. Turn on the main switch (10a) on the control panel. Switch on the induction heater (10c) and set the power mode (10e). Use the rotary control (10f) to set the desired capacity of the induction heater.
2. When the vapor temperature at the head of the column is stabilized (approx. 15 min), start the measurement.
3. Close the valve (3a) and at the same time switch on the stopwatch. Measure the time *(τ)* required for the selected volume of liquid phase *(VL)* reflux to flow in the measuring container (3). Open the shut-off valve (3a). On the basis of the measured data, calculate the flow rate of the liquid phase *()* from the fifth stage. Enter the measured data in the *Initial* in Table 1.
4. After five minutes, take samples of the liquid phase using the sampling valves (5a - 5c) from each of the five stages, the condenser (9) and the boiler (1a). Wash the sample pipette with the liquid from the sampling valve before the actual sampling.
5. Determine the refractive index *()* of the individual liquid phase samples refractometrically (Instructions are in the ATTACHEMENT).
6. At the same performance setting, repeat the sampling and analysis of samples according to step 4 and 5 four more times at five-minute intervals.
7. Repeat the measurement of the flow rate of the liquid phase from the fifth stage according *()* to point 3 (write it in the *Final*). From the initial and final liquid phase flow rate measurements from the fifth stage, calculate its average value.
8. Set the second value of the required induction heating power. Wait for the conditions in the rectification column to stabilize and then carry out the measurements according to points 3 to 7. While the conditions are settling, note down the atmospheric pressure value *().*
9. Each measured refractive index value *()* is calculated using the calibration dependence (ATTACHEMENT) to the mole fraction of the more volatile component *()*.
10. For each sampling position and for both heating power settings, calculate the arithmetic average of the mole fractions of the more volatile component in the liquid phase *().*

**Note:** The efficiency of component separation in the mixture also depends on the system's hydrodynamics and the retention of liquid on the stages. Therefore, it is necessary to observe the retention on the stages after the column's state stabilizes, the mixing of the vapor and liquid phase, and whether there can be carryover of mixture droplets from a lower stage to a higher one, etc. In the discussion, relate the determined efficiencies of the stages to the behaviour of the liquid and vapor phases on the stages.

**END OF THE MEASUREMENT**

Turn off the Induction Heater (10c) and the main switch (10a) on the control panel. Do not turn off the cooling water. Do not empty the boiler contents.

**YOU CAN SEE THE DESCRIPTION OF THE DEVICE AS WELL AS THE WORK PROCEDURE IN THE VIDEO:**

Obrázok, na ktorom je vzor, steh, pixel

Automaticky generovaný popishttps://[www.youtube.com/watch?v=0SPI](http://www.youtube.com/watch?v=0SPI) M9cry\_8&list=PL81QAQg818vjDK7pm9W O6tW36sdBpPGYa&index=3

**QUESTIONS - WHAT THE TEACHER MAY ASK YOU BEFORE THE EXPERIMENT**

* Describe the different parts of the experimental equipment.
* Explain what you will measure, how and why.

**MEASUREMENT DATA PROCESSING**

**Note:** Record all parameter values highlighted in RED in *Table 3*.

1. Based on the equilibrium data (Chemical Engineering Tables), plot an equilibrium y-x diagram.
2. The average values of the liquid phase composition are used to calculate the enrichment efficiency of the individual stages () according to equation (6). The equilibrium composition of the vapor phase, , is obtained from the equilibrium y-x diagram.
3. From equation (8) calculate the value of the coefficient (*K*) for each stage of the column.

* The relative volatility () of the more volatile component (A) with respect to the less volatile component (B) of the binary mixture is calculated as:

|  |  |
| --- | --- |
|  | (9) |

where is replaced by the average value of the mole fraction of the more volatile component () at a particular stage. is determined from the equilibrium y-x diagram. The mole fractions and are obtained from the sum of all the mole fractions in mixture = 1.

* The dynamic viscosity of the liquid phase () at the stage is calculated according to equation (10)

|  |  |
| --- | --- |
|  | (10) |

the dynamic viscosity of the pure more volatile () and less volatile component () is calculate using the equations given in the Chemical Engineering Tables fir the temperature at the stage. The temperature at the stage is obtained from the t-x,y diagram ((APPENDIX) for the average value of the mole fraction of the more volatile component () at a particular stage.

1. For each setting of heating performance (10e), calculate the average value of the flow rate by volume using the mixture density (we know the average composition and temperature at stage 5) to the mass flow rate, and by mole weight to the mass flow rate of the liquid phase in the column.

|  |  |
| --- | --- |
|  | (11) |

1. At infinite reflux ratio, the flow rate of vapor and liquid phase in the column is the same ). Using the ideal gas equation of state, calculate the vapor flow rate from the molar flow rate and the atmospheric pressure value (*Patm*) and then the flow rate of the vapor () in the free cross-section of the 5th stage ( ).

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| --- | --- |
|  | (12) |

where *R*  is the gas constant, *T* is the thermodynamic temperature at the fifth stage, and *A* is the free cross-sectional area of the column (Note: the inner diameter of the column is 8 cm).

1. In the equilibrium *y–x* diagram, plot rectangular steps between the operating line and the equilibrium dependence that correspond to the theoretical number of stages (NTS) required to achieve the actual column separation effect. Using equation (3), calculate the enrichment efficiency of the column *(η)*  for both performance settings on the control transformer.

**TABLES**

*Tab. 1* Measured data record for power setting ............ W on induction heater

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | Volume flow rate measurement | | | | | |
|  |  | |  | |  | |
| Initial |  | |  | |  | |
| Final |  | |  | |  | |
| Average |  | |  | |  | |
| **Measurement** | Boiler | | 5.stage | | 4.stage | | 3.stage | | 2.stage | | 1.stage | | Distillate | |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 1 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 4 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 5 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

*Tab. 2* Measured data record for power setting ............ W on induction heater

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | Volume flow rate measurement | | | | | |
|  |  | |  | |  | |
| Initial |  | |  | |  | |
| Final |  | |  | |  | |
| Average |  | |  | |  | |
| **Measurement** | Boiler | | 5.stage | | 4.stage | | 3.stage | | 2.stage | | 1.stage | | Distillate | |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 1 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 4 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 5 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

*Tab. 3* Results of measured data evaluation

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Power | Parameter | Boiler | 5.stage | 4.stage | 3.stage | 2.stage | 1.stage |  | *NTS* |  |
| …… W | *E*N |  |  |  |  |  |  |  |  |  |
| K |  |  |  |  |  |  |
| …… W | *E*N |  |  |  |  |  |  |  |  |  |
| K |  |  |  |  |  |  |

**ATTACHEMENT**

**Obrázok, na ktorom je rad, vývoj, text, diagram

Automaticky generovaný popis**

**INSTRUCTION FOR THE USE OF DIGITAL REFRACTOMETER KRÜSS DR301-95**

1. Connect the device to the power supply.
2. Press the ON/OFF button.
3. Open the lid and clean the prism (if necessary).
4. Drip about 3-4 drops onto the prism so that the entire surface of the prism is covered with liquid, be careful of bubbles.
5. Close the lid.
6. Press the READ/ENTER button.
7. The index of refraction will appear on the display.
8. Clean and dry the prism with a cotton stick.
9. The instrument is ready for further measurements.
10. When finished, turn off the refractometer with the ON/OFF button and disconnect the instrument from the power supply.