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Treball Final de Grau

Study of the operating variables of an absorption column in the CO_2 /water system for the EEQ2 practices

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Libros, caminos y días dan al hombre sabiduría

Proverbio árabe

Quiero aprovechar para dar mi más sincero agradecimiento a ambos de mis tutores Eliana Ramírez y Bernardí Bayarri, por depositar su confianza en mí, por dedicar su tiempo y brindar su apoyo tanto en el laboratorio como en la aportación de sus conocimientos y sugerencias que han sido fundamentales para este trabajo. Agradecer también a los técnicos de laboratorio por el apoyo y la facilitación del material de trabajo.

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SUMMARY

The Department of Chemical Engineering has acquired an experimental system (CE 400) for the absorption of carbon dioxide (CO_2) in water, with the aim of providing students with a practical tool to study phenomena of mass and energy transfer. The equipment consists of three partial processes: gas mixing, absorption and desorption. The absorption is carried out in a packed column fed with water and a gas, pure CO_2 or CO_2/air mixture, in order to investigate the behaviour of CO_2 absorption as a function of different operating conditions.

The fundamental objective of this project is to put the installation into operation and to carry out a systematic study of the effect of the different variables that influence the CO_2 absorption process. These variables include the water flow rate, the gas flow rate, the composition of the feed gas (percentage of CO_2 in the air) and the temperature of the water.

To achieve this objective, the equipment is previously implemented, ensuring that it is correctly installed and calibrated. Next, a study of the process variables is carried out adjusting different water flow rates, gas compositions and water temperatures.

Finally, the main goal of this TFG project is the formulation of a guide for the experimental laboratory practices implemented in the subject Experimentation in Chemical Engineering 2 (EEQ2).

Keywords: absorption, mass and energy transfer, carbon dioxide, water, solubility.

RESUM

El Departament d'Enginyeria Química ha adquirit un sistema experimental (CE 400) per a l'absorció de diòxid de carboni (CO_2) en aigua, amb l'objectiu de proporcionar als estudiants una eina pràctica per estudiar fenòmens de transferència de massa i energia. L'equip consta de tres processos parcials: barreja de gas, absorció i desorció. L'absorció es porta a terme en una columna de rebliment alimentada amb aigua i un gas, CO_2 pur o una barreja de CO_2/aire, amb la finalitat d'investigar el comportament de l'absorció de CO_2 en funció de diverses condicions operatives.

L'objectiu fonamental d'aquest projecte és posar en funcionament la instal·lació i realitzar un estudi sistemàtic de l'efecte de les diferents variables que influeixen en el procés d'absorció de CO_2. Entre aquestes variables es troben el cabal d'aigua, el cabal de gas, la composició del gas d'alimentació (percentatge de CO_2 en l'aire) i la temperatura de l'aigua.

Per a aconseguir aquest objectiu, es realitza una posada en marxa prèvia de l'equip, assegurant la seva correcta instal·lació i calibratge. A continuació, es realitza un estudi de les variables del procés ajustant diferents cabals d'aigua, composicions de gasos i temperatures de l'aigua.

Finalment, l'objectiu principal d'aquest projecte de TFG és la formulació d'una guia per a les pràctiques experimentals de laboratori implementades en l'assignatura Experimentació en Enginyeria Química 2 (EEQ2).

Paraules clau: absorció, transferència de massa i energia, diòxid de carboni, aigua, solubilitat.

SUSTAINABLE DEVELOPMENT GOALS

The development of this TFG responds to two of the sustainable development goals from the United Nations Environment Programme, the quality education and the climate action.

Quality education. By putting the CO_2 absorption system into operation and integrating it into the curriculum (specifically in the EEQ2 course), the project enhances the practical education of chemical engineering students. It promotes hands-on learning, critical thinking, and understanding of environmental technologies.

Climate action. One of the 10 objectives of green and sustainable chemistry is to minimise chemical emissions and pollution, which coincides with one of the main applications of the equipment, to reduce the emission of polluting gases from a gaseous stream coming out of a process such as combustion.

In this work the effect of different parameters on the absorption of CO_2 by water is evaluated, it is therefore important to study these parameters to increase the efficiency of the absorption of these gases and/or for an optimal design of the absorption columns.

1. INTRODUCTION

1.1. ABSORPTION PROCESS

According to Seader and Henley (1998), the absorption (also called gas scrubbing) is a unit operation where a gas mixture is contacted with a liquid (the absorbent or solvent) to selectively dissolve one or more components by mass transfer from the gas to the liquid. The components transferred to the liquid are referred to as solutes or absorbate. The reverse process of absorption is the stripping (also called desorption) and consists of eliminating a volatile component from a liquid solution, where the agent is a stream of dry hot air that desorbs dragging the component contained in the initial liquid current

Depending on the type of solute/solvent absorption system, there is a classification between physical absorption, where the absorption is due to the dragging of the component in the gas by the liquid through intramolecular interactions. Or chemical absorption where the component in the gas is absorbed by reaction with the liquid.

1.1.1. Applications

Absorption has many uses in industry. It is used to separate gas mixtures; remove impurities, contaminants, pollutants, or catalyst poisons from a gas; or recover valuable chemicals (Seader and Henley 1998).

Absorption is mainly used to reduce the emission of pollutant gases from an outgoing gas stream of a process such as combustion. It is also used in industry for the extraction of chemicals from liquid or gaseous streams. It is particularly useful in obtaining valuable products from complex mixtures.

As for desorption is commonly used to remove impurities and unwanted components from liquids as a purification process. Examples are the removal of odours, fumes and other toxic components. And to regenerate solvents, in various industrial processes, solvents are used which, over time, can become less effective due to the accumulation of impurities. To reuse them and minimise waste, absorption is used in the regeneration of these chemicals.

Industrial example of absorption applications

An industrial example of absorption is the reduction of CO_2 from various gas industrial streams, including flue gas emitted by power plants and industrial processes. As it is mentioned in the article, Carbon Capture Science and Technology published in 2023, during the last years the concerns about the climate change have been raising leading to a faster development of a sustainable industry with the objective to reduce the greenhouse gas (GHG) emissions, leading to the importance CO_2 separation technologies.

The absorption is one of the methods for post-combustion CO_2 capture. This operational process is one of the most mature methods of reducing this solute due to its high processing capacity, excellent adaptability, and reliability. It has been made a significant progress in recent years studying different physical, chemical or combined solvents for this process (Borhani and Wang, 2019).

The process typically takes place in a packed column. The gas flows through the column, it interacts with the absorbent, and the CO_2 molecules are absorbed into the liquid phase, leaving the other gasses behind. Once the CO_2 has been absorbed, the solvent is then transferred to a regeneration unit, where it is heated to release the captured CO_2 (de Meyer and Jouenne, 2022). The captured CO_2 can be compressed and stored or utilized for other applications.

Another example of an absorption industrial application is the recovery of ammonium and aromatic compounds (benzene, toluene and xylene) that accompany the lighting gas obtained in coke ovens by dry distillation of the bulla. The gas is first passed through water to separate the ammonium compounds and then through oil to absorb the aromatics.

1.1.2. Absorption equipment

The absorption equipment is designed with intent to favour the contact of both phases, to reduce gas pressure losses and avoid preferential channels for the gas to pass through.

Absorption and desorption processes are carried out mainly in trayed towers and packed columns, and less often in spray towers, bubble columns, and centrifugal contactors (Seader and Henley 1998). In an absorption tower usually, the inlet gas stream flows counter current to the liquid, the gas is introduced from the bottom and rises due to the pressure difference between the inlet and outlet of the column.

Packed column

Packed columns are used for continuous contact of gas and liquid. The absorption tower consists of a cylindrical column, of larger height than diameter, in which the gas normally enters from the bottom following the distribution space below the packed section and rises through the openings or the packing interstices and exits at the top. The other way round, the liquid enters from the top, is poured using a liquid distributor ensuring a uniform distribution of liquid over the cross-sectional area of the column and flows through the same packing interstices and is collected at the bottom.

As Seader and Henley mention, there are many different types of tower packings that have been developed. It is possible to use randomly poured packings, such as an open structure block or such as 'Mellapak'. These structures contain fillers that can have different shapes, each presenting a contact surface, e.g. Raschig ring, sadder, Pall ring and Lessig ring. Or it also possible to use a structured packaging, which is more compact in size although it is more expensive.

The packing in the column provides a large surface area for intimate contact between the gas and liquid, enhancing heat and mass transfer. According to Smith and Mccabe (2018), the absorption fillers must meet certain minimum requirements. They must be chemically inert to the tower fluids, mechanically resistant without being too heavy, allow good phase contact and avoid excessive liquid retention or significant pressure drop.

Packings come in different nominal diameter sizes, and as their size increases, the efficiency of mass transfer and pressure drop can decrease.

Therefore, for a given column diameter there is an optimum packing size that represents a compromise between these two factors, low pressure drops and high mass transfer rate.

Trayed towers

A plate column, typically used in distillation, is composed of multiple perforated plates configured to allow vapours to rise through a layer of fluid, as well as fluids to descend in the opposite direction to the vapour. The liquid flows through each plate and falls through a downstream collector to the bottom plate. The gas flows up through the openings in each plate, bubbling through the liquid.

As Seader and Henley mention, for most of the absorption applications, either packed columns or tray towers are used. The packed column is preferred when the column diameter is less than 2 feet (0.6 meters), and the packed height does not exceed 20 feet (6.1 meters) and for high liquid velocities. Tray towers, on the other hand, are preferred for more reliable design and scaling. They are quite expensive structures but are a good choice for a new installation when the pressure drop must be very low or to replace existing trays. Tray towers are preferred when liquid velocities are low.

2. OBJECTIVES

The main objectives in this work are the following:

To operate the CE400 installation for the absorption of CO_2 in water, ensuring the correct installation and calibration of the equipment. This includes the verification of the operation of the pressure manometers, heat exchanger, flow measurement systems and CO_2 sensors.

To study the effect of the water flow rate and temperature of the water flow with a supply of pure CO_2 in the inlet gas current on the global volumetric coefficient of mass transfer.

To study the effect of the water flow rate and composition in the inlet gas current with a supply of a CO_2 /air mixture in the inlet gas current on the global volumetric coefficient of mass transfer.

And finally, to present a proposal for an experimental practice to be developed in the EEQ2 subject.

3. THEORICAL FOUNDATIONS

In the following section, the most relevant theoretical models described for the absorption operation will be explained, followed by the mass balances and the design equation for a packed absorption column.

3.1. DOUBLE FILM THEORY

In a column in which a gas and a liquid circulate in contact, and the concentration of the solute in the gas is higher than that corresponding to the equilibrium with the liquid, a transfer of solute from the gas to the liquid occurs. As it is represented in the Figure 1, the solute transfer is initiated from the sine of the gas where the solute first reaches the interface (*if*) by diffusion. The interface, a layer of molecules in which gas/liquid are in contact molecule by molecule forming a layer of laminar flow, the transfer of mass occurs rapidly reaching a thermodynamic equilibrium. And subsequently the solute is transferred by diffusion from the interface to the liquid phase sine.

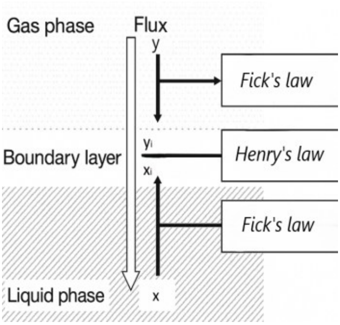


Figure 1. Mass transfer across a gas–liquid interface.

The double film theory proposed by Whitman in 1923 describes this solute transfer as a transfer through a series of two resistances corresponding to the gas and liquid phases (Smith and Mccabe, 2018).

When looking at the concentrations of the solute along the phases, a concentration gradient is observed, with the solute concentration in the gas being higher than at the interface, and that at the interface higher than within the liquid. It is precisely this gradient the driving force of the mass transfer process.

As mentioned in Fick's diffusion law (1), in a system in which concentration gradients exist, the solute in the gas follows, relative to the global movement of the mixture, in the direction of the negative concentration gradients (Mans Teixidó et al. 1998).

$$J_i = -D\frac{\partial c_i}{\partial x} \quad (1)$$

where:

J_i = molar flux of solute i (mol/s)

∂c_i = concentration gradient of solute i (mol/m^3)

∂x = thickness (m)

D = diffusivity of the solute i in the medium (m^2/s)

Therefore, the mass transfer will depend on the difference in concentrations between the gas sine of the gas and the interface and the distance between them, and the same is applicable between the liquid sine and the interface.

3.2. EQUILIBRIUM

3.2.1. Raoult's law

As Correa Noguez (2004) mentions, in a gas-liquid system, ideal mixture, in which there is a common component in both phases and where there is a thermal, pressure and composition equilibrium Raoult's law (2) is applicable for the dissolved gas.

$$p_i = x_i \cdot p_i^o(T) \quad (2)$$

where:

p_i = partial pressure of solute i gas (Pa)

p_i^o = vapour pressure of the pure solute i at the system temperature (K)

x_i = mole fraction of the solute i on the liquid phase

3.2.2. Henry's law

As Correa Noguez remarks, to obtain an approach to describe gas solubility, an ideal dilute solution is considered in which the gas component is dissolved in an excess amount of solvent. For the gas solubility of component *i* in the solvent, Henry's Law (3) is applied for ideal dilute solutions.

$$p_i = H_{i,px} \cdot x_i \quad (3)$$

Henry's constant (H_i) is a measure of the absorption capacity from the gas phase to the liquid phase where the volatile substance is in equilibrium with the liquid phase. There are several versions of Henry's Law, in Table 1 there are some examples with the respective units given by J.Boxhammer in 2019.

Table 1. Some versions of Henry's Law with constants (gas in water at 298 K).

