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Laboratory of separation processes

Excercise n.14

PARTIAL MASS TRANSFER COEFFICIENT IN LIQUID PHASE

# IN A NUTSHELL

Absorption is a diffusive separation process aimed at separating or removing components from a gas mixture. The principle of absorption is based on the varying solubility of gases in a liquid solvent (absorbent). Gas absorption is encountered in everyday life as well. Thanks to the absorption of atmospheric oxygen into water, fish can "breathe", and the bubbles in carbonated drinks are achieved through absorption. In the carbonation of beverages, carbon dioxide is forced into the water where it dissolves - it is absorbed. The solubility of gases in liquids depends on temperature as well as pressure. Higher pressure and lower temperature promote absorption. Therefore, carbonated beverages are produced at higher pressure and reduced temperature. Conversely, fish in summer may suffer from a lack of oxygen due to increased temperature and thus impaired oxygen absorption into water. Understanding absorption is crucial to grasp the theory of mass transfer and film theory.

In general, the mechanism of mass transfer is divided into molecular diffusion and convective mass transfer. Molecular diffusion, abbreviated as diffusion, occurs at the molecular level and takes place in stationary environments. The transport of substance itself occurs from a region of higher concentration to a region of lower concentration of the transported component. The driving force for mass transfer is the previously mentioned concentration difference, also known as the concentration gradient. The mass transfer continues until thermodynamic equilibrium is reached. In everyday life, we can observe diffusion when making tea, as the entire volume of the cup gradually changes color (Figure 1a). The rate of mass transfer by diffusion depends on the concentration gradient as well as the resistance of the medium to the transferring substance.

In the case of a moving medium, mass transport occurs by convection. The equalization of concentration gradients is achieved by local currents and eddies. The rate of mass transport by convection depends on the nature of the flow (hydrodynamic conditions). Compared to diffusion, the rate of mass transport by convection is significantly higher. We can observe this again when making tea. After inserting the tea bag, we stir the entire volume of the cup, noticing faster coloration of the water (Figure 1b).

Obrázok, na ktorom je nápoj, nápojové sklo, džús, nealkoholický nápoj

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Figure 1 An example of mass transfer through diffusion and convection can be seen in the process of brewing tea.

In practice, both mentioned mechanisms of mass transfer are almost always applied simultaneously, constituting combined mass transfer by convection and diffusion.

Several models of combined mass transfer have been published in the literature, of which the film theory and penetration theory are most commonly used. The advantages of the film theory of mass transfer are related to its clarity, simplicity, and relatively good agreement with experimental data. Due to these advantages, we will further focus on the film theory of mass transfer.

During absorption, two phases come into contact: the gas phase containing the absorbed component, and the liquid phase into which it is absorbed. The point of mutual contact is called the phase boundary (Figure 2). A laminar film (boundary layer) forms near the phase boundary on both the liquid and gas phase sides. Mass transfer in the laminar region occurs by diffusion. Outside the laminar region, mass transfer mainly occurs by convection. The rate-determining step in combined mass transfer is the slowest process, in this case, diffusion. The greater the velocity of the phase flow (higher turbulence), the smaller the influence of diffusion on mass transfer, and conversely, at lower flow intensity, the influence of diffusion on mass transfer is more pronounced. This is related to the thickness of the laminar film, which depends on turbulence.

In industry, countercurrent absorption columns are often used, where the gas mixture is introduced at the bottom of the column, and the liquid absorbent is introduced at the top of the column (Figure 3). The absorbent flows down from the top of the column, while the gas mixture flows in the opposite direction. Inside the column, there is packing with a large surface area, ensuring sufficient contact between the liquid and gas phases. The absorbent enriched with the absorbed gas component is removed from the bottom of the column, while the gas depleted of this component exits from the top.

Obrázok, na ktorom je text, kruh, snímka obrazovky, písmo

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Figure 2 The illustrative representation of the film theory of CO2 absorption into water.

Diagram

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Figure 3 Flow diagram of a countercurrent absorber. The subscript (1) denotes incoming or outgoing streams at the bottom of the column, and (2) denotes streams at the top of the column. Gas-phase streams are shown in green, liquid-phase streams in blue. The color gradient indicates the concentration of the absorbed component along the column.

