

Project no. 608535

INTERACT

INnovaTive Enzymes and polyionic-liquids based membRAnes as CO₂ Capture Technology

Deliverable 2.2:

Complete robust dataset for kinetics and kinetic model for most promising sets of enzyme-solvents systems for model validation

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Summary

Absorption experiments for four different solvent types were conducted in a wetted wall column setup with different enzyme concentration. The different solvents investigated were, MEA (monoethanolamine), AMP (aminomethylpropanol), MDEA (methyldiethanolamine) and K_2CO_3 . MEA showed under comparable absorber conditions the highest absorption flux, followed by AMP, then MDEA and K_2CO_3 for the pure solvent. MDEA (2.6 fold) and K_2CO_3 (4.2-fold) showed a great absorption rate enhancement when adding 0.5 % (w/w) enzyme solution to the solvent, whereas the absorption rate of AMP (1.1 fold) was just slightly increased. For MEA the addition of enzyme showed a negative effect. Solvents from the group of tertiary amines (MDEA) and carbonate salt solutions (K_2CO_3) show the best effect from enzyme addition. For the solvent K_2CO_3 the effect of enzyme concentration on the absorption rate was also investigated for 4 different enzyme concentrations in the range of 0-1%. A clear increase in absorption rate could be observed with increase of enzyme concentration. When assuming pseudo first order reaction regime the increase in overall reaction rate was found to be linear dependent on the enzyme concentration.



1. Introduction

1.1. Scope of the project

In this task enzyme kinetics will be investigated for solvents that showed promising stability for the enzyme. Small scale experiments were carried out both incorporating mass transfer limitation and avoiding mass transfer limitation. Absorption rate measurements are carried out in a wetted wall column under similar process conditions as in the absorber. In a flow cell reactor the pure enzyme kinetics are observed with no mass transfer limitation.

1.2. Methodology

In order to determine the potential of carbonic anhydrase enzymes in the carbon capture technology different solvent types are investigated. Based on their different structure and different reaction mechanism with CO₂, monoethanolamine (MEA) a primary was chosen, besides that methyldiethanolamine (MDEA), a tertiary amine, aminomethylpropanol (AMP), a sterically hindered primary amine and potassium carbonate (K₂CO₃), an alkaline salt are compared in their performance together with and without the addition of enzyme.

Monoethanolamine (MEA), is a fast reacting primary amine solvent that can be seen as a reference in the field of carbon capture. Methyldiethanolamine (MDEA) represents the group of tertiary amines, with slow kinetics but favourable desorption characteristics. Aminomethylpropanol (AMP) is a sterically primary hindered amine, which follows a similar reaction mechanism as MEA, but contrarily forms bicarbonate as a product instead of carbamate. Potassium Carbonate (K₂CO₃) is a alkaline salt that follows a base catalysis mechanism in reaction with CO₂.

All chosen solvents except MEA form bicarbonate under the ambient absorber conditions. In the following experiments it will be investigated how the enzyme addition changes the absorption rate. The extend of absorption rate change can reveal how the enzyme interact with the solvent and shows which solvent groups is the most promising in working with enzymes.

1.3. Reaction mechanism

The chemical absorption technology used for carbon capture obtains its advantage over physical absorption from the reaction between the solvent and CO₂. The decrease in dissolved CO₂ can increase the mass transfer as it produces a sharper concentration gradient. The group of amines have proven over years that they are the most promising group of chemical solvent for post combustion carbon capture. This group can be divided depending on the number of substituents on the N-atom into primary, secondary and tertiary amine; when the α-C atom has a substituent the term sterically hindered amine is used.

Primary and secondary amines that have a free Hydrogen group on the N-atom can directly attack the CO₂ in solution and form an unstable zwitterion as it can be seen in equation (1). This zwitterion is then reduced to a carbamate by the removal of the hydrogen from a base. In an aqueous solution the base (B) can be a second amine, the hydroxide (OH⁻) or water, however usually only the amine can be regarded as a deprotonation base.