Equation	$H_{i,cp}=\frac{c_{i,aq}}{p_{gas}}$	$H_{i,pc}=\frac{p_{gas}}{c_{i,aq}}$	$H_{i,px}=\frac{p_{gas}}{x_i}$	$H_{i,cc}=\frac{c_{i,aq}}{c_{i,gas}}$
Units	$\frac{mol_{gas}}{atm \cdot L}$	$\frac{atm \cdot L}{mol_{gas}}$	$\frac{atm \cdot mol_{aq}}{mol_{gas}}$	1

(a) ci,aq is the concentration of the solute i that the liquid contains
(b) ci,gas is the concentration of the solute i that the gas contains

Henry's constant is actually not constant, since it changes with temperature, which is why it is also called Henry's coefficient. Its values can be found tabulated, or there are formulations that relate this dependence, an example is expression (4).

$$H_{i,cp} = H_{i,cp,T25} \cdot e^{\left(C \cdot \left(\frac{1}{T} - \frac{1}{T25}\right)\right)} \quad (4)$$

where:

T_{25} = standardised temperature 25 ºC (298 K)

C = constant for gas CO_2 (2400 K)

For the graphical representations of the equilibrium diagrams, it is convenient to change the values of the constants $H_{i,cp}$ to $H_{i,px}$ using the equation (5).

$$H_{i,px} = \frac{p_{gas}}{x_i} = \frac{\rho_{aq}}{M_{aq} \cdot H_{i,cp}} \quad (5)$$

where:

M_{aq} = molecular mass of the liquid phase (g/mol)

ρ_{aq} = density of the liquid phase (kg/m^3)

3.3. GRAPHICAL REPRESENTATION

To represent the equilibrium curves of gas-liquid systems with a common component, the graphical representation partial pressure of the component versus its mole fraction is used.

However, as Correa Noguez notes, in absorption, the most common diagrams used for the graphical representation of phase equilibrium are the composition diagrams, where the concentrations of the solute in the gas versus in the liquid are represented.

3.3.1. Equilibrium diagram y_i vs x_i

This diagram shows the physical characteristics of substances in a linear form. Dividing both terms of the equality of equation (3) by the total pressure gives the expression (6).

$$y_i = \frac{H_{i,px}}{P} \cdot x_i \quad (6)$$

where:

P = total pressure (Pa)

y_i = mole fraction of the solute *i* on the gas phase

By looking at the expression the approximation to a straight line (y = m * x) can be made, where its slope (m) is equal to Henry's constant divided by the pressure.

3.3.2. Equilibrium diagram Y_i vs X_i

In the equilibrium diagram using mole-loadings (Y_i vs X_i) when the equilibrium expression is represented takes a non-linear tendency, yet it is still used because of its advantage over representing the balance expression representation as a linear tendency.

The following definitions apply:

$$Y_i = \frac{Molar\ flow\ rate\ of\ the\ gas\ component\ to\ be\ absorbed\ i}{Molar\ flow\ rate\ \ of\ the\ residual\ inert\ gas\ mixture}$$

$$X_i = \frac{Molar\ flow\ rate\ of\ the\ gas\ component\ to\ be\ absorbed\ i}{Molar\ flow\ rate\ of\ pure\ solvent}$$

For the conversion of mole-loadings to mole fractions, the expressions (7) for the gas phase and (8) for the liquid phase are applicable.

$$y_i = \frac{Y_i}{1+Y_i} \text{ (7)}$$

$$x_i = \frac{X_i}{1+X_i} y \text{ (8)}$$

If the expressions for x_i and y_i are substitute into the equilibrium equation (6) the linear equations (9) and (10) for both phases in the equilibrium are obtained.

$$Y_i = \frac{\frac{H_{i,px}}{P}\cdot X_i}{1+\left(1-\frac{H_{i,px}}{P}\right)\cdot X_i} \quad \text{(9)}$$

$$X_i = \frac{Y_i\cdot\frac{P}{H_{i,px}}}{1+(1-\frac{P}{H_{i,px}})\cdot Y_i} \text{ (10)}$$

The temperature and pressure dependence of the absorption equilibrium has been seen above with Raoult's Law and Henry's Law. Meaning that for each temperature and pressure there is a linear equation.

3.4. ABSORPTION RATE APPLYING THE DOUBLE FILM THEORY

3.4.1. Individual coefficients of mass transfer

According to Geankoplis in 1998, the flux density of the solute i (N_i) is the average number of moles of i that are transferred per area of the interface per unit time. It is proportional to the driving force which is the difference between the composition on the sins of the liquid/gas phases and the interface.

The individual coefficients of mass transfer (k), indicate the average facility with which mass can be transferred, the higher the coefficient, the higher the transfer capacity.

The different expressions for the flux densities and the individual coefficients of mass transfer for the gas and liquid phase are given by the formulations in the Table 2. For all cases, the units of N_i will be the same by changing only the units of the individual coefficients.

Table 2. Expressions of flux density and individual coefficient of mass transfer in gas and liquid phases.

	N_i [mole i / m^2 · s]	**Coefficients**
Gas:	$N_i = k_g(p_i - p_i^{if})$	k_g
	$N_i = k_y(y_i - y_i^{if})$	$k_y = k_g \cdot P$
Liquid:	$N_i = k_l(c_i^{if} - c_i)$	k_l
	$N_i = k_x(x_i^{if} - x_i)$	$k_x = k_l \cdot \rho_M$

where:

p_i^{if} = partial pressure of the solute i at the interface in the gas phase (Pa)

c_i^{if} = concentration of the solute i at the interface in the liquid phase (mol/m^3)

y_i^{if} = mole fraction of the solute i at the interface in the gas phase

x_i^{if} = mole fraction of the solute i at the interface in the liquid phase

k_g /k_y = individual coefficients of mass transfer in the gas phase

k_l /k_x = individual coefficients of mass transfer in the liquid phase

There are also two types of coefficients, the individual coefficients of mass transfer (k), which have already been described, and the global coefficients of mass transfer (K).

3.4.2. Global coefficients of mass transfer

As Geankoplis continues describing, the use of global coefficients of mass transfer is due to the fact that the composition at the interface (x^{if}, y^{if}) cannot be measured, meaning that the differences ($x - x^{if}$) or ($y - y^{if}$) cannot be taken as the driving forces. If instead of looking at the individual coefficients of mass transfer for each phase, a more global view of the solute absorption is taken, the global coefficients of mass transfer for the gas phase (K_G) and the gas phase (K_L) can be used as both expressions are (11) and (12).

Global coefficient for gas: $$\frac{1}{K_G} = \frac{1}{k_g} + \frac{H}{k_l} \quad (11)$$

Global coefficient for liquid: $$\frac{1}{K_L} = \frac{1}{k_l} + \frac{1}{k_g H} \quad (12)$$

The expressions of both coefficients are assimilated to the expression for a global resistance (1/K) that sums two resistances of each phase in series.

With the expressions of global coefficients of mass transfer and transforming the previous Ni expression adding Henrys Law, the new expression N_i (13) is obtained.

$$N_i = K_G \cdot P(y_i - y_i^e) = K_L \cdot \rho_M (x_i^e - x_i) \quad (13)$$

where:

y_i^e = mole fraction in the gas of component i in the equilibrium

x_i^e = mole fraction in the liquid of component i in the equilibrium

Considering the nature of the equation of equilibrium in the form of a straight line, the expression for N_i (14) can also be used,

$$N_B = K_y(y_i - y^e) = K_x \cdot \rho_M (x_i^e - x_i) \quad (14)$$

changing the expressions for also the respective global coefficients of mass transfer in each phase (15) and (16) with the slope (m) of the straight line.

Global coefficient for gas: $\frac{1}{K_y} = \frac{1}{k_y} + \frac{m}{k_x}$ (15)

Global coefficient for liquid: $\frac{1}{K_x} = \frac{1}{k_x} + \frac{1}{k_y m}$ (16)

3.4.3. Absorption rate

The expression for the absorption rate can be obtained considering the fact that, for each differential area of mass transfer there is a flux density of solute transfer. The flow rate is given as a differential equation (17).

$$dW_i = N_i dA = K_G \cdot P \cdot (y_i - y_i^e) dA = K_L \cdot \rho_M \cdot (x_i^e - x_i) dA \quad (17)$$

where:

dW_i = absorption flow rate (mol/s)

dA = area of transference (m^2)

The surface can be calculated using the specific filling area (a) with the expression (18). This value depends on the type of filling, its dimensions and material and can be expressed as the transfer area per filling volume.

$$dA = a \cdot S \cdot dV \quad (18)$$

where:

a = specific filling area (m^{-1})

S = cross-sectional area of the abosrtion column (m^2)

dV = absorption column volume (m^3)

Substituting expression (18) to (17), the final expression of the absorption flow rate (19) can be obtained.

$$dW_i = K_y a (y_i - y_i^e) S dz = K_x a (x_i^e - x_i) S dz \quad (19)$$

3.5. DESIGN OF ABSORPTION COLUMNS IN DILUTE SYSTEMS

In order to obtain the design equations for packed columns, it is first necessary to obtain the operation line equation which is deduced from a previous global and solute mass balance.

3.5.1. Mass balance and the balance line

The mass balance is performed for dilute systems where the solute concentration is low and for a countercurrent flow. To obtain the global balance, all the inlet an outlet currents of both phases are considered as shown in Figure 2, obtaining the expression (20) where no chemical reaction takes place.

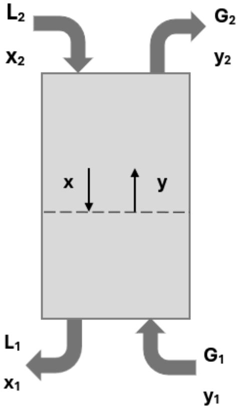


Figure 2. Representation of the flow inlets and outlets in an absorption column.

$$L'_1 + G'_2 = L'_2 + G'_1 \quad (20)$$

where G is the flux of moles (mol/m^3) in the gas phase and L is the flux of moles in the liquid phase at the levels “1” and “2”.

For the solute, the mass balance follows the expression (21)

$$L'_1 x_1 + G'_2 y_2 = L'_2 x_2 + G'_1 y_1 \quad (21)$$

If the balance for the solute is carried out, from any level below ‘2’ to the level “1” assuming that at any height of the column the gas is in equilibrium with the liquid and consider that the gas molar flow rate G is almost constant, as well as the liquid molar flow rate L, the operating line (22) is obtained.

$$y = y_1 + \frac{L\prime}{G\prime}(x - x_1) \quad (22)$$

The operating line represents a straight line in the equilibrium diagram where its slope is given by the quotient of the molar flow rates of the two phases (L/G).

3.5.2. Representation of the operation line

If the operating line is represented along with the equilibrium curve on the same X vs Y diagram the representation in the Figure 3 is obtained.

For small solute concentrations and practically constant phase molar flow rates the values of molar fractions (x_i, y_i) and mole-loadings (X_i, Y_i) are the same. This means that at low solute concentrations, the expression (22) takes the form of a line while the equilibrium expression takes form of a curve.

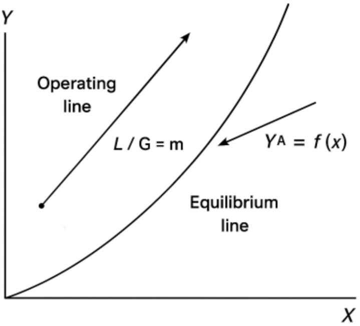


Figure 3. Representation of the operating line along the equilibrium curve in the same diagram X vs Y.

3.5.3. Design equation

For packed absorption columns that work in continuous operation, the packed height is determined involving mass-transfer coefficients for the liquid and vapor phases (Seader and Henley). To calculate such height, the equations (23) for the gas phase and (24) for the liquid phase are used. The use of one or the other depends on the phase that has a higher resistance. If the controlling phase is the gas (23) is used, an if the controlling phase is the liquid (24) is used. If simultaneously phases control the mass transfer, then either of the two can be used.

$$\text{Gas phase:} \qquad Z = \int_{y2}^{y1} \frac{d(G\prime \cdot y)}{K_y a(y - y^e)S} \quad (23)$$

$$\text{Liquid phase:} \qquad Z = \int_{x2}^{x1} \frac{d(L\prime \cdot x)}{K_x a(x^e - x)S} \quad (24)$$

where:

L'/G' = molar flow rate of liquid and gas respectively (mol/m^3)
x/y = molar fractions of liquid and gas respectively
x^e/y^e = molar fractions of the liquid and gas respectively at equilibrium
S = cross-sectional area of the column (m^2)
$K_x \cdot a$ = global volumetric coefficient of mass transfer for the liquid phase
$K_y \cdot a$ = global volumetric coefficient of mass transfer for the gas phase

These expressions can be simplified by considering that the global coefficients of mass transfer are constant along the column and by considering that the mole fraction of CO_2 in the liquid current is very low, the expressions then become (25) and (26).

$$G\approx \text{cte} \qquad Z=\frac{G'/S}{K_y a}\int_{y2}^{y1}\frac{dy}{(y-y^e)} \quad (25)$$

$$L\approx \text{cte} \qquad Z=\frac{L'/S}{K_x a}\int_{x2}^{x1}\frac{dx}{(x^e-x)} \quad (26)$$

If the equilibrium and operation equations are linear at the operation solute concentrations, the integral of the equations for Z can be solved using the average logarithmic. In those cases where this is not applicable, numerical resolution is employed considering the equilibrium equation.

For the supply of a gas stream with the pure solute to be absorbed, the equations for Z can be solved using the expressions (27) for the gas phase and (28) for the liquid phase.

$$G\approx \text{cte} \qquad Z=\frac{G'/S}{K_y a}\ln\left(\frac{y_1-y^e}{y_2-y^e}\right) \quad (27)$$

$$L\approx \text{cte} \qquad Z=\frac{L'/S}{K_x a}\ln\left(\frac{x^e-x_2}{x^e-x_1}\right) \quad (28)$$

4. MATERIALS AND METHODS

For the experiments carried out, a system was used in which the diluent gas was air, the solvent liquid was water and the solute to be transferred was CO_2. The air used was taken from the atmosphere by a compressor, the water was deionised from the faculty's network, and the CO_2 came from a gas cylinder with a purity between 99.5% -100% supplied by Linde.

4.1. SOLUTE: CARBON DIOXIDE

Carbon dioxide (CO_2) is a colourless, odourless, non-flammable and slightly acidic gas. It is a molecule consisting of one carbon atom bonded to two oxygen atoms and has a molecular mass of 44 g/mol. Due to its linear geometry; despite having polar bonds, it is a non-polar molecule, soluble in water. It is in a gaseous state at ordinary temperature and pressure and its vapour pressure at 20°C is 57.3 bar.