# THEORY

The component being absorbed (A) in the gas phase is called the absorbate, while in the liquid phase, it is referred to as the absorbent. Besides this component, the gas phase may also contain additional components. If this additional component in the liquid phase is not absorbed, it is termed as an inert (i). The predominant component in the liquid phase is the absorbent (l), which may consist of one or more non-volatile components.

In further considerations, we will assume that the system under study is two-phase (gas-liquid), two-component (carbon dioxide-water), with substance transfer occurring only in one direction, where CO2 transfers from the gas to the liquid phase.

In chemical engineering practice, it is customary to quantify the gas composition using partial pressures and the concentration in the liquid solution. The mechanism of combined mass transfer by film theory can thus be graphically represented as follows:

Obrázok, na ktorom je diagram, text, rad, plán

Automaticky generovaný popis

Figure 4 Concentration profile of the absorbing component during unidirectional substance transfer.

where represents the pressure of component (A) in the gas core, the pressure of the component at the gas-liquid phase interface,  the thickness of the laminar layer on the gas side,  the thickness of the laminar film on the liquid side, the concentration of the component at the phase interface, and  the concentration of the component in the liquid core.

Film theory of mass transfer assumes that there is a laminar layer on both sides of the phase interface. The thicknesses of the laminar layers on the gas and liquid phase sides depend on the turbulence intensity in the main flow of each phase (expressible using the Reynolds criterion, *Re*).

Under steady-state conditions, the amount of substance transferring from the turbulent core of the flowing gas phase through the laminar film to the phase interface must equal the amount of substance transferring from the phase interface through the laminar film of the liquid phase into its turbulent core. Film theory of mass transfer further assumes attainment of equilibrium at the phase interface, i.e., the rate of mass transfer across the phase interface is very high, and conversely, the resistance to mass transfer across the phase interface is negligibly small. The rate of mass transfer from the gas to the liquid phase corresponds to the rate of the slowest process, i.e., diffusion of the absorbed component through the laminar film near the phase interface. Since in our case the gas phase consists only of pure carbon dioxide, the partial pressure of the absorbate in the gas phase as well as at the phase interface is practically the same and independent of the position in the adsorber.:

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

Therefore, the resistance to mass transfer is concentrated in the laminar layer of the liquid phase. Based on the kinetic theory of gases, the following relationship for mass transfer by diffusion from the interfacial boundary to the turbulent core of the flowing liquid phase was derived:

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

where  represents the flow of the absorbing component (carbon dioxide) from the phase interface to the turbulent core of the liquid phase,  is the binary diffusion coefficient of the transferring substance in the liquid phase,  is the sum of concentrations of all components in the liquid phase, in our case, it is the concentration of water and carbon dioxide in water.  (m) is the thickness of the laminar film on the liquid side,  is the logarithmic mean concentration of components in the liquid phase,  is the area of the phase interface. Parentheses represent the driving force of mass transfer, where  is the concentration of component (A) at the phase interface, and  is the concentration of the component in the turbulent core of the liquid. We can rewrite equation (2) in a shortened form:

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

Where partial coefficient of mass transfer  It is defined as the fraction on the right-hand side of equation (2).

Absorbers typically operate as countercurrent devices, where the liquid flows downward by gravity while the gas phase flows upward. Consequently, the concentration of the absorbed component in the liquid phase and also at the phase interface changes, increasing from the top to the bottom of the column , as illustrated in Figure 3. Therefore, the driving force of mass transfer also changes, and equation (3) applies to an infinitesimal surface area of the phase interface:

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

The amount of transported component into the liquid phase for an infinitesimal surface area of the phase interface can also be expressed by the material balance of the transferring component in the liquid phase:

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

where  represents the volumetric flow rate of the liquid phase.

Let's assume that the solubility of the absorbing component is low, implying that the volumetric flow rate of both the liquid and gas phases along the column remains unchanged. Based on equation (1), the concentration at the interfacial boundary will also remain unchanged . By combining equations (4) and (5) and integrating them from the top to the bottom of the column for the total surface area of the phase interface, we obtain the equation for calculating the partial coefficient of mass transfer in the liquid phase:

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

Similarly, by integrating the equation of material balance (5), we obtain its integral form:

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

In the previous equations, the concentration of carbon dioxide at the phase interface appears. This value can be obtained by several methods. Most commonly, Henry's law is applied, which is defined for the gas-liquid system as:

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

where  represents the partial pressure of the gas component at the phase interface, symbol  epresents Henry's constant, and  represents the mole fraction of the absorbed component in the liquid phase. The concentration of the component at the phase interface can be expressed as follows:

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

where  is the density and  is the molar mass of the liquid phase.