Tertiary amines cannot react directly with CO₂, but follow a bases catalysis mechanism:



Sterically hindered amines represent a special group, as they follow a reaction mechanism according to their number of substituents on the N-atom, for instance a primary sterically hindered amine still reacts directly with a CO₂ forming a zwitterion, but unless normal primary amines it does not react further with a base forming a carbamate. Due to the steric hindrance this amine group cannot form a stable carbamate species, the zwitterion is afterwards hydrolysed to Bicarbonate and a deprotonated amine.

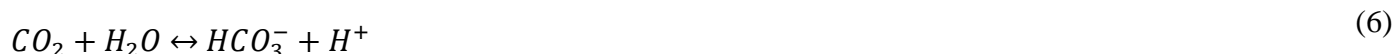
In aqueous solutions parallel reactions are occurring between water and CO₂ which are:



Carbonate salt solutions react in a base mechanism according to equation (5).

The direct reaction mechanism of primary and secondary amines is much faster than the base catalysis leading to fast kinetics and higher mass transfer. Contrarily the heat of reaction is lower for tertiary amines and no additional energy is required to split the carbamate in the desorption process leading to more favourable desorption. It can be seen, that the bicarbonate (HCO₃⁻) plays a major role in the reaction kinetics of tertiary amines and carbonate salt solutions and to some extent in sterically hindered primary amines as their zwitterion is hydrolysed to bicarbonate. It should be noted that these reaction mechanisms are describing reactions ongoing in ambient absorber conditions which is a temperature of about 40 °C, ambient pressure and a CO₂ concentration of around 10-12 vol%. At much higher pressures or CO₂ concentrations there might be different reaction mechanisms involved.

When looking at the reaction mechanism of the enzyme carbonic anhydrase it becomes obvious why the bicarbonate formation is an important issue as it catalyses the reaction between dissolved CO₂ and water to form bicarbonate



As bicarbonate is formed in tertiary, sterically hindered amines and alkaline carbonate salt solutions, carbonic anhydrase supplementation may influence the reaction rate and absorption rate.

1.4. Mass transfer theory

Mass transfer between two phases can be best explained with the two film model where it is assumed, that on both sides of the gas/liquid interface a laminar film exists through which the compound can just pass by diffusion. The partial pressure and concentration profile of CO₂ in a chemical absorption process can be seen in Figure 1. In the partial pressure profile the curve does not change at the interface whereas when the liquid side is described with concentration rather than partial pressure a jump at the interface can be observed, this can be easily explained with the fact that for describing a gas system with a partial pressure being in equilibrium to a liquid with a concentration some sort of conversion factor is needed. The Henry's coefficient (H) is used for that correlation:

$$P_{CO_2}^{int} = H_{amine} \times C_{CO_2}^{int} \quad (7)$$

This reveals that there is actually no jump at the interface; the left side is using partial pressure as basis, whereas the right side uses concentration and the multiplier between those two is the Henry's coefficient. When looking at the profiles in both phases it appears that in the gas phase the profile is linear whereas there is a curvature in the liquid side. The gas side profile can be described with physical diffusion through the laminar layer, but in the liquid



side besides diffusion also reaction of the component occurs. This will lead to a higher decrease than sole diffusion and is the reason for enhanced mass transfer in chemical absorption. A very simple way of describing this mass transfer increase is the use of an Enhancement factor E , which is the ratio of mass transfer with chemical reaction to mass transfer purely by physical means:

$$E = \frac{\text{mass transfer with chemical reaction}}{\text{mass transfer physical}} \quad (8)$$