The CO_2 is supplied from a cylinder located outside the building in a cabinet connected by pipes to the equipment. The cylinder safety data sheet can be found on the Table 3.

Table 3. Information from the safety data sheet of the compressed carbon dioxide supplied by Linde.

Product identification	
Name	Carbon dioxide
CAS Nº	24-38-9
Purity	99.5% -100%
Classification of the substance or mixture	
Compressed gas: H280	
Identification of the hazard(s)	
Hazard pictograms (GHS):	 GHS04
Warning words (GHS):	H280: Contains gas under pressure; danger of explosion if heated. OSHA - H01: May displace oxygen and cause rapid asphyxiation. CGA - HG01: May cause freezer burns.
Precautionary statements (GHS):	P202: Do not handle until all safety precautions have been read and understood. P261: Avoid breathing gas. P262: Avoid contact with eyes, skin and clothing. P271+P403: Use and store only outdoors or in well-ventilated areas. CGA-PG05: Use a backflow prevention device in the pipeline. CGA-PG06: Close valve after each use and when empty CGA-PG02: Protect from sunlight

4.2. CARRIER: AIR

Air is a homogeneous gas mixture with an ideal compartment, composed mainly of nitrogen, oxygen and argon, but also containing other components, consult Table 4. Its molecular mass is approximately 28.96 g/mol and its density depends on ambient temperature and pressure. It is colourless, odourless, tasteless and transparent. It is also temperature sensitive, expanding with heat and contracting with cold.

Table 4. Chemical composition of air in volumetric percentages.

Gas	Formula	Volume fraction in %
Nitrogen	N_2	78.08
Oxygen	O_2	20.95
Argon	Ar	0.93
Carbon dioxide	CO_2	0.04
Water vapor	H_2O	*varying amounts*
Trace amounts of other gases, such as: Helium (He), Neon (Ne), Methane (CH_4), Krypton (Kr), Hydrogen (H_2), Xenon (Xe), Ozone (O_3), Nitrogen dioxide (NO_2), Iodine (I_2), Carbon monoxide (CO)		

The air used comes from the atmosphere itself driven by means of a compressor, therefore, environmental conditions may slightly modify the volumetric percentages of its composition.

4.3. ABSORBENT: WATER

Water is a substance which molecule is composed of two hydrogen atoms and one oxygen atom, with a molecular mass of 18 g/mol. At room temperature 25°C and atmospheric pressure it is in a liquid state.

The chemical properties of water are due to its molecular structure, a non-linear geometry, with the two hydrogen atoms forming an angle to each other. Due to its polarity, water in the liquid state is a very strong solvent for many different substances, which is why it is also used for the absorption of CO_2 gas.

The distilled water is supplied from the faculty's own network. Is specifically used the distilled one for the tests to obtain more accurate measurements and to reduce the error that can be caused by the condition of the network water.

4.4. SOLUTE AND ABSORBENT SOLUBILITY

In the absorption process the solute does not condense at the working temperatures, so it is not possible to initiate the saturated vapour pressure or to describe the phase equilibrium. Therefore, solubility is used to determine the solute compositions in the phases using Henry's Law. When the Henry's Law is used along with Dalton's partial pressure the expression (6) is obtained, where the Henry's coefficient depends on the temperature and the solute, see expression (2).

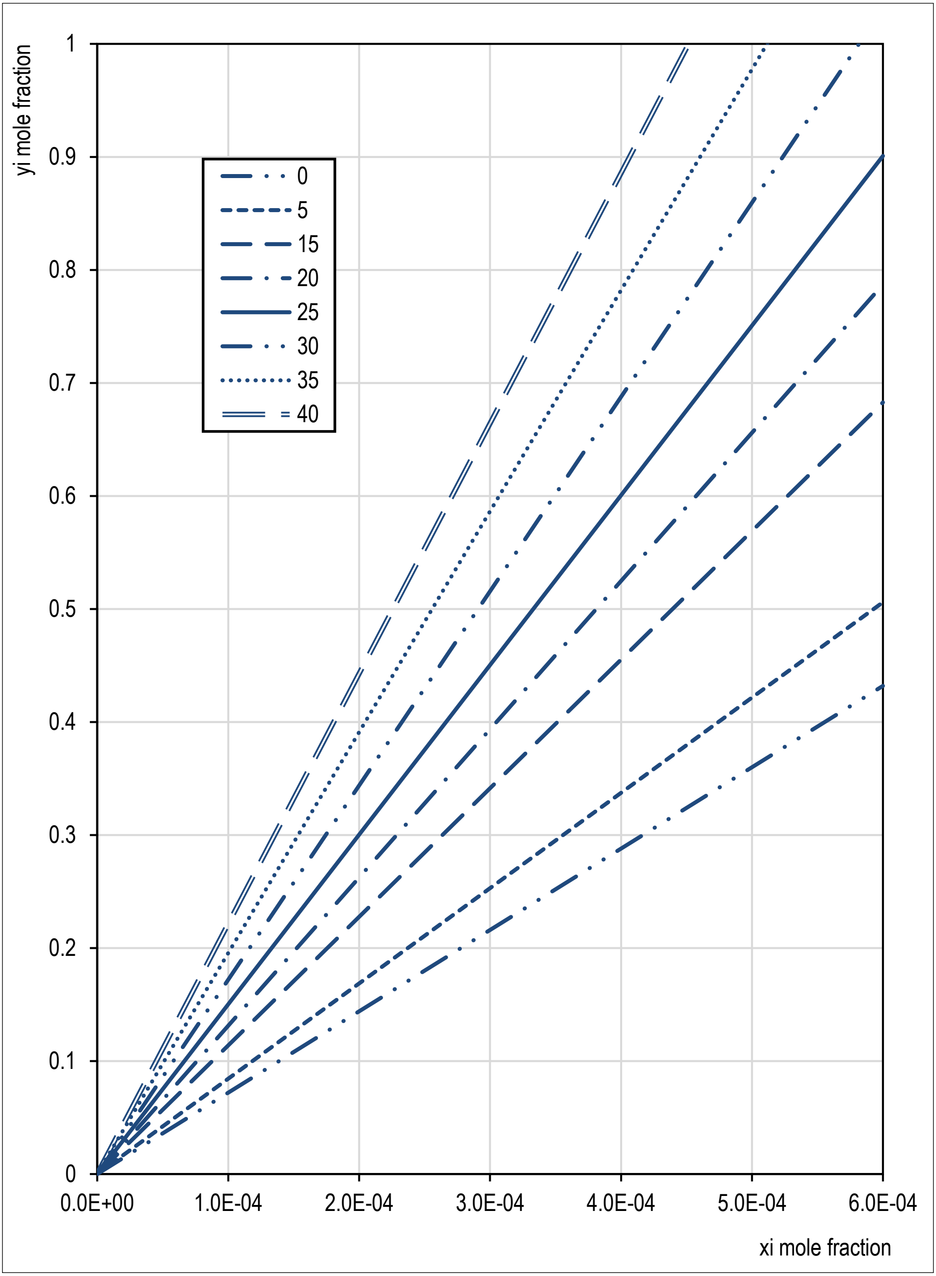
Using both previous expressions for each temperature, the equilibrium line that correlates the CO_2 concentrations in water to the CO_2 concentration in the gas phase can be obtained. In Table 5 the Henry coefficients of CO_2 are calculated for different temperatures and at the diagram in Figure 4 the representation of the equilibrium lines at different temperatures at a pressure of 1.1 bar are shown.

Table 5. Henry's coefficients for different temperatures.

T *(ºC)*	0	5	10	15	21	25	30	35	40
$H_{i,px}$ *(T in Kelvin)*	782	915	1066	1235	1464	1634	1866	2122	2404

As can be seen from the diagram, the higher the temperature, the steeper the slope of the equilibrium line. For example, for the mole fraction of 0.5 in the gas phase, the lower the temperature is the higher the mole fraction of CO_2 in water that can be reached, meaning that solubility increases at lower temperatures.

Figure 4. Equilibrium diagram for CO_2/water at 1.1 bar.



4.5. EXPERIMENTAL SETUP

The CE 400 equipment used for the experimentation is a laboratory equipment designed by the company Gunt Humburg to demonstrate and study the absorption process of separating gases using water as a solvent. In Figure 5, the flow chart for the equipment is represented, the equipment symbols and valves are listed in the Appendix 1 and their nomenclature and use are listed in the Appendix 2.

CE400 is an illustrative plant for the absorption of gases, in this case the separation of a mixture of air and carbon dioxide CO_2 by using water. The system can be divided into three sub-processes: the gas mixture, the absorption and the desorption.

The first sub-process consists of mixing air and CO_2, and it is carried out manually by regulating both gas flows using two valves. In the absorption stage, the gas mixture is circulated in counterflow with the water in which the CO_2 is washed out, for this purpose a two-part glass packing column with two upper and lower sections is employed. Lastly, in the desorption stage, CO_2 is removed from the water in a glass column using a slight depression to recirculate it.

The equipment also has a series of instruments for measuring pressures, pressures drop, temperatures, and different sampling points for both gas and liquid.

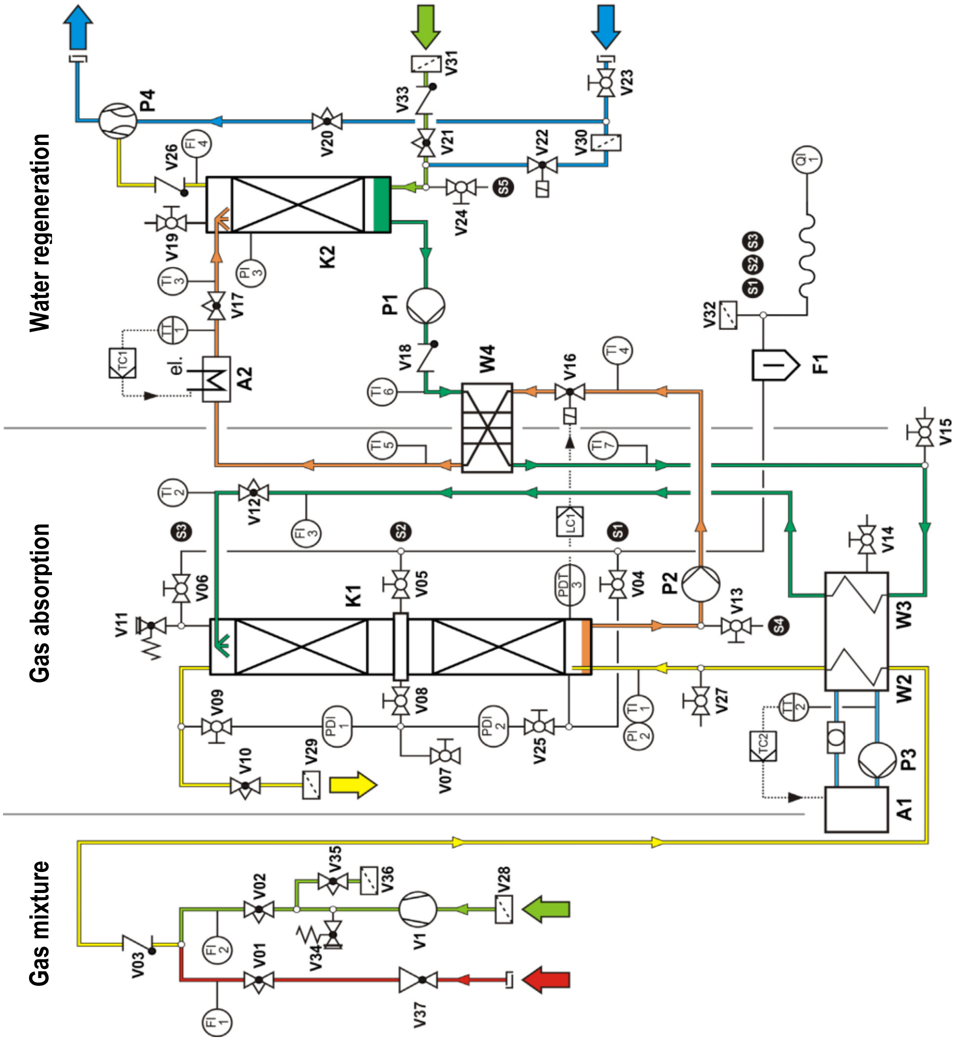


Figure 5. Flowchart of the CE 400 installation

4.5.1. Control elements/ distribution cabin

The CE400 installation has a switch cabinet from which all equipment requiring electrical power can be controlled. In Figure 6 it is shown the main control components, (1) the absorber feed pump P1, (2) the cooler and circulation pump P3, (3) the desorber supply pump P2, (4) the heater, (5) the compressor, (6) the fresh water supply, (7) the cooler temperature regulator, (8) the heater temperature regulator and (9) the principal switch.

Figure 6. CE400 switch cabinet

4.5.2. Gas mixture

In this stage prior to absorption, the CO_2 is mixed with air, the CO_2 is supplied from a cylinder and the air is sucked from the environment using the compressor (V1).

The flows of both gases are regulated by using valves and rotameters as it can be seen in the Figure 7. The CO_2 flow is regulated with valve V01 and by observing the rotameter FI2. The air flow is regulated with valve V02 while observing the rotameter FI1. Finally, the mixing is carried out when the two pipes connect with a non-return valve (V3) to prevent water from entering the absorption column.

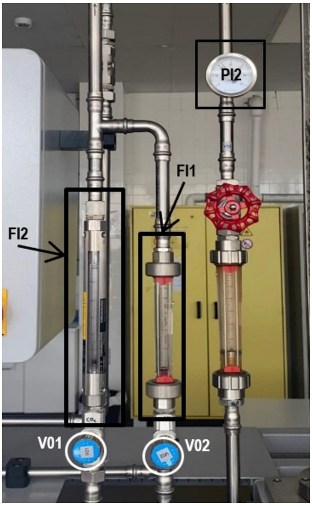


Figure 7. Gas mixture

The maximum outlet pressure of the compressor is 2 bar relative (rel.). The safety valve is responsible for limiting the max. pressure to approx. 1 bar rel. The rotameters are calibrated for volumetric flow rates at 20°C and 1 bar.