The absorption column is open to the atmosphere, so we can assume that the pressure of carbon dioxide in the column as well as at the interfacial boundary is equal to atmospheric pressure:

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

Henry's constant is a function of temperature. By combining equations (8), (9), and (10), we obtain the final relationship for calculating the concentration of the absorbed component at the phase interface:

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

When determining the value of the partial coefficient of mass transfer in the liquid phase according to equation (6), we need to know the volumetric flow rate of the liquid phase, the size of the phase interface area, and the concentration of the absorbed component in the liquid phase at the bottom and the top of the column. These values can be experimentally determined in a disk column. Based on experimental data obtained in a standard absorption column, an empirical relationship was derived in the literature for calculating the value of the partial coefficient of mass transfer in the liquid phase as a function of the physical properties of the liquid phase, hydrodynamic conditions, and characteristics of the observed system regarding mass transfer. Assuming isothermal conditions in the column, this empirical relationship takes the form:

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

where and are parameters of the empirical model, and represents the volumetric wetting intensity defined by the equation:

|  |  |  |
| --- | --- | --- |
|  |  | (13) |

where the variable  represents the average circumference of the disk wetted by the liquid phase. In the case of a disk column,  is calculated from the equation:

|  |  |  |
| --- | --- | --- |
|  |  | (14) |

where the parameter represents the diameter of the disk and  its thickness.

# Objectives of the work

Experimentally obtain data necessary to determine the value of the partial coefficient of mass transfer in the liquid phase for the carbon dioxide-water system and its dependence on the flow rate of the liquid phase.

Determine the parameters of the correlation relationship (12) by nonlinear regression. Subsequently, compare the calculated values of  from the model with experimental data. Assess the reliability of the model against the experiment.

# WORK ASSIGNMENT

1. Determine the values of the partial coefficient of mass transfer in the liquid phase, . for the carbon dioxide-water system at five liquid phase flow rates. The position of the float on the rotameter (6) to determine the carbon dioxide flow rate is *h*CO2 = ..... mm.

Calculate the values of parameters *a* and *b* in equation (12) from the obtained data. Illustrate the dependency graphically on a logarithmic scale.

# EQUIPMENT DESCRIPTION

The experiment is conducted in a laboratory, disc countercurrent absorption column, whose flow diagram is depicted in Figure 5.

Diagram

Description automatically generated

Figure 5 Diagram of the apparatus for determining the partial coefficient of mass transfer in the liquid phase: absorption column (1), pressure flask with CO2 (2), reducing valve (3), frit (4), flow control valve for CO2 (5), rotameter for CO2 (6), manostat (9), water valve (10), storage tank (11), flow control valve for water (12), rotameter for water (13), siphon (14), sampling valve (15), thermometer (16), sensor and regulator for liquid level in the tank (22), automatic water supply valve to the tank (23).

Water is dosed into the system from a storage tank (11). Water is supplied to the tank (11) from the water main through a valve (10). The level of water in the tank is maintained by a sensor and controller (22), to which a solenoid valve for water supply (23) is connected. From the tank, water passes through a needle control valve (12) and a rotameter (13) into the absorption column (1). The temperature of the water at the inlet of the column (1) is monitored by a thermometer (16). Samples of water entering the column (1) are taken through a sampling valve (15). Water from the column flows out through a siphon (14), where the temperature of the water is measured again, and a water sample is taken from the outlet of the absorber.

Diagram

Description automatically generated

Figure 6 Detail view of the disc absorption column

The column consists of a glass tube (25) with an inner diameter of 25 mm, which is sealed with stoppers (32). Along the axis of the tube, a wire (26) is stretched, on which disks (27) are perpendicular to each other. The disks have a diameter *d* = 1.5 cm and a thickness δ = 0.45 cm, with a total number of *z* = 35. The guiding wire (26) is attached to the structure (28). Water and carbon dioxide are introduced into the column in countercurrent flow. Water enters the column through the tube (29) to flow along the guiding wire (26) and disks (27) into the outlet tube (30). It then proceeds to the siphon (14), where the temperature of the water at the outlet of the column is measured, and to the waste. The siphon (14) serves as a hydrostatic seal to prevent CO2 leakage. It is adjusted so that the water level in the outlet tube (30) is 3-4 cm below its upper edge. The tap (31) is used to drain water that accumulates at the bottom of the column outside the outlet tube (31). Carbon dioxide is introduced into the column from below through the tube (33) and removed at the top of the column through the tube (34).