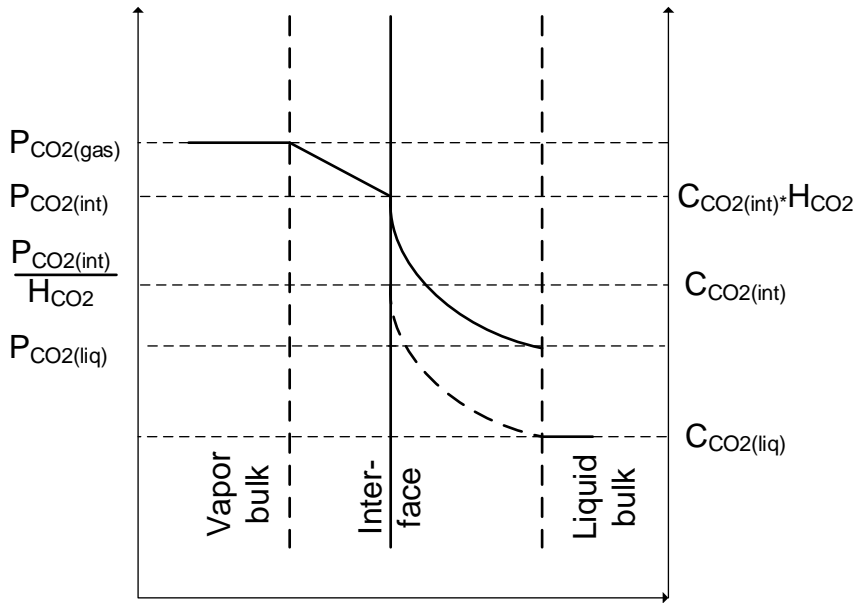


Figure 1: Partial pressure and concentration profile of CO_2 in a chemical absorption process in the two film model; solid line corresponds to the partial and dashed line to concentration.

In the absorption process the flux of CO_2 from the gas bulk to the interface can be described as the product of the gas side mass transfer and the driving force, which is the difference of CO_2 partial pressure between the bulk and the interface:

$$N_{\text{CO}_2}^{\text{gas}} = k_{\text{CO}_2}^{\text{gas}} \times (P_{\text{CO}_2}^{\text{gas}} - P_{\text{CO}_2}^{\text{int}}) \quad (9)$$

In the same manner can the flux from the interface to the liquid bulk can be described

$$N_{\text{CO}_2}^{\text{liq}} = k_{\text{CO}_2}^{\text{liq}} \times (P_{\text{CO}_2}^{\text{int}} - P_{\text{CO}_2}^{\text{liq}}) = K_{\text{CO}_2}^{\text{liq}} (C_{\text{CO}_2}^{\text{int}} - C_{\text{CO}_2}^{\text{liq}}) \quad (10)$$

In that left expression the term partial pressure of CO_2 in the liquid phase is used instead of concentration of CO_2 . But through the Henry's law the partial pressure can be translated into concentration using the apparent Henry's coefficient for that solvent. As there is no accumulation of CO_2 at the interface the flux of CO_2 through the gas film is the same as the flux through the liquid film, so the flow can also be describe with an overall mass transfer coefficient k_{ov} :

$$N_{\text{CO}_2} = K_{\text{CO}_2}^{\text{ov}} \times (P_{\text{CO}_2}^{\text{gas}} - P_{\text{CO}_2}^{\text{liq}}) \quad (11)$$

A resistance is the ratio between the driving force and its corresponding flux. Applying this analogy for the mass transfer from the gas to the liquid and using the equations 9 to 11 the overall resistance to the mass transfer can be described as the combined resistances of the liquid and gaseous laminar film

$$\frac{1}{K_{CO_2}^{ov}} = \frac{He}{Ek_{CO_2}^{liq}} + \frac{1}{k_{CO_2}^{gas}} \quad (12)$$

When the liquid side mass transfer is the rate determining step of the absorption and the contribution of gas side is considered small, the gas side resistance can be neglected. Absorption into an unloaded solvent, the concentration/partial pressure of CO₂ in the liquid is then 0, leads to a simplified equation:

$$\frac{N_{CO_2}}{P_{CO_2}^{gas}} = k_{CO_2}^{liq} \quad (13)$$

This equation predicts a linear dependency between the absorption flux of CO₂ and the partial pressure in the gas phase, the ratio of these two is the overall mass transfer and can be approximated the liquid side mass transfer in regions where the gas side contribution is small.