4.5.3. Absorption column (K1)

The absorption stage consists of a packed Duran glass column (K1), in which the water current is introduced at the top of the column using the pump P1 and running downwards, crossing the gas mixture and absorbing CO_2. The gas mixture enters at the bottom of the column through a bubbler nozzle and flows to the top where it exits through the pressure regulator valve (V10) and a silencer. This valve reduces the gas that exits the column, so that it is possible to influence both the gas flow rate through the column and the gas pressure. In addition, the column is equipped with a safety valve (V11) to prevent overpressure, the maximum working column pressure is 1 bar rel. and is indicated on the manometer PI2.

The column structure consists of two same length columns (750 mm) separated by a central perforated flange. Both columns contain a height of Raschig rings packing made of 1.4301 stainless steel with dimensions of 8 x 8 x 0.3 mm. The rest of the technical specifications can be found in the Appendix 3.

Thanks to the structure of the column, it is possible to determine pressure drops along the column using two U-manometers and to sample gas and water at different points of the column.

The absorption liquid can be supplied directly from the water distribution network as fresh water or recirculated into the unit by using the desorption column. For this purpose, once the water has passed through the column K1 and absorbed the CO_2, it is sent to the heat exchanger W4 with the help of the return pump P2, where it is heated and sent to the heater (if it is switched on) to improve the desorption. After being heated, it is introduced into the desorption column K2, where this CO_2 is desorbed and the water recirculated back to the absorption column.

In addition, to ensure that the gas mixture with the wash water does not escape from the bottom, a liquid level of approximately 8 cm is maintained at the base of the column. This accumulated water level is regulated by activating the level-dependent solenoid control valve (V16). The level is detected by measuring differential pressures of the liquid (PDT3).

4.5.4. Heat exchanger (W2/W3)

The CE400 equipment has a refrigeration unit prior to the absorption stage that allows working at different absorption temperatures in column K1. This unit contains a refrigeration installation (A1) that reduces the temperature of a mixture of water and glycol contained in the heat exchanger to reduce both the gas and the liquid inlet currents temperatures. The lower limit temperature of the cooling circuit is 4 °C. For its use is recommended to switch it on half an hour earlier to allow faster cooling.

4.5.5. Refrigeration unit (A1)

The refrigeration unit (A1) allows to reduce the temperature of the entering currents in K1. This system consists of a compressor, condenser, pressure switch, collector and expansion valve. The expansion valve injects the cooling agent directly into the plate heat exchanger for evaporation. With the help of the temperature sensor (TT2) prior to the heat exchanger W2/W3 and an electronic temperature controller, a temperature below 5 °C is prevented. A pressure switch monitors the pressures inside the refrigeration system.

4.5.6. Desorption column (K2)

After the absorption stage, the water current leaves the absorption column loaded with CO_2 and in order to recirculate it, the CO_2 content is reduced through desorption. For this purpose, it is transferred with the help of the pump P2 to the head of the regeneration column (K2) through the heat exchanger (W4) and the heating system (A2), allowing to pre-heat the water current for a better desorption. In addition, there is a control valve (V17) which can be used to influence the counterpressure downstream of the solenoid control valve (V16).

Once the liquid current reaches the head of the K2 column, it is released into the column with a disperser and collected at the base of the column. For better desorption, a depression is built in the column using a water jet pump (P4). Its flow rate can be regulated with the valve V20, which is used to reduce the flow rate of water through the water jet pump, thus regulating the depression (PI3) in the desorption column. In addition, valve V21 is available to supply external air for a better desorption.

The regenerated water is pumped with P1 to the head of the absorption column but before that it is fed to the heat exchangers W4 for heat recovery and W2/W3 to cold down. The water flow can be adjusted with the control valve V12 and measured on the rotameter FI3.

4.5.7. Heating (A2)

The heating unit (A2) is used to increase the temperature of the water current prior to desorption in order to promote the separation of the CO_2 gas from the water.

For safety reasons, the heater is equipped with a temperature limiter, which is activated in case of overheating and must be re-set manually. To do this, the lower screw nut is removed, and the push button is pressed.

4.5.8. Heat exchanger (W4)

The heat exchanger (W4) is a countercurrent plate heat exchanger used to re-utilise the thermal energy of the liquid outlet current of the desorber to heat the liquid inlet current to that same column. This way, the thermal volume that is supplied by the heating system (A2) is used to remain in the regeneration process and is not supplied to the absorption process.

4.5.9. Air compressor (V)

The air compressor (V) which is used to supply the air flow can deliver a maximum of 30 L/min and generate a maximum pressure of 2 bar, depending on the characteristic curve. It has a safety valve (V34) with a pressure limit of 1 bar. For very small flow rates (less than 5 L/min at 0.5 bar), a by-pass with the V35 valve is necessary to reduce the pressure.

The pressure in the absorption column can be adjusted by manipulating the valve V10 and with the help of the manometer PI2. It must be considered that the pressure in the K1 must be set sightly high enough, so the gas mixture reaches the sampling gas points for CO_2 concentration measurements.

4.5.10. Supply of CO_2

The gas is supplied from the CO_2 pipelines installation, which is connected to a gas bottle located in an outdoor cabinet. The CO_2 line is connected to the absorption equipment by using a pressure reducer. It is important the use of a suitable regulation unit to prevent the gas from reaching high pressures and to ensure that it remains constant, avoiding fluctuations in the concentration of the gas. For this purpose, a maximum CO_2 inlet pressure of 0.8 bar rel. is recommended.

4.5.11. Liqui level regulation

The liquid level regulation system in the base of the absorption column prevents the gas from the K1 being drawn in by the water as it can be seen in Figure 8.

The level control is carried out by measuring the pressure differential between the liquid pressure in the Pitot tube (a) and the pressure at the base of the column with a differential pressure meter (b). It is supplied to an electronic regulator located in the switch cabinet which activates the electromagnetic regulating valve (c), so that the level is maintained at a height of between 5 and 8 cm.

This system requires a regular checking to avoid air bubbles in the pressure measuring tubes.

Figure 8. Liquid level regulation

4.5.12. Regulators for the heating, the cooling and the liquid level at K1

In the control cabinet there are two temperature controllers, one for the heating and one for the cooling system, and a third controller for the column water level inside the cabinet.

For their use, the current value of the variable to be monitored is shown in red at the top of the display and the setpoint value is shown in green below it.

4.5.13. U-tube manometers

Two U-shaped tube manometers are used to measure the pressure drops in the absorption column. The first manometer (PD1) measures the pressure drop between the upper and middle point in the K1 and the second manometer (PD2) measures the pressure drop between the middle and lower point.

Its function consists of two vertical and parallel branches connected in a U-shape in which water is introduced until the two water branches are levelled. When different pressures are applied at both ends, the water level varies in each branch. With this difference in height between the two points, the pressure drop can be calculated.

4.5.14. Vacuum water jet pump

The vacuum jet pump is used to create a vacuum in the desorption column. Its operation requires only a strong water flow from the faculty net water supply without requiring any mechanical moving parts.

4.5.15. Needle manometer

The manometers used in the equipment are Bourdon types. Their main characteristics are their high accuracy, high precision and resistance to vibrations. Its operation is based on a tube bent into a C-shape. Their accuracy is indicated on the graduated scale, e.g. with cl. 1.6. Class 1.6 means that a maximum error of ±1.6 per cent of the full-scale value is permissible.

4.5.16. Rotameters

The volumetric flows of carbon dioxide, air and water are measured using rotameters.

They function using a vertical tube that expands vertically with the flow and a floating float that moves up and down. The medium flows from the bottom upwards through the tube. In this process the float rises until an annular gap is created between the wall of the tube and the float, so that the forces acting on the body are balanced. Depending on the scale of the rotameter, each float height corresponds to a certain flow rate. The flow rate can be read at the upper border of the conical collar of the float or at the centre of the ball.

4.6. ANALYTICAL METHODS

4.6.1. Sampling and analysis of CO_2 content on gas

Samples of the gas mixture from the K1 column can be taken at three different points during the running process, at the gas inlet to the column (S1), at half height of the column (S2) and at the gas outlet of the column (S3).

For the measurements it is required that the gas samples are dry, this is why a water separator is included it collects the water carried by the gas and prevents it from reaching the measuring instrument.

To proceed with the gas sampling the tap of the sample to be taken is opened (keeping the other two taps closed). Then the gas must expel the air thru the water separator waiting 15 to 20 seconds. If there is water accumulated in the separator it must be drained. The CO_2 measurements can be carried out at the sampling point using the CO_2 measuring instrument.

For the CO_2 concentration measurements, the AtomCheck device is used. This measuring instrument is designed to measure O_2 and CO_2 concentrations from gas samplings with a measuring range of 0 to 100 % volume for both gases.

The device contains an integrated pump that sucks the gas sample into the integrated measuring chamber and its measuring cells. For measurements it is necessary to add a nozzle and a short hose in the sampling point of the AtmoCheck device. It is also needed to ensure that no liquids or particles are sucked when the gas sampling is carried out.

To proceed with CO_2 measurement with the device, the power button is pressed, it requires a few seconds for the equipment to start while it self-checks the O_2/CO_2 sensors. The instrument will then automatically request the calibration of the O_2 sensors by pressing the "Go" button and waiting, the calibration is performed with ambient air (20.9 % O_2). Once the calibration is finished, the measurements can be taken placing the device nozzle on the gas sampling point and pressing the "Go" button. The instrument takes 10 seconds to perform the measurement. Once the measurements are finished to switch off the instrument, the down arrow must be press for about 5 seconds.

4.6.2. Sampling and analysis of CO_2 content on the absorbent

For the water sampling two point are provided on the CE400 equipment, the sampling point after the absorption at K1 using the valve V13 (S4) and the sampling point after the water regeneration at K2 using the valve V15 (S5).

To determine the CO_2 content on the absorbent a back titration is carried out. For this purpose, a list of instruments is used, a 20 mL graduated pipette, a 20 mL bulb pipette, three 200 mL beakers for each sample point, a 250 mL beaker, two drop counters and a 25 mL burette.

For the determination of CO_2, the liquid is sampled over a standard solution of 0.1 M sodium hydroxide (NaOH) slightly in excess. This will prevent the CO_2 content from varying by changing it to ionic carbonate (CO_3^{2-}). To do this, it is first necessary to calculate the volume of NaOH to be used.

For a sample volume of water, the maximum concentration of CO_2 is calculated, which in this case corresponds to the equilibrium concentration. With this value can be then determine the required volume of NaOH. For a 20 mL sample, corresponding to 1.11 moles of water it is obtained that there are $7.83 \cdot 10^3$ moles of CO_2 using the equation for the mole fraction of solute at equilibrium (14).

$$x_A^e = \frac{P}{H_{CO2}} = \frac{1}{1420} = \frac{mol\ CO2}{mol\ CO2 + mol\ H2O} \quad (14)$$

Using the conversion factor (15), it is obtained that 16 ml of NaOH are needed.

$$7.83 \cdot 10^{-3} CO_2 = \frac{2\ mol\ H^+}{1\ mol\ CO_2} \cdot \frac{1\ mol\ NaOH}{1\ mol\ H^+} \cdot \frac{1\ eq\ NaOH}{1\ mol\ NaOH} \cdot \frac{1\ L}{1\ eq\ NaOH} \cdot \frac{1000\ mL}{1\ L} = 16\ mL\ NaOH \quad (15)$$

The solution is then titrated with a drop of phenolphthalein (indicator) and a 0.1M hydrochloric acid (HCl) solution. The changeover occurs when all the excess hydroxide (OH^-) is consumed and practically all the CO_3^{2-} ions have been transformed into ionic hydrogen carbonate (HCO_3^-). The volume of HCl consumed is noted.

Continue titrating, adding two drops of methyl orange (indicator) until a change in colour is observed. This change indicates that HCO_3^- ions change to carbonic acid (H_2CO_3). The volume consumed is noted again. From the difference between these two volumes of HCl consumed, the CO_2 concentration is calculated using the (16) conversion factors.

$$c\ (mol\ CO_2/L) = \frac{\boldsymbol{x}\ mL\ HCl \cdot \frac{1\ L}{1000\ mL} \cdot \frac{0.1\ eq\ HCl}{1\ L} \cdot \frac{1\ mol\ HCl}{1\ eq\ HCl} \cdot \frac{1\ mol\ H_2CO_3}{1\ mol\ HCl}}{V_{sample}\ (L)} \quad (16)$$

Note that it is necessary to do a blank of the distilled water containing the sodium hydroxide solution NaOH, to determine the CO_2 in the form of carbonates that it may contain.

The colour jumps to be observed in the titration are for the first titration with phenolphthalein when the solution turns from magenta to colourless. And the second titration with methyl orange occurs when the solution changes from a yellowish to an orange colour.

5. INSTALLATION OPERATION

For the use of the CE400, it is necessary to carry out some previous checking on the different equipment's and elements of the installation. Then the installation can be started up and the experiments can be carried out. Once finished, the installation needs to be disconnected.

5.1. PRE-CHECKING OF THE INSTALLATION

Supply of CO_2

Prepare the CO_2 supply from the outside cabinet where the steel cylinders are located. Make sure that the pressure manometer indicates a certain pressure when the cylinder valve is opened.

Filling the cooling circuit

To fill the cooling circuit first close valve V14, then proceed to fill container W2/W3 with a water/glycol mixture at a ratio of 3:1 up to the sight glass. There is a hazard of destroying the heat exchanger or the lines by freezing at temperatures below 4°C if the cooling circuit is not filled with a water/glycol mixture.

Checking of the operation of the P3 pump in the cooling circuit

For new fillings of the cooling system or for longer downtimes the P3 pump operation needs to be check. In order to do that, firstly the cooler needs to be connected as well as the cooling pump from the switch cabinet. Then the presence of water flow to the heat exchanger W2/W3 needs to be checked with the help of the flow indicator. Finally, the pump needs to be bled by loosening the knurled screw on the pump casing and letting the air escape.