# WORK PROCEDURE

## SAFETY MEASURES

1. Only the instructor is allowed to manipulate the reducing valve (3) on the gas cylinder!
2. During measurements, we take care to ensure that the flow of water and carbon dioxide remains constant.

## PrEPARATION FOR MEASUREMENT

1. Familiarize yourself with the equipment and check the completeness of the accessories according to the documentation. Verify that the titration solutions and indicator are replenished. Write down the concentrations of both solutions in Tab. 1  as well as their dilution factors .

Check if the control valve (12) for water supply to the rotameter is closed and open the water supply (valve (10)) to the storage tank (11).

Turn on the level regulator in the tank (11) using the switch on the wall.

Read the atmospheric pressure from the barometer in the room  and record it in Tab. 1.

The instructor sets the reducing valve (3) so that the flow of CO2 through the manostat (9) is appropriate.

Adjust the needle control valve (5) for the supply of carbon dioxide so that the height of the float in the rotameter (6) corresponds to the desired value. Using the calibration curve  (Appendix), calculate the volumetric flow rate of carbon dioxide entering the column. Record its value in Tab. 1.

1. Record the parameters of the column in the table, *z* = number of disks, *d* = diameter of the disk,  = thickness.

## MEASUREMENT

1. Open the control valve (5) to adjust the water flow. By turning it, set the highest suitable water flow (maximum water flow at which there is no water splashing from the disks (27) onto the walls of the absorption column (25)). Determine the water flow  from the calibration curve  (Appendix).
2. Wait for the temperature, flow of CO2, and water passing through the column to stabilize.
3. At the beginning of the measurement, determine the carbon dioxide content in the water flowing into the head of the absorption column. Record the measurement in row A of Tab. 1. Read the temperature  of the water flowing into the column and the temperature  at the outlet of the siphon (14) on the thermometer (16). Record their average value in the tpriem ​ column. Take a water sample at the inlet of the column using the sampling valve (15).
4. Determination of the CO2 content in the sample using back titration:

* Between individual steps, always weigh the titration flask before and after, based on which we will know the exact weights of the added solutions.
* Add approximately 18 mL of sodium hydroxide solution to the titration flask. Record the exact weight of the added sodium hydroxide solution  in Tab. 1.
* Add directly approximately 50 mL of the sample water being determined to the titration flask with NaOH. Record its exact weight  in Tab. 1. Add 1-2 drops of indicator to the mixture.
* With continuous stirring, carefully add sulfuric acid solution until the solution is stable in color. Record the weight of the added sulfuric acid titration solution .

The first measurement, Tab. 1, is performed at maximum water flow.

We record the data about the position of the float in the rotameter for setting the water flow, , and the volumetric water flow rate, . On the thermometer (16), we read the temperature  of the water flowing into the column and the temperature  at the outlet of the siphon (14). We record their average value, . We determine the CO2 content in the water at the outlet of the column. We take a water sample at the outlet of the column from the siphon (14) and proceed with the determination of the CO2 content according to point 4.

We repeat the measurement according to point 6. at four additional volumetric water flow rates. In each subsequent measurement, we decrease the flow rate by approximately 0.2 L min⁻¹. After changing the volumetric water flow rate, we wait for the temperature and the flow of CO2 as well as the water passing through the column to stabilize.

Finally, we repeat the determination of the CO2 content in the water flowing into the absorption column according to point 3. We record the measurement in row B of Tab. 1.

## END OF MEASUREMENT

1. We stop the flow of water through the column by closing the needle control valve (5).

By closing the water tap (10), we stop the flow of water into the thermostat.

Close the needle valve (5) for adjusting the flow of CO2.

If there is any water remaining at the bottom of the column, drain it using the tap (24).

## IF YOU WISH TO REVIEW THE EQUIPMENT DESCRIPTION AND THE PROCEDURE, YOU CAN FIND IT IN THE VIDEO:

<https://www.youtube.com/watch?v=k2jPMC1fD1s&list=PL81QAQg818vjDK7pm9WO6tW36sdBpPGYa&index=5>



# QUESTIONS – what the teacher may ask you before the experiment

* What is absorption? What are its uses?
* What forms of mass transfer do you know, and what does the film theory say about it?
* What and how will you measure?
* What do you understand by the term "steady state"?
* How will you perform a back titration?