The liquid side mass transfer coefficient $k_{CO_2}^{liq}$ can be described with the Enhancement factor from equation (8). This Enhancement factor is equal with the Hatta number Ha if the conditions of pseudo first order reaction are met ($3 < Ha$). This turns equation (13) into:

$$\frac{N_{CO_2}}{P_{CO_2}^{gas}} = \frac{\sqrt{D_{CO_2}k_{ov}}}{H_{CO_2}} \quad (14)$$

With D_{CO_2} being the diffusion coefficient of CO₂ in the liquid and k_{ov} the overall reaction constant of CO₂ in the liquid phase, not to be mistaken for $K_{CO_2}^{ov}$ the overall mass transfer coefficient. This overall rate constant is sometimes also referred to as k_{obs} as it is the observed rate constant and accounts for all ongoing reactions. The terms on the left side in this equation can be measured with the wetted wall column, the Diffusion coefficient and the Henry's coefficient needs to be taken from literature or from independent experiments. It is though possible to compare the overall reaction rate in the liquid with and without enzyme without the knowledge of the Henry's coefficient and the diffusion coefficient, assuming that this value is not altered from adding small amounts of enzyme to the solution:

$$\frac{k_{ov}^{enz}}{k_{ov}^{sol}} = \frac{\left(\frac{N_{CO_2}}{P_{CO_2}^{gas}}\right)_{enz}^2}{\left(\frac{N_{CO_2}}{P_{CO_2}^{gas}}\right)_{sol}^2} \quad (15)$$



2. Experimental Equipment

The following chapter describes the experimental setup and procedure of the wetted wall column that is used for measuring the enzyme kinetics.

2.1. Wetted wall column

2.1.1. Setup

The wetted wall column is a gas-liquid contactor that allows carrying out mass transfer experiments a very well defined process conditions. This setup consists of two different systems, the liquid system and the gas system that get in contact in the wetted wall chamber. Inside this chamber the liquid is flowing down a small metal pipe in contact with the upstreaming gas stream. From all the auxiliary equipment it is possible to set the desired process conditions very precisely and conduct steady state experiments.

The liquid system (the blue part of Figure 2) works with a closed liquid loop and a big liquid reservoir ensuring no change in CO₂ concentration in the liquid during experiments. The liquid is pumped from the tank through a heat exchanger to heat up the solvent to the desired reactor temperature and then to the wetted wall column, the flow rate can be monitored on a Rotameter. The liquid then enters the chamber from the bottom by rising up inside the small metal pipe, and then it flows down by gravimetric force resulting in a ripple free film. From a correlation of a free falling liquid film running down a vertical plate the film thickness can be estimated. It can be seen from Figure 3, that the actual contact area between the gas and liquid is higher than the sole dimensions of the pipe, as the liquid film thickness needs to be accounted for. From the high liquid to gas ratio inside the reaction chamber it can be assumed that the CO₂ concentration in the liquid does not change inside the wetted wall, as the amount of absorbed CO₂ is small compared to the liquid flow. The liquid is collected at the bottom and then pumped back into the liquid reservoir.

The gas system (the red part of Figure 2) is an open system. This gas stream is pumped through two saturators, one at ambient temperature and the other at reactor temperature, to ensure saturation of the gas stream. This gas stream can now be led either through the wetted wall column or through the bypass. Independently which way the gas follows the gas temperature and CO₂ concentration are measured.