Connections for freshwater service

To supply the system with fresh water, fresh water must be provided using a tube from the network to the V15 valve. To prepare the distilled water/tap water connection, the valve V23 must first be closed. Next, the water supply lines must be connected to the water pipes of the faculty water network, as well as the vacuum water jet pump. Finlay the valve V20 needs to get closed.

Filling the U-manometers

To fill the tubes of the U-manometers with water, first the ball valves V9, V8 and V25 need to be closed. Then, the 3 top ball valves of the tubes need to be opened, and the air filters screwed off. With the help of a wash bottle the tubes are filled little by little with water until the zero line is reached. It is necessary to make sure that no air bubbles are trapped inside the tubes, if so, the air must be carefully pushed with the help of another empty wash bottle. Finally, the 3 ball valves need to be closed and the air filters screwed back on.

5.2. OPERATING PROCEDURE

For the start-up and commissioning of the installation the equipment is turned on with the main switch from the distribution control.

If the experimental procedure requires the use of the cooler, then it must be connected 30 minutes before continuing with the start-up. For it the P3 is connected from the distribution control, then the water flow is checked with the help of the P3 flow indicator. If no flow is detected the air needs to be purged out of the pump by loosening the knurled screw on the pump casing. To regulate the cooler temperature the setpoint value is introduced in the TC2.

To proceed with the filling of the absorption medium (water) into the columns, the V13, V24, V15, V8, V25, V6, V5, V4, V21 and V20 must be closed and the V10, V19, V23, V17 and V12 must be opened. Then it is needed to ensure that deionised water is supplied, closing the valve of the tap water line and opening the valve of the distilled water line. To allow water to run into the desorption column up to half full the black ‘Water supply’ switch on the control cabinet is used. In case of excess water, it can be drain through V24. It is important to check if the water once supplied is dirty. If it is, then the water must be drain through V24 and refilled until is clear. This prevents the plate heat exchanger W4 from clogging.

To fill the absorption column with water to a height of 10 to 20 cm the P1 must be used while leaving a heigh of 1/3 of water in the desorption column, if is necessary water can be added after. This step needs to be done carefully to avoid getting the K1 flooded causing the water to start flowing into the compressor.

To let the water spray into the absorption column the P1 is switched on and once the water level in K1 starts to rise the P2 is also switched on. Then the water flow rate sprayed into column K1 can be adjusted using V12. If the water level in column K1 does not stop rising, the V17 is gradually opened until the water level is constant.

To adjust the level in the absorption and desorption columns K1 and K2. The K2 column is refilled with distilled water until the desired level is maintained without exceeding 1/3 of its capacity.

To supply air, the V10 and V02 are opened, and the compressor is switched on. The air flow rate desired is set using valve V02 and paying attention to the rotameter FI2 and manometer PI2. In case the air flow is too big V35 is closed.

The vacuum in the K2 regeneration column is adjusted closing V19 and making sure that tap water is supplied. Then the control valve V20 is opened until a depression of approximately - 0.2 bar rel. vacuum is reached, to check the pressure the manometer PI3 is used. If the pressure dropped below level desired, then the control valve V21 is slightly opened letting some air in. Once the experiments are being carried out, the manometer PI3 needs to be monitored in case the pressure rises.

For the experiments carried out with the heating system in operation, the switch A2 can be turned on and the setpoint temperature can be adjusted using the arrows on the temperature controller (TC2).

Finally, the CO_2 is supplied adjusting approximately a 0.4 bar rel. pressure with the pressure reducer and the volumetric flow regulating valve V1. In case the air flow at the rotameter does not stabilise, the pressure at the CO_2 pressure is reduced to 0.3 bar and the CO_2 volumetric flows readjusted.

Differential pressure measurement

To carry out the measurements with the U-manometer the 3 ball valves above the manometer are closed and the V8, V9 and V25 are opened in the order are mentioned. Then the measurements are carried out using the rule readings. Finally, the ball valves V8, V9 and V25 are closed and the 3 ball valves are opened/ closed to reset the manometer water levels to point 0.

5.3. DISCONNECTION OF THE EQUIPMENT

In order to start disconnecting the equipment the supplying of the CO_2 is stopped closing the valve cylinder. Then the heating, the cooling system and the compressor are switched off.

The water supply to the vacuum water jet pump can be stopped adding a pressure compensation opening the V19. The pumps P1 and P2 can be switched off following the order in which are mentioned. Then the remaining CO_2 in the absorption column can be purged making air circulate though the absorption column. To do so the compressor is switch on and the valves V1 and V10 are opened to their maximum for 3 minutes.

Finally, the equipment can be turned off using the main switch.

6. EXPERIMENTAL PROPOSALS

In this section, two different experimental proposals are presented for the students of chemical engineering to be put into practice in the EEQ4 course. These two experiments of CO_2 absorption in water are proposed to study the effects that different variables have on the absorption process. For the tests, the CE400 installation is used and the sampling and analysis methods for CO_2 concentrations previously seen are putted in practice.

Prior to both experiments, a blank sample containing only 20 mL of distilled water and 16 mL of sodium hydroxide is titrated, as it is needed for the measurement of CO_2 concentration in water.

6.1. ABSORPTION OF PURE CO_2 IN WATER

In the first experimental proposal the installation is set-up to carry a CO_2 absorption in water, supplying only pure CO_2 as the gas current and with water regeneration. The experiment is carried out to study the effect of two variables, the water flow rate and the absorption temperature, on the global coefficients of mass transfer.

For this purpose, an inlet current of CO_2 is kept constant to the absorption column while pressure and CO_2 in water concentrations are measured for different water volumetric flow rates. A doble experiment is carried out with two different cooling temperatures in column K1.

Experimental procedure

To carry out the experiment, the installation is first started up and the pre-checking's are carried out while the refrigeration unit is switch on for half an hour before proceeding. The first experiment is carried with a 12ºC cooling temperature setpoint an the second one at 20ºC. Then the heating is set-up to a 40ºC and the pressure in column K1 is set to 0.1 bar rel. with valve V10.

While the equipment temperatures stabilize the material necessary for the titrations are prepare. It is necessary to add the 0.1 M sodium hydroxide solution as quickly as possible to the water samples of S4 and S5 to prevent the CO_2 from desorbing into the air. For this purpose, it is recommended to have the amounts of NaOH prepared in advance.

As for the gas/water currents, the desired water volumetric flow rate is set first, and then volumetric CO_2 flow is set to 2 L/min with an inlet pressure of 0.4 bar rel.

Before carrying out the experimental measurements the temperature and pressures measured by the thermometers and manometers of the CE400 installation are noted.

Three measurements of the pressure dops (PD1 and PD2) in the absorption column are taken, as well as for the absorption medium samples S4 and S5 to determine their CO_2 content.

6.2. ABSORPTION OF CO_2 IN AIR IN WATER

In the second experimental proposal the installation is set-up to carry a CO_2 absorption in water, supplying a CO_2/air mix in the gas current and with water regeneration. The experiment is carried out to study the effect of two variables, the water flow rate and the CO_2 concentration at the inlet current, on the global coefficients of mass transfer.

For this purpose, the inlet flux of CO_2/air mix is kept constant to the absorption column while pressure and CO_2 in water and gas concentrations are measured for different water volumetric flow rates. A doble experiment is carried out with different concentration of CO_2 at the inlet gas current of the absorption column.

Experimental procedure

To carry out the experiment, the installation is first started up and the pre-checking's are carried out while the refrigeration unit is switch on for half an hour before proceeding. The experiments are carried with a 13ºC cooling temperature setpoint. The heating is not use for this experiment and the pressure in column K1 is set to 0.1 bar rel. with valve V10.

While the equipment temperatures stabilize the material necessary for the titrations are prepare. In this experiment only the S5 water samplings are taken.

As for the gas/water currents, the desired water volumetric flow rate is set first. Then the air is set to a flow rate of 5 Nm^3/h. And lastly, the CO_2 flow is set to 1 L/min for the first concentration experiment and 0.5 L/min for the second one, with an inlet pressure of 0.4 bar rel.

Before carrying out the experimental measurements the temperature and pressures measured by the thermometers and manometers of the CE400 installation are noted.

While waiting 5 minutes for the system to stabilise the three measurements of the pressure dops (PD1 and PD2) in the absorption column can be taken.

Passed the 5 minutes, the three medium samples of S5 can be taken and their CO_2 content can be determined.

Finally, the CO_2 concentration measurements in the gas inlet and outlet current of the absorption column (S3 and S1) are taken in this exact order. For each sampling point 10 measurements are taken. These measurements need to be taken every minute, and each time the K1 has to be pressured once the sampling valve is closed.

This experimental process is then repeated for a total of 3 water flow rates, previously emptying the system with the water already CO_2 saturated.

7. RESULTS AND DISCUSSIONS

In this section, the values of the measurements carried out in both tests with the CE400 absorption equipment will be presented and analysed. It must be considered that the reproducibility of these tests can be affected by the performance of the different components, the skill in the field of the experimental technique and the environmental conditions.

In Table 6 the result for the blank test made is presented.

Table 6. Titrations performed for the blank sample.

Sample	**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)
1	14.2	15.5	1.3
2	14.9	16.0	1.1
		$\overline{\Delta V}$ =	1.2

7.1. ABSORPTION OF PURE CO_2 IN WATER

In Table 7 and 8, are collected some examples of the readings obtained from the air and CO_2 rotameters, the measurements of pressure differences and the volume titrations of the water samples for the water inlet and outlet of K1 for both tests. All the rest of measurements for these experiments are collected in the Appendix 5.

Table 7. Mesurements examples obteined from the experiment 1 with a cooling temperature of 11.5ºC.

Flow rates		**Pressure drops**		**Water samples S4**			**Water samples S5**		
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)	**V^*_1 HCl** (mL)	**V^*_2 HCl** (mL)	**ΔV^*** (mL)
2	100	1	110	10.7	18.2	7.5	14.7	18.4	3.7
2	200	1	110	10.1	18.1	8.0	14.6	18.5	3.9
2	280	1	100	9.8	18.1	8.3	13.3	18.1	4.8

Table 8. Mesurements examples obteined from the experiment 1 with a cooling temperature of 20ºC.

Flow rates		**Pressure drops**		**Water samples S4**			**Water samples S5**		
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)	**V^*_1 HCl** (mL)	**V^*_2 HCl** (mL)	**ΔV*** (mL)
2	100	1	150	12.0	18.1	6.1	14.9	18.3	3.4
2	200	1	140	13.0	18.6	5.6	14.0	18.5	4.5
2	280	4	100	10.8	18.1	7.3	14.4	18.0	3.6

With the collected measurements, the average value of the pressure drops and the difference in HCl volumes of the titrations are calculated in the Tables 9 and 10. As can be seen in the first manometer (PD1), practically no pressure drop is observed in any of the cases, since the gas flow rate circulating is very low in both experiments.

Table 9. Average experimental data of the pure CO_2 and water system at 11.5ºC cooling temperature.

Volumetric flow rates		**Pressure drops**		**Water samples**	
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**ΔV HCl S4** (mL)	**ΔV HCl S5** (mL)
2	100	1	113	7.5	3.7
2	200	1	108	8.0	3.9
2	280	1	103	8.3	4.8

Table 10. Average experimental data of the pure CO_2 and water system at 20ºC cooling temperature.

Volumetric flow rates		**Pressure drops**		**Water samples**	
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**ΔV HCl S4** (mL)	**ΔV HCl S5** (mL)
2	100	1	156	6.13	3.33
2	200	1	132	7.07	4.50
2	280	4	110	7.17	3.73

The readings of the stabilised temperatures and pressures of the equipment for the experiment 1 for both cooling temperatures can be found in the Appendix 4.

7.1.1. Comparison of the global coefficients of mass transfer for different cooling temperatures

To proceed with the calculation of the global coefficients of mass transfer, the molar water and CO_2 flow rates and the mole fractions of CO_2 in water currents must first be calculated.

The conversion from volumetric flow rates to molar flow rates of water is calculated using the expression (17).

$$L' = L \cdot \frac{\rho_{H2O}}{M_{H20}} \quad (17)$$

where:

L’ = molar water flow rate (mol/s)

L = volumetric water flow rate from the flowmeters readings (m^3/s)

ρ_{H20} = water density at temperature TI1 (kg/m^3)

M_{H20} = water molecular mass (kg/mol)

To convert the volumetric CO_2 flow rates to molar flow rates it is requires knowing the pressure and temperature conditions under which the gas flows through the column. The pressure can be calculated using the average pressure drops of the absorption column and for the temperature the one indicated by the TI1 (water inlet current temperature) thermometer is used. To do so the expression (18) is used.

$$G'_{CO2}\left(\frac{mol}{s}\right) = G_{CO2}\left(\frac{L}{min}\right) \cdot \frac{P_{average} \cdot 1min}{R \cdot TI1 \cdot 60s} \quad (18)$$

where:

G’ = molar CO_2 flow rate (mol/s)

G = volumetric CO_2 flow rate from the flowmeters readings (L/min)

R = Ideal gas constant (J/mol · K)

$P_{average}$ = Average pressure in K1 (Pa). Can be calculated using the expression (19).

$$P_{average} = PI1 \cdot \frac{(PI1+PI2)}{2} \quad (19)$$

To calculate the CO_2 mole fractions at the inlet and outlet of the column, the quantity of moles of CO_2 for each sample needs to be calculated first. To do so, the difference between the two HCl volumes consumed (between the first and the second colour change) are used, subtracting the volume of HCl consumed in the blank. To simplify the calculations, the quantity of CO_2 moles is considered much smaller than the water. The expressions (20), (21) and (22) are used to obtain the mole fractions.

$$\mathrm{n_{CO2}} = (\Delta \mathrm{V_{sample}} - \Delta \mathrm{V_{blank\ test}}) \cdot \frac{1\ \mathrm{L}}{1000\ \mathrm{mL}} \cdot \frac{0.1\ \mathrm{eq\ HCl}}{1\ \mathrm{L}} \cdot \frac{1\ \mathrm{mol\ HCl}}{1\ \mathrm{eq\ HCl}} \cdot \frac{1\ \mathrm{mol\ H_2CO_3}}{1\ \mathrm{mol\ HCl}} \quad (20)$$

$$\mathrm{n_{total}} = \frac{0.02\ \mathrm{L\ water\ sample} \cdot \rho_{\mathrm{H2O}}}{\mathrm{M_{H2O}}} \quad (21)$$

$$\mathrm{x}_{CO2} = \frac{\mathrm{n_{CO2}}}{\mathrm{n_{total}}} \quad (22)$$

where:

x_{CO2} = CO_2 molar fraction in the water

n_{CO2}= CO_2 mole quantity in the water sample (mol)

n_{total} = moles of water in the water sample (mol)

Bearing in mind that the phase controlling the transfer of mass, i.e. the one that presents the greatest resistance, is the liquid phase since the gas compositions is pure CO_2, it is then proceeded to calculate the values of the global coefficients of mass transfer for the liquid phase ($K_x \cdot a$). For the pure CO_2 system, the mole fraction in the gas phase is constant throughout the experiment (y=1), therefore, the equilibrium concentration value is constant x^e. Taking this into account, the expression (28) can be used.