# ANALYSIS OF MEASURED DATA

1. The content of carbon dioxide in all samples will be determined by back titration. Back titration can be understood as two consecutive chemical reactions expressed by stoichiometric equations:



* Assuming 100% conversion and stoichiometric consumption of H2SO4 in the second reaction, we can calculate the amount of excess NaOH from the material balance.

|  |  |  |
| --- | --- | --- |
|  |  | (15) |

where  is the mass of the added sulfuric acid solution with a concentration of  and a dilution factor of . We assume that the density of the solution under these conditions is equal to 1000 kg/m3

* The amount of reacted NaOH in the first reaction can be determined as:

|  |  |  |
| --- | --- | --- |
|  |  | (16) |

where  is the mass of the added sodium hydroxide solution with a concentration of  and a dilution factor of . We assume that the density of the solution under these conditions is equal to 1000 kg/m3.

* Assuming 100% conversion of CO2 in the first reaction, then we can calculate the amount of CO2 in the specified sample:

|  |  |  |
| --- | --- | --- |
|  |  | (17) |

* We will calculate the concentration of CO2 in the samples using the formula:

|  |  |  |
| --- | --- | --- |
|  |  | (18) |

We substitute the density of the sample with the density of water at average temperature tpriem. We write down the calculated concentration of CO2 at the outlet of the column in the liquid phase in the column  (Tab. 2) for each measurement.

We determine the concentration of carbon dioxide in the liquid phase at the inlet of the column as the average value from the measurement of concentration in the inlet water (experiment 'A-B'). We write down this value in Tab. 2.

We will calculate the equilibrium concentration of carbon dioxide at the phase interface  using equation (11). We will find the value of the Henry's constant at the average temperature of measurement in the chemical engineering tables (page 82). The concentration will be recorded in Tab. 2.

We will calculate the approximate area of the phase interface from the equation below and record it in Tab. 2.

|  |  |  |
| --- | --- | --- |
|  |  | (19) |

where  represents the number of disks,  stands for the diameter of the disk, and  represents its thickness.

For each volumetric flow rate, we will calculate the value of the partial mass transfer coefficient in the liquid phase  according to equation (6). The values will be recorded in Tab. 2.

From equations (13) and (14), we will calculate the average circumference of the disk and for each measurement, we will also calculate the volumetric wetting intensity. The values of these parameters will be recorded in Tab. 2.

We will determine the values of the parameters of model (12) through regression and record them in Tab. 2.

We will construct a graph showing the dependence of , where we will compare the determined values of the partial mass transfer coefficient (points) from experimental data and those calculated by the model (line), depending on the wetting intensity. Both scales on the diagram are logarithmic

# TABLES

Tab. 1 Record of measured data

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| *P*atm  (Pa) | (mL min-1) | *z*  (-) | *d*  (mm) | (mm) | *C*NaOH  (mol L-1) | *f*NaOH  (-) | *C*H2SO4  (mol L-1) | *f*H2SO4  (-) |
|  |  |  |  |  |  |  |  |  |
| Measurement | Water flow rate | | Temperatures | | | Titration | | |
| *h*l (mm) |  | *t*2 (°C) | *t*1 (°C) | *t*priem (°C) | *m*NaOH (g) | *msample* (g) | *m*H2SO4 (g) |
| A |  |  |  |  |  |  |  |  |
| 1 |  |  |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |  |  |
| 4 |  |  |  |  |  |  |  |  |
| 5 |  |  |  |  |  |  |  |  |
| B |  |  |  |  |  |  |  |  |

Tab. 2 Results of data evaluation

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| A = | (cm2) | | Oz = | | (cm) | m = | (-) | | a = | | (-) |
| Measurement | | *C*CO2(1) (mol L-1) | | *C*CO2(2) (mol L-1) | | *C*CO2f (mol L-1) | | *k*lc (m s-1) | | *I* (m3 s-1) | |
| 1 | |  | |  | |  | |  | |  | |
| 2 | |  | |  | |  | |  | |
| 3 | |  | |  | |  | |  | |
| 4 | |  | |  | |  | |  | |
| 5 | |  | |  | |  | |  | |

ATTACHMENTS