The absorption flux is measured by a component balance over the gas stream. The gas is first set on bypass mode and all process condition, including CO₂ concentration are taken down, the gas stream is then set through the wetted wall column and the new CO₂ is measured in the gas stream. The absorption flux is calculated from that CO₂ concentration decrease.

$$\Delta N_{CO_2} = \frac{N_{Inert} \times (y_{CO_2in} - y_{CO_2out})}{(1 - y_{CO_2in}) \times (1 - y_{CO_2out})} \quad (1)$$

Table 1: Equipment used on the wetted wall column

Device	description	specification
Mass flow controller	EL-FLOW from Bronkhorst	0-20 nl/min N ₂ and 0-2 nl/min CO ₂
CO ₂ probe	Vaisala	0-20 vol % at ambient conditions
Liquid flowmeter	Sho-rate rotameter	Non corrosive sapphire ball
Wetted wall chamber	Custom built	See dimensions in Figure 3



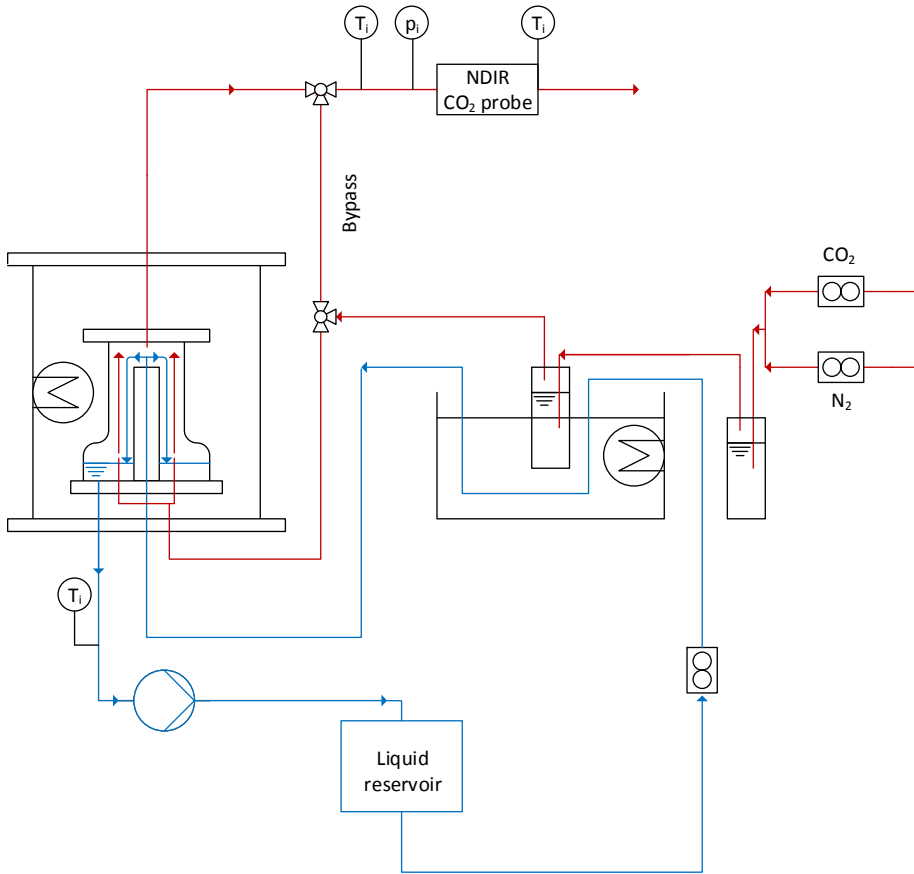


Figure 2: Wetted wall column setup, liquid system in blue color and gas system in red color