To obtain the value of the mole fraction at equilibrium CO_2, x^e is isolated from Henry's law (6). Where $H_{i,px}$ can be calculated using expressions (4) an (5) as a function of column temperature or taken from the equilibrium diagram in Figure 4. The partial pressure of CO_2 in this case is the same as the total pressure.

The equilibrium values for both experiments at different temperatures are given in the Table 11.

Table 11. Pure CO_2/water equilibrium values according to column temperature.

	Equilibrium values T cooling= 11.5ºC	Equilibrium values T cooling= 20ºC
$H_{i,px}$=	1505	1634
x^e =	7.21E-04	6.64E-04

A way of looking at the relationship between the liquid flow rate and the mass transfer coefficient is to study the relationship of it to the Reynolds number (Re) whose expression is (23).

$$Re = \frac{L/_S}{a_S \cdot \mu(T)} \quad (23)$$

where:

L = volumetric water flow rate (m^3/s)

S = cross-section of column K1 (m^2)

μ = water viscosity at the temperature TI1 (Pa·s)

a_s = specific area of the filling (m^{-1})

The Tables 12 and 13 summarise the calculations made according to the above points for the cooling temperature experiments at 11.5°C and 20°C respectively.

Table 12. Determination of the parameters of the pure CO_2 /water system at 11.5ºC cooling temperature

G_{CO2} (mol/s)	**L** (mol/s)	**$x_{1,CO2}$**	**$x_{2,CO2}$**	**$K_x \cdot a$** (mol/m³·s)	**Re**	**log (K_x·a)**	**log (Re)**
1.49E-03	1.54	5.68E-04	2.25E-04	897	7.97	2.95	0.90
1.49E-03	3.08	6.16E-04	2.40E-04	2321	15.94	3.37	1.20
1.49E-03	4.24	6.40E-04	3.27E-04	3314	21.91	3.52	1.34

Table 13. Determination of the parameters of the pure CO_2 / water system at 20ºC cooling temperature

G_{CO2} (mol/s)	**L** (mol/s)	**$x_{1,CO2}$**	**$x_{2,CO2}$**	**$K_x \cdot a$** (mol/m³·s)	**Re**	**log (K_x·a)**	**log (Re)**
1.47E-03	1.54	4.44E-04	1.92E-04	585	8.16	2.77	0.91
1.47E-03	3.08	5.29E-04	2.97E-04	1523	16.33	3.18	1.21
1.47E-03	4.31	5.38E-04	2.28E-04	2649	22.86	3.42	1.36

After obtaining the values of the global coefficients of mass transfer and Reynolds numbers, the logarithms of both are represented in Figure 9 for two experiments carried out at different cooling temperatures.

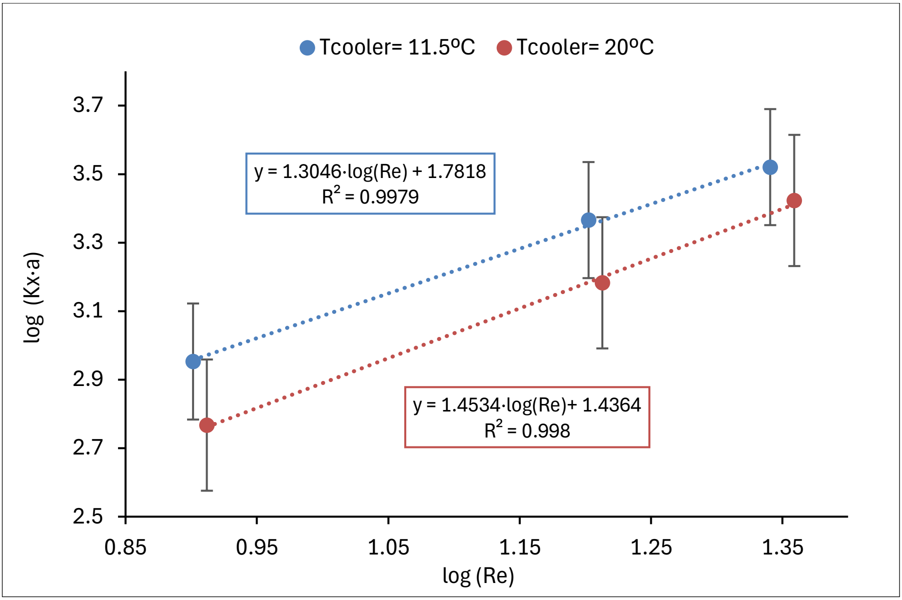


Figure 9. Variation of log (K_x·a) vs log (Re) for the CO_2 pure absorption in water in experiment 1

From the graphic and the values obtained, it is observed that as the Reynolds increases so does the global coefficient of mass transfer. This is mainly due to the fact that as the flow of water through the column increases, more intense turbulence appears. Remembering the Fick's law, the transfer of mass depends on the difference in concentrations between the sinus of the phases and the interface, and the distance between them. This distance is affected by the turbulence in the phases, in this case, as the turbulence in the water increases, the distance is reduced, causing a higher solute transfer.

On the other hand, comparing the values obtained at different cooling, i.e. the temperature in the absorption column (K1), it can be affirmed that the lower the absorption temperature, the higher is the global coefficient of mass transfer. This happens because the solubility of CO_2 in water decreases as the temperature increases. The increase in temperature causes the gas and water molecules to gain kinetic energy, with faster and more vigorous movements, making it more difficult to keep the CO_2 molecules in solution.

Looking at Henry's law, it relates solubility to the partial pressure of gas in the liquid, by means of Henry's coefficient. This coefficient depends on the temperature and the gas, and as already seen, this coefficient increases with increasing temperature, which means that the concentration at which equilibrium (x^e) is reached in water is lower.

Remembering the expression of the global coefficient of mass transfer it contained the Henry's coefficient, therefore its dependence on the temperature.

7.2. ABSORPTION OF CO_2 IN AIR IN WATER

In Table 14 and 15, are collected some examples of the readings obtained from the air and CO_2 rotameters, measurements of pressure differences, the measurements of CO_2 concentration in gas at the inlet of the K1 and the outlet, and the titration of the water sample at the inlet of K1. All the rest of the measurements for these experiments are collected in the Appendix 6.

Table 14. Measurements examples of the experiment with a gas mixture 1 L/min CO_2 + 0.3 Nm^3/h air.

Flow rates			**Pressure drops**		**Gas samples**				**Water sample S5**		
CO_2 (L/min)	**Air** (Nm^3/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**%vol. CO_2 S1**		**%vol. CO_2 S3**		**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)
1	0.3	100	90	55	23.4	23.5	23.3	23.3	15.9	18.7	2.8
1	0.3	200	100	60	23.2	23.2	23.5	23.7	15.8	18.7	2.9
1	0.3	225	105	60	23.0	22.8	23.6	23.7	16.0	18.7	2.7
1	0.3	275	115	62	22.5	22.3	23.9	23.7	16.1	18.7	2.6

Table 15. Measurements examples of the experiment with a gas mixture 0.5 L/min CO_2 + 0.3 Nm^3/h air.

Flow rates			**Pressure drops**		**Gas samples**				**Water sample S5**		
CO_2 (L/min)	**Air** (Nm^3/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**%vol. CO_2 S1**		**%vol. CO_2 S3**		**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)
0.5	0.3	100	105	100	13.8	13.8	13.5	13.6	16.7	18.5	1.8
0.5	0.3	200	85	110	13.9	14.0	13.5	13.6	16.4	18.6	2.2
0.5	0.3	225	70	58	13.9	13.9	13.5	13.5	16.4	18.5	2.1
0.5	0.3	275	85	60	14.0	14.0	13.3	13.4	16.4	18.6	2.2

With the collected measurements, the averages values of the pressures drop, the average values of the CO_2 concentrations in gas, and the difference in HCl volumes of the titrations are calculated in the Tables 16 and 17 for each experiment.

Table 16. Average measurements of the experiment with a gas mixture of 0.5L/h CO_2 + 0.5Nm³/h air

Volumetric flow rates			**Pressure drops**		**Water sample**	**Gas samples**	
CO_2 (L/min)	**Air** (Nm³/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**ΔV HCl S5** (mL)	%**vol. CO_2 S1**	%**vol. CO_2 S3**
0.5	0.3	100	105	105	0.60	13.81	13.59
0.5	0.3	200	88	103	0.93	13.98	13.56
0.5	0.3	225	75	61	0.90	13.98	13.44
0.5	0.3	280	82	55	0.97	13.99	13.39

Table 17. Average measurements of the experiment with a gas mixture of 1L/h CO_2 + 0.5Nm³/h air

Volumetric flow rates			**Pressure drops**		**Water sample**	**Gas samples**	
CO_2 (L/min)	**Air** (Nm³/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**ΔV HCl S5** (mL)	%**vol. CO_2 S1**	%**vol. CO_2 S3**
1	0.3	100	88	55	1.6	23.51	23.26
1	0.3	200	98	59	1.7	23.66	23.12
1	0.3	225	103	58	1.5	23.66	22.82
1	0.3	275	113	62	1.4	23.78	22.45

With the values obtained from the measurements of CO_2 concentration in the gas inlet and outlet currents of K1 and the respective average values at each point, a graphical representation is made versus the sampling time in the Figures 10 and 11 for each experiment.

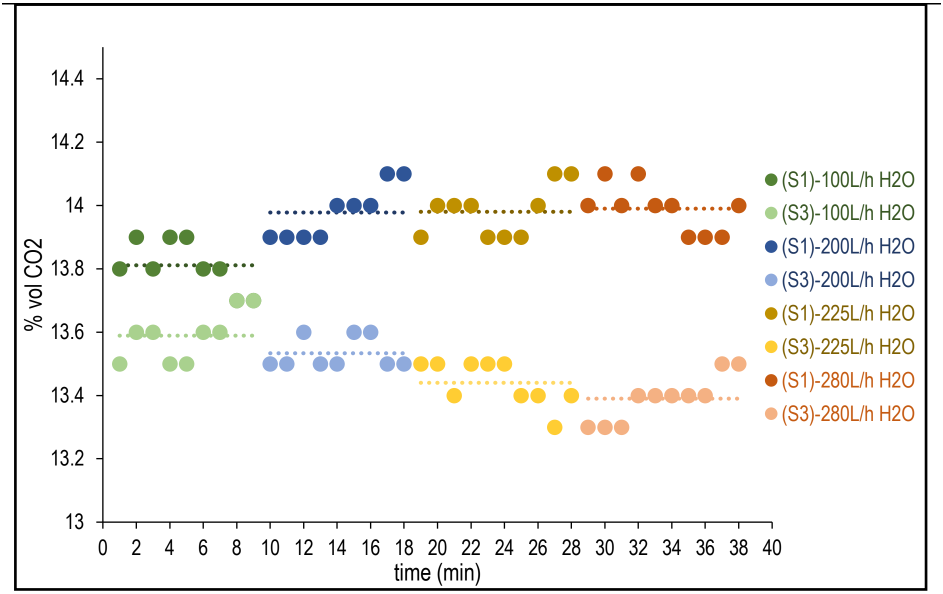


Figure 10. CO_2 concentrations in volumetric percentages at points S1 and S3 for different flow rates of water for the experiment carried with a gas mixture of 0.5 L/h CO_2 + 0.5 Nm3/h air.

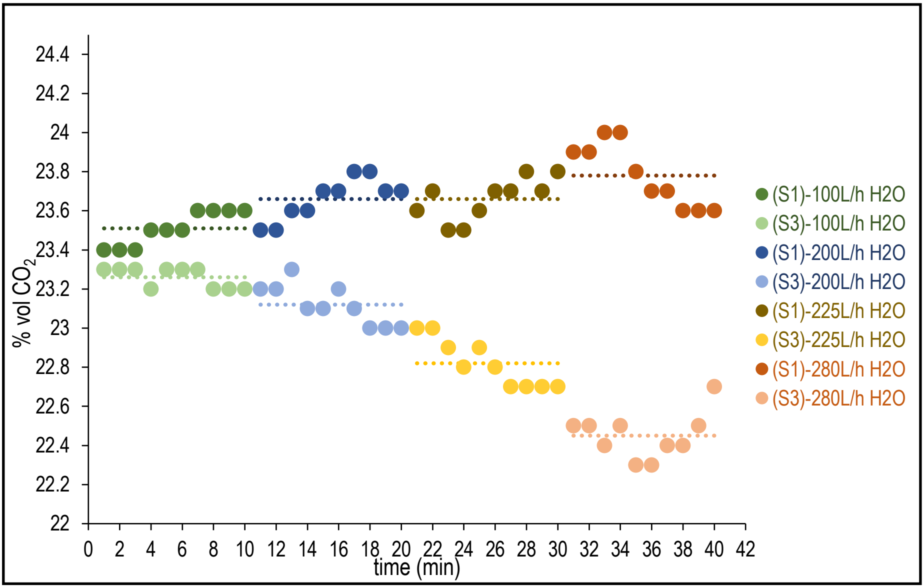


Figure 11. CO_2 concentrations in volumetric percentages at points S1 and S3 for different flow rates of water for the experiment carried with a gas mixture of 1 L/h CO_2 + 0.5 Nm3/h air.