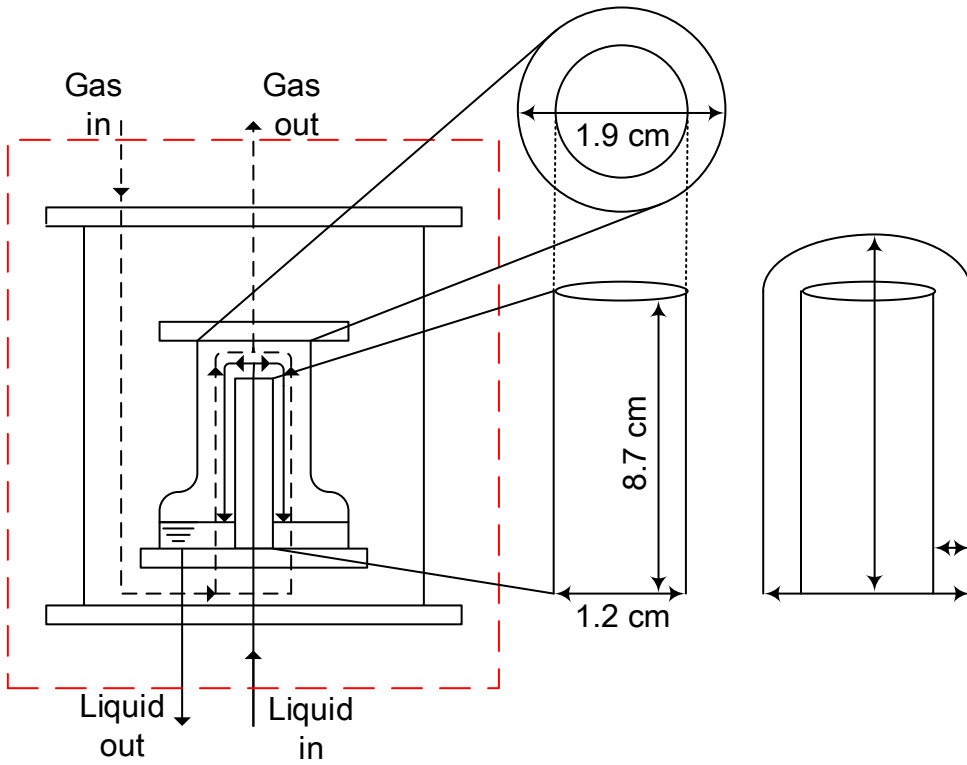


Figure 3: Dimensions of the wetted wall column



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2.1.2. Experimental procedure

The absorption experiments were conducted under similar conditions. The liquid flow was pumped in a closed loop; the mass flux (approx. 4 ml/s) as well as the solvent temperature was observed. The gas stream was set with the mass flow controller to result in a total gas stream of 3 nl/min. This gas stream was first bypassed and the CO₂ conc was checked, when the concentration was stable the valve was switched and the gas was led through the wetted wall column and the decrease in CO₂ was measured, after all process parameters were stabilized, the gas stream was set into bypass mode again and a different CO₂ concentration was set with the mass flow controller. In these experiments the CO₂ concentration was varied between 0 and 18 vol% resulting in CO₂ partial pressures of 0-20 kPa.

Working with the same total gas stream in all experiments allows better comparison, as the gas velocity influences the gas side mass transfer. The experimental results are easier to compare when the gas side resistance is the same in all experiments. The experiments are carried out under conditions where the liquid side mass transfer is the rate determining step of absorption. An overview of the process parameters for the wetted wall column experiments can be seen in Table 2.

Table 2: Process conditions for wetted wall experiments

Process parameter	description	specification
Gas flow	3 nl/min	Total gas, N ₂ plus CO ₂
Liquid flow	Approx. 4 ml/s	Liquid flow high enough to make no impact on absorption
CO ₂ conc.	0-18%	N ₂ flow was lowered accordingly
Temperature	313 K	For gas and liquid system
pressure	Approx. 1,2 bar	

2.2. Flowcell reactor

In the previous chapter, it was shown how the mass transfer coefficients can be determined. For a transfer of those coefficients to other systems and technologies, it would be beneficial to cancel out the effect of the pure reaction from the mass transfer coefficients. One possibility to do so, is to measure the intrinsic reaction rates on a first step which can be subsequently subtracted from the measured mass transfer coefficients to determine the intrinsic mass transfer coefficients. Therefore, a setup based on a flowcell reactor for measurement of the intrinsic reaction kinetics has been developed. Alternatively, a pressurized reactor setup will be used to compare the experiments in both setups. This also enables the quick screening of potential other solvent candidates in the future.

2.2.1. Setup

The setup is briefly described in Figure 5. Basically, the main idea is to distinguish between a zone in which CO₂ is transferred from the gas to the liquid side and a zone in which the actual reaction takes place after the reactive solvent with the enzyme is mixed with the CO₂ filled water.



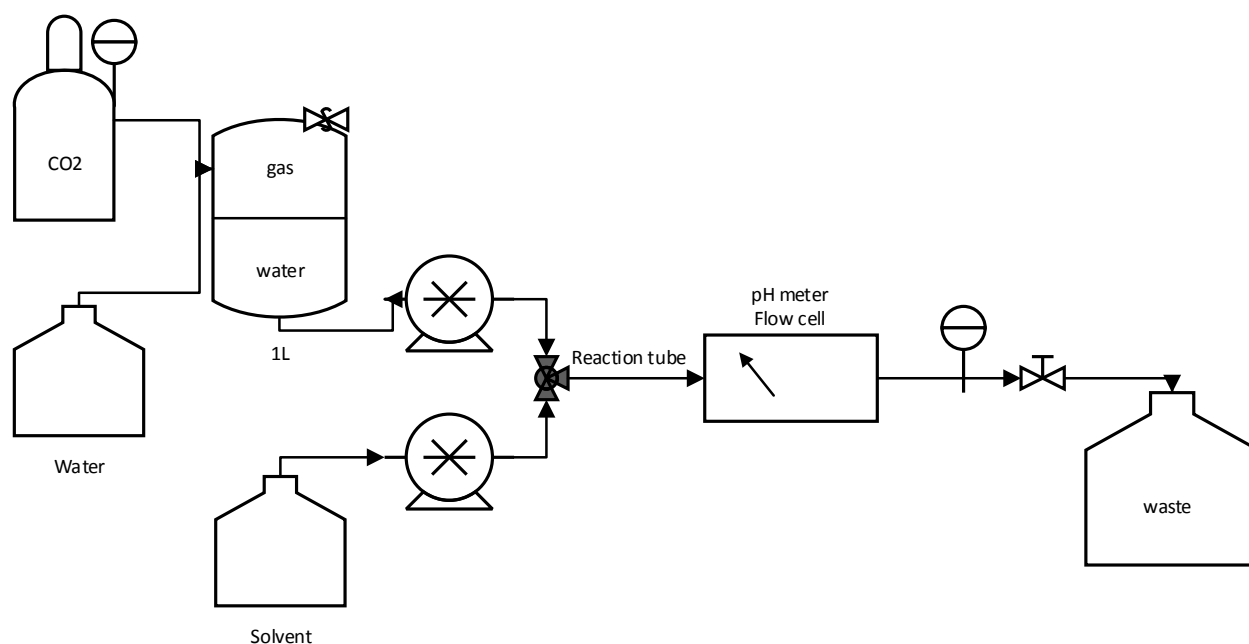


Figure 4: Experimental set up for continuous flow-cell reactor.

2.2.2. Experimental procedure

Preparation: 0.5L of H₂O is added to tank, 100% CO₂ from tank is added, up to 5 bar, several times with release of gas in between each addition to ensure that air is removed. The tank is then left over night to equilibrate. Dissolved CO₂ concentration ranges from 70mM (2Bar) to 180mM (5Bar).

Before reaction the solvent solutions are prepared, they contain 10-30 w/w % solvent (MEA, MDEA, AMP, K₂CO₃, and so on), and from 0-3% enzyme solution.

Reaction: The solvent and saturated water solution is pumped at 0.1-2 mL/min in the reaction tube which is 0.5mm in diameter and either 5 (0.0092 mL) or 100 cm (0.2mL) long. Thus, reaction time varies between 0.1s and 60s. pH is measured in the flow meter, and from this reaction kinetics is determined.

Initial tests with this set-up were carried out with the solvent MDEA and K₂CO₃, the preliminary results shown a rate enhancement by the enzyme.

3. Results

3.1. Wetted wall column

3.1.1. Gas side mass transfer contributions

In a first set of experiments the absorption rate into unloaded 30 wt% MEA was investigated at different gas velocities