Comparing both graphs for both experiments at different concentrations of CO_2 in gas, it may be observed that as the water flow rate increases the CO_2 concentrations at the outlet of the column decrease, as discussed in the previous experiment, this happens because the turbulence increases which causes a higher transfer of mass between the phases.

The readings of the stabilized temperatures and pressures of the equipment for the experiments carried out for a gas mixture of 0.5 L/h CO_2 + 0.5 Nm^3/h air and 1 L/h CO_2 + 0.5 Nm^3/h air are collected in the Appendix 4.

7.2.1. Comparison of the global coefficients of mass transfer for different gas composition mixtures

To proceed with the calculation of the global coefficients of mass transfer, the molar flow rates of water, air and CO_2 must be calculated as well as the mole fractions of CO_2 in both phases currents of the K1 column.

For the conversion of volumetric flow rates to molar flow rates of the water the expression (17) is used the same way as in the experiment 1.

For the conversion of volumetric flow rates to molar flow rates of the air, the volumetric flow of air is previously calculated under standard conditions (0ºC and 1.013 bar), using the equation of state for ideal gases. For it, the expression (24) is used and then divided by the molar volume as was done for the previous experiment in expression (18) for the CO_2 gas.

$$G_{air,St.cond.} = G_{air,Exp.} \cdot \left(\frac{P_{exp} \cdot T_{St.cond}}{P_{St.cond} \cdot T_{exp}} \right) \quad (24)$$

where:

$G_{air,St.cond.}$ = volumetric air flow rate at standard conditions (m^3/h)
$G_{air,Exp.}$ = volumetric air flow rate at experimental conditions (Nm^3/h)
P_{exp} = experimental column K1 pressure measured with PI2 (Pa)
T_{exp} = experimental air inlet current temperature measured with TI1 (K)
$P_{St.cond}$ = pressure in standard conditions (Pa)
$T_{St.cond}$ = temperature in standard conditions (K)

For the calculation of the CO_2 molar flow rate the expression (25) derived from the mole fraction of CO_2 in the inlet gas current is used.

$$G'_{CO2,1} = \frac{y_1}{1-y_1} G'_{air,1} \quad (25)$$

where:
$G'_{CO2,1}$ = molar CO_2 flow rate at the inlet current of K1 (mol/s)
$G'_{air,1}$ = molar air flow rate at the inlet current of K1 (mol/s)
y_1 = mole fraction in the gas inlet current of CO_2

For the calculation of the mole fraction of CO_2 in the water at K1 inlet current ($x_{1,\,CO2}$) the same expressions are used as in the previous experiment. And for the calculation of the mole fraction of CO_2 in the water at K1 current outlet ($x_{2,\,CO2}$) the expression of the operating line obtained from the global balance (28), is used for this purpose.

For the process of absorption of CO_2 in water, as the phase that presents the greatest resistance in the transfer of mass is the liquid phase, the global mass transfer coefficient for the liquid phase ($K_x \cdot a$) is used as well. When the solute to be absorbed is found in a gas mixture, its mole fraction in the gas will decrease along the column as it is absorbed. Consequently, the equilibrium points will also decrease, so that the mole fraction in the CO_2 equilibrium (x^e) will not be constant.

Therefore, the resolution of the integral that contains the expression of ($K_x \cdot a$) must be solved using numerical methods, in this case the trapezoidal rule. This requires the calculation of the x^e values at the inlet and outlet of the column using Henry's law, the calculations are the same as in the previous experiment, the only difference being that the partial pressure of CO_2 depends on the mole fraction in the gas phase at the respective point.

In Tables 18 and 19 the values obtained in the calculations described above and the equilibrium values for both mixtures used in this experiment are given.

Table 18. Determination of different parameters of the experiment carried with a gas mixture of 0.5 L/h CO_2 + 0.5 Nm^3/h air

G_{CO2} (mol/s)	**G_{air}** (mol/s)	**G** (mol/s)	**L** (mol/s)	**$y_{1, CO2}$**	**$y_{2, CO2}$**	**$x_{1, CO2}$**	**$x_{2, CO2}$**	**x^e_1**	**x^e_2**
6.65E-04	4.15E-03	4.82E-03	1.54	0.1381	0.1359	5.41E-05	6.10E-05	1.30E-04	1.28E-04
6.75E-04	4.15E-03	4.83E-03	3.08	0.1398	0.1356	8.41E-05	9.07E-05	1.32E-04	1.28E-04
6.77E-04	4.16E-03	4.84E-03	3.47	0.1398	0.1344	8.11E-05	8.86E-05	1.32E-04	1.27E-04
6.77E-04	4.16E-03	4.84E-03	4.24	0.1399	0.1339	8.71E-05	9.39E-05	1.32E-04	1.26E-04

Table 19. Determination of different parameters of the experiment carried with a gas mixture of 1 L/h CO_2 + 0.5 Nm^3/h air

G_{CO2} (mol/s)	**G_{air}** (mol/s)	**G** (mol/s)	**L** (mol/s)	**$y_{1, CO2}$**	**$y_{2, CO2}$**	**$x_{1, CO2}$**	**$x_{2, CO2}$**	**x^e_1**	**x^e_2**
1.27E-03	4.13E-03	5.40E-03	1.54	0.2351	0.2326	1.41E-04	1.50E-04	2.15E-04	2.12E-04
1.28E-03	4.13E-03	5.40E-03	3.08	0.2366	0.2312	1.53E-04	1.63E-04	2.16E-04	2.11E-04
1.29E-03	4.12E-03	5.41E-03	3.47	0.2366	0.2282	1.38E-04	1.51E-04	2.16E-04	2.08E-04
1.29E-03	4.12E-03	5.41E-03	4.24	0.238	0.225	1.26E-04	1.43E-04	2.17E-04	2.05E-04

The Tables 20 and 21 show the calculations of the global coefficients of mass transfer and their respective Reynolds numbers. In the Figure 12 the logarithms of these values are represented and an approximation of the trend of the experimental points are obtained by means of linear regression for both gas mixtures.

Table 20. Calculation of the global mass transfer coefficient and the Reynolds of the experiment carried with a gas mixture of 0.5 L/h CO_2 + 0.5 Nm^3/h air

$K_x \cdot a$ ($mol/m^3 \cdot s$)	**Re**	**log ($K_x \cdot a$)**	**log (Re)**
74	454	1.87	2.66
237	907	2.38	2.96
292	1021	2.47	3.01
373	1247	2.57	3.10

Table 21. Calculation of the global mass transfer coefficient and the Reynolds of the experiment carried with a gas mixture of 1 L/h CO_2 + 0.5 Nm^3/h air.

$K_x \cdot a$ ($mol/m^3 \cdot s$)	**Re**	**log ($K_x \cdot a$)**	**log (Re)**
99	454	1.99	2.66
261	907	2.42	2.96
335	1021	2.52	3.01
468	1247	2.67	3.10

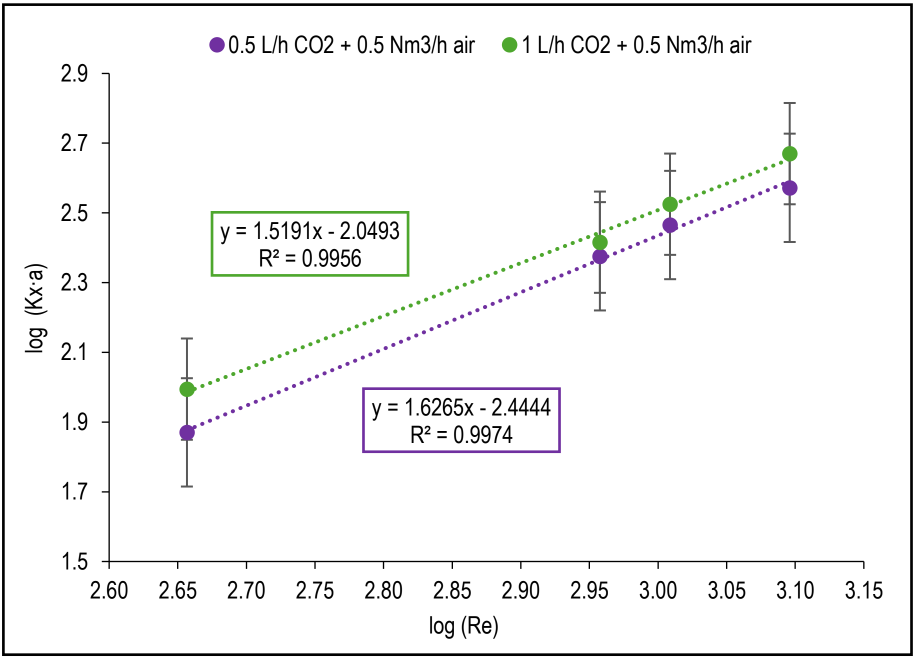


Figure 12. Variation of log ($K_x \cdot a$) versus log (Re) for the absorptions for to different CO_2/air mixtures.

Observing the graphical representation and the values obtained, the first conclusion that can be drawn is that the same tendency respecting the experiment carried out with pure CO_2 can be seen for the global coefficient of mass transfer. It increases as the Reynolds number does, the same explanation from the past experiment is valid.

On the other hand, if the experiments carried out where only the CO_2 concentrations at the gas inlet are varied are compared. Without using any correlation, when the air and water flows are kept constant and only the CO_2 flow is varied, it can be observed that the global mass transfer coefficient slightly increases. Even if the global coefficient of mass transfer does not depend on the concentrations of both phases, it is also true that as the CO_2 flow rate increases, as everything is related to the flow, the turbulence in the gas phase also increases, which moves the system away from equilibrium, facilitating the transfer of mass at the interface.

8. CONCLUSIONS

When designing absorption columns, one of the most important factors to be taken into account is the global coefficients of mass transfer. As has been seen, the value of this coefficient is affected by different operating parameters and by the technical limits of the equipment itself.

In this work where two different absorption experiments are carried out, it has been studied the effect of three parameters, water flow rate, solute concentration at the gas supply inlet and the absorption temperature, on the global coefficients of mass transfer.

The first conclusions obtained is, in both experiments, when the water flow rate is increased, the mass transfer coefficient increases. In other words, by increasing the Reynolds, the turbulence in the liquid increases, reducing the distance of the interface and distributing the CO_2 better within the liquid, making the gradient created at the interface greater, both of which translate into a higher mass transfer velocity.

As for the first experiment, when the absorption temperature decreases, the mass transfer coefficient increases. This happens because the solubility of CO_2 in water increases, and so does the Henry's coefficient, altering the equilibrium values (x^e).

And for the second experiment, when the CO_2 concentration in the inlet stream increases, the mass transfer coefficient also increases slightly. Although this is not expected because the mass transfer coefficient does not depend on the concentrations, increasing the CO_2 flux while keeping the rest constant generates an increase in turbulence that pushes the system away from equilibrium and facilitates the transfer of mass.

On the other hand, it is necessary to underline the limitations that have been faced throughout the experimentation.

One of the aspects that must be taken into account before proceeding to work with the equipment are the working absorption and desorption temperatures. The minimum absorption temperature is specified by the cooling capacity and the possible heat transfer in the heat exchanger, and the maximum desorption temperature is specified by the available heating capacity and the possible heat recovery from the heat exchanger. Both temperatures depend on each other and on the ambient temperature, and therefore, if these temperatures are not selected correctly, they can lead to experimental errors.

Following on from the previous point, when working with the equipment in a closed circuit, i.e. with desorption in operation, it is important to consider that the lower the desorption temperature, the lower will be the desorption capacity of the equipment. Consequently, the CO_2 concentrations in the inlet stream of the absorption column will increase, gradually reducing the absorption capacity until the water is saturated.

Finally, another aspect to take into account is the working pressure in the absorption column. It is recommended that this be slightly higher than atmospheric pressure to overcome the pressure drop in the column without affecting the gas sampling.

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10. ACRONYMS

Symbols for formulas and units	
n_i	Amount of substance of component i
n	Amount of substance CO_2
G'_{air}	Molar flow rate of air
G'_{CO2}	Molar flow rate of CO_2
G'	Molar flow rate of gas mixture
G_{CO2}	Volumetric flow rate of CO_2
G_{air}	Volumetric flow rate of air
G	Volumetric flow rate of gas mixture
L'	Molar flow rate of the water
L	Volumetric flow rate of the water
p	Pressure
p_i	Partial pressure of component i
$p_{0,\mathrm{i}}\,(\mathrm{T})$	Saturation vapor pressure of component i
$PD1$	Differential pressure in the upper half of the column
$PD2$	Differential pressure in the lower half of the column
R_m	Universal gas constant
T_0	Temperature at 0°C in Kelvin: 273 K
T_{25}	Temperature at 25°C in Kelvin: 298 K
T	Temperature
V	Volume

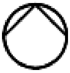
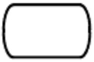
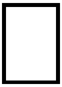
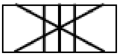
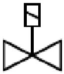
Z	Height of column filling
S	Area
x_i	Mole fraction in the liquid of component i
X_i	Liquid loading with component i
y_i	Mole fraction in the gas of component i
Y_i	Gas loading with component i
x^{if}	Mole fraction in the liquid of component i in the interface
y^{if}	Mole fraction in the gas of component i in the interface
x^e	Mole fraction in the liquid of component i in the equilibrium
y^e	Mole fraction in the gas of component i in the equilibrium
a	Specific area filling
k_x	Individual volumetric coefficient of mass transfer for the liquid phase
k_y	Individual volumetric coefficient of mass transfer for the gas phase
K_x	Global volumetric coefficient of mass transfer for the liquid phase
K_y	Global volumetric coefficient of mass transfer for the gas phase
K_L	Global volumetric coefficient of mass transfer for the liquid phase
K_G	Global volumetric coefficient of mass transfer for the gas phase
M	Molecular mass
N_i	Flux density of the solute i
H_{cp}	Henry Coefficient. Units: $mol_{gas} / (L \cdot atm)$
H_{px}	Henry Coefficient. Units: $(atm \cdot mol_{water}) / mol_{gas}$
J_i	Diffusion of solute i
ρ	Density
ρ_M	Molar density
pH	Hydrogen Potential
m	Slope

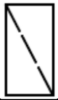
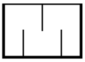
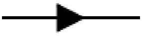
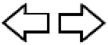
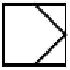
Substances chemical formula

CO_2	Carbon dioxide
H_2O	Water
N_2	Nitrogen
O_2	Oxygen
Ar	Argon
He	Helium
Ne	Neon
CH_4	Methane
Kr	Krypton
H_2	Hydrogen
Xe	Xenon
O_3	Ozone
NO_2	Nitrogen dioxide
I_2	Iodine
CO	Carbon monoxide
CO_3^{2-}	Ionic carbonate
HCO_3^-	Ionic hydrogen carbonate
H_2CO_3	Carbonic acid
NaOH	Sodium hydroxide
HCl	Hydrochloric acid
OH^-	Ionic hydroxide
H^+	Hydron

APPENDICES

11. APPENDIX 1: LIST OF SYMBOLS FOR THE FLOWCHART

SYMBOL	DENOMINATION
	Pump, in general
	Vacuum pump of the pumped medium
	Compressor
	Deposit, in general
	Deposit, in general
	Heating or cooling
	Plate heat exchanger
	Tube accessory, in general
	Tube accessory, manual actuation
	Tube accessory with magnetic actuation
	Tube accessory with constant adjustment behaviour

	Tube accessory for ventilation and air venting
	Tube accessory for retention
	Valve, in general
	Safety valve
	Sludge collector
	Silencer
	Coupling
	Pressure reducer
	Rotameter
	Flow line
	Line of action
	Measuring point with on-site indication
	Measuring point with remote evaluation
	Input/output of a substance
	Regulator
	Flexible tube

12. APPENDIX 2: EQUIPMENT ABBREVIATIONS

Equipment abbreviations

A1	Cooling unit
A2	Heating prior to the entrance of the desorber
K1	Absorption column (absorber)
K2	Regeneration column (desorber)
P1	Pump for transport to the absorption column
P2	Pump for transport to the regeneration column
P3	Circulation pump in the cooling circuit
P4	Vacuum water jet pump
V	Air compressor
W2/W3	Heat exchanger
W4	Plate heat exchanger
V1	CO_2 regulation valve
V2	Air regulation valve
V3	Non-return valve
V4	Spherical tap for gas sampling at the base of the column
V5	Spherical tap for gas sampling in the centre of the column
V6	Spherical tap for gas sampling at the column head
V7	Spherical tap
V8	Ball valve for releasing the pressure measuring point from the centre of the column
V9	Ball valve for releasing the pressure measuring point of the column head
V10	Regulation valve for adjusting the column pressure

V11	Safety valve in the absorption column
V12	Regulating valve for adjusting the volumetric water flow rate
V13	Spherical tap for taking liquid samples from the absorption column
V14	Ball valve for draining the water-cooling circuit
V15	Spherical tap for sampling of the regenerated absorption medium
V16	Solenoid control valve for level control in the absorption column K1
V17	Control valve for the absorption medium upstream of the regeneration column K2
V18	Non-return valve
V19	Spherical tap to compensate for depression in column K2 after the test
V20	Regulating valve for the water volume of the vacuum water jet pump
V21	Regulating valve for adjusting the volume flow of the supply air
V22	Solenoid valve for filling the columns (K1 and K2) in water
V23	Ball valve for blocking the water supply tap by hand
V24	Spherical tap for emptying the regeneration column
V25	Ball valve to release the pressure measuring point at the base of the column
V26	Non-return valve
V27	Spherical tap
V28	Sludge trap
V29	Silencer
V30	Sludge trap
V31	Sludge trap
V32	Silencer
V33	Non-return valve
V34	Overpressure safety valve.
V35	Regulating valve
V36	Silencer

Characteristic letters for equipment, machines, pipe fittings and tubing:

Characteristic letter	**Measured variable or other measurand of input variable, actuator**		**Processing**
	As initial	**Supplementary letter**	**As a successive letter (orden I, R, C)**
C			Automatic regulation
F	Flow rate	Relation	
I			Indication
P	Pressure		
T	Temperature		Transmitter

13. APPENDIX 3: TECHNICAL DATA OF THE CE400

Measurements of the columns:

- Material: Duran glass
- Outer diameter: 90 mm
- Inner diameter: 80 mm
- Length of K1: 750 mm
- Filling height at K1: 400 mm
- Length of K2: 750 mm
- Filling: The tests are carried out with Raschig rings made of stainless steel 1.4301 with the dimensions: 8 x 8 x 0.3 mm and with a specific filling area of 690 m^2/m^3

Rotameter FI1 (CO_2):

- Measuring range: 0.4 to 5.4 L/min
- Pressure: 1.013 bar abs.
- Temperature: 20.0 °C

Rotameter FI2 (air):

- Measuring range: 0.2 to 2.4 m^3/h
- Pressure: 1.013 bar abs.
- Temperature: 20.0 °C

Rotameter FI3 (water):

- Measuring range: 50 to 600 L/h
- Temperature: 20 °C

Measuring instrument for CO_2:

- Measuring range: 0 - 100 Vol% CO_2 in steps of 0.1 %.
- Measuring principle: NDIR sensor
- Temperature range: 5 to 40 °C

14. APPENDIX 4: TEMPERATURES AND PRESSURES MEASURED IN THE EXPERIMENTS 1 AND 2

Readings of the temperatures and pressures of the equipment for the experiments carried out:

	Absorption column K1			Desorption column K2			Other parameters		
Exp1 at 11.5ºC	Water height=	variable	cm	Water height=	variable	cm	P CO_2 inlet =	0.3	bar
	PI2 (gas inlet) =	0.1	bar	PI3 =	-0.2	bar	T heater =	40	ºC
	TI1 (gas inlet) =	22	ºC	TI3 (liquid inlet) =	40	ºC	T cooler =	20	ºC
	TI2 (liquid inlet) =	19	ºC	TI6 (liquid outlet) =	38	ºC	TI5 =	36	ºC
	TI4 (liquid outlet) =	20	ºC				TI7 =	36	ºC
Exp1 at 20ºC	Water height=	variable	cm	Water height=	variable	cm	P CO_2 inlet =	0.3	bar
	PI2 (gas inlet) =	0.1	bar	PI3 =	-0.2	bar	T heater =	40	ºC
	TI1 (gas inlet) =	20.4	ºC	TI3 (liquid inlet) =	40	ºC	T cooler =	20	ºC
	TI2 (liquid inlet) =	20.3	ºC	TI6 (liquid outlet) =	38	ºC	TI5 =	36	ºC
	TI4 (liquid outlet) =	20.4	ºC				TI7 =	36	ºC
Exp. 2 (0.5 L/min CO_2)	Water height=	variable	cm	Water height=	variable	cm	P CO_2 inlet =	0.3	bar
	PI2 (gas inlet) =	0.1	bar	PI3 =	-0.2	bar	T heater =	-	ºC
	TI1 (gas inlet) =	25	ºC	TI3 (liquid inlet) =	(23 to 26)	ºC	T cooler =	13	ºC
	TI2 (liquid inlet) =	20	ºC	TI6 (liquid outlet) =	23	ºC	TI5 =	23	ºC
	TI4 (liquid outlet) =	21	ºC				TI7 =	21	ºC
Exp. 2 (1 L/min CO_2)	Water height=	variable	cm	Water height=	variable	cm	P CO_2 inlet =	0.3	bar
	PI2 (gas inlet) =	0.1	bar	PI3 =	-0.2	bar	T heater =	-	ºC
	TI1 (gas inlet) =	27	ºC	TI3 (liquid inlet) =	26	ºC	T cooler =	13	ºC
	TI2 (liquid inlet) =	20	ºC	TI6 (liquid outlet) =	27	ºC	TI5 =	26	ºC
	TI4 (liquid outlet) =	21	ºC				TI7 =	21	ºC

15. APPENDIX 5: RESULTS OF EXPERIMENT 1

Experimental data of the pure CO_2 and water system for a cooling temperature of 11.5ºC:

Flow rates		**Pressure drops**		**Water samples S4**			**Water samples S5**		
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)	**V^*_1 HCl** (mL)	**V^*_2 HCl** (mL)	**ΔV^*** (mL)
2	100	1	110	10.7	18.2	7.5	14.7	18.4	3.7
2	100	1	120	10.8	18.3	7.5	14.6	18.3	3.7
2	100	1	110	10.7	18.2	7.5	14.7	18.4	3.7
2	200	1	105	10.2	18.2	8.0	14.5	18.4	3.9
2	200	1	110	10.1	18.2	8.1	14.7	18.5	3.8
2	200	1	110	10.1	18.1	8.0	14.6	18.5	3.9
2	280	1	110	9.8	18.1	8.3	13.3	18.1	4.8
2	280	1	100	9.9	18.2	8.3	13.2	18.1	4.9
2	280	1	100	9.8	18.1	8.3	13.3	18.1	4.8

Experimental data of the pure CO_2 and water system for a cooling temperature of 20ºC:

Flow rates		**Pressure drops**		**Water samples S4**			**Water samples S5**		
CO_2 (L/min)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)	**V^*_1 HCl** (mL)	**V^*_2 HCl** (mL)	**ΔV^*** (mL)
2	100	1	170	12.0	18.4	6.4	15.0	18.3	3.3
2	100	1	150	12.1	18.0	5.9	15.1	18.4	3.3
2	100	1	150	12.0	18.1	6.1	14.9	18.3	3.4
2	200	1	135	16.2	25.4	9.2	14.0	18.5	4.5
2	200	1	122	12.9	19.3	6.4	13.9	18.4	4.5
2	200	1	140	13.0	18.6	5.6	14.0	18.5	4.5
2	280	4	130	11.3	18.4	7.1	14.5	18.2	3.7
2	280	5	100	11.4	18.5	7.1	14.4	18.3	3.9
2	280	4	100	10.8	18.1	7.3	14.4	18.0	3.6

16. APPENDIX 6: RESULTS OF EXPERIMENT 2

Experimental data of the experiment with a gas mixture 1 L/min CO_2 + 0.3 Nm^3/h air:

Flow rates			**Pressure drops**		**Gas samples**				**Water sample S5**		
CO_2 (L/min)	**Air** (Nm^3/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**%vol. CO_2 S1**		**%vol. CO_2 S3**		**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)
1	0.3	100	90	55	23.4	23.5	23.3	23.3	15.9	18.7	2.8
1	0.3	100	85	53	23.4	23.6	23.3	23.3	16.0	18.8	2.8
1	0.3	100	90	57	23.4	23.6	23.3	23.2	16.0	18.7	2.7
1	0.3	100			23.5	23.6	23.2	23.2			
1	0.3	100			23.5	23.6	23.3	23.2			
1	0.3	200	100	60	23.2	23.2	23.5	23.7	15.8	18.7	2.9
1	0.3	200	100	60	23.2	23.1	23.5	23.8	15.8	18.7	2.9
1	0.3	200	95	57	23.3	23.0	23.6	23.8	15.8	18.7	2.9
1	0.3	200			23.1	23.0	23.6	23.7			
1	0.3	200			23.1	23.0	23.7	23.7			
1	0.3	225	105	60	23.0	22.8	23.6	23.7	16.0	18.7	2.7
1	0.3	225	105	60	23.0	22.7	23.7	23.7	16.0	18.8	2.8
1	0.3	225	100	55	22.9	22.7	23.5	23.8	16.0	18.7	2.7
1	0.3	225			22.8	22.7	23.5	23.7			
1	0.3	225			22.9	22.7	23.6	23.8			
1	0.3	275	115	62	22.5	22.3	23.9	23.7	16.1	18.7	2.6
1	0.3	275	110	60	22.5	22.4	23.9	23.7	16.2	18.8	2.6
1	0.3	275	115	65	22.4	22.4	24.0	23.6	16.1	18.7	2.6
1	0.3	275			22.5	22.5	24.0	23.6			
1	0.3	275			22.3	22.7	23.8	23.6			

Experimental data of the experiment with a gas mixture 0.5 L/min CO_2 + 0.3 Nm^3/h air:

Flow rates			**Pressure drops**		**Gas samples**				**Water sample S5**		
CO_2 (L/min)	**Air** (Nm^3/h)	**Water** (L/h)	**PD1** (mm c.a.)	**PD2** (mm c.a.)	**%vol. CO_2 S1**		**%vol. CO_2 S3**		**V_1 HCl** (mL)	**V_2 HCl** (mL)	**ΔV** (mL)
0.5	0.3	100	105	100	13.8	13.8	13.5	13.6	16.7	18.5	1.8
0.5	0.3	100	105	110	13.9	13.8	13.6	13.6	16.6	18.4	1.8
0.5	0.3	100	105	105	13.8	13.7	13.6	13.7	16.7	18.5	1.8
0.5	0.3	100			13.9	13.7	13.5	13.7			
0.5	0.3	100			13.9	13.8	13.5	13.6			
0.5	0.3	200	85	110	13.9	14.0	13.5	13.6	16.4	18.6	2.2
0.5	0.3	200	90	100	13.9	14.0	13.5	13.6	16.4	18.5	2.1
0.5	0.3	200	90	100	13.9	14.1	13.6	13.5	16.3	18.4	2.1
0.5	0.3	200			13.9	14.1	13.5	13.5			
0.5	0.3	200			14.0	14.0	13.5	13.6			
0.5	0.3	225	70	58	13.9	13.9	13.5	13.5	16.4	18.5	2.1
0.5	0.3	225	75	60	14.0	13.9	13.5	13.4	16.4	18.5	2.1
0.5	0.3	225	80	65	14.0	14.0	13.4	13.4	16.4	18.5	2.1
0.5	0.3	225			14.0	14.1	13.5	13.3			
0.5	0.3	225			13.9	14.1	13.5	13.4			
0.5	0.3	275	85	60	14.0	14.0	13.3	13.4	16.4	18.6	2.2
0.5	0.3	275	80	50	14.1	13.9	13.3	13.4	16.4	18.6	2.2
0.5	0.3	275	80	55	14.0	13.9	13.3	13.4	16.4	18.5	2.1
0.5	0.3	275			14.1	13.9	13.4	13.5			
0.5	0.3	275			14.0	14.0	13.4	13.5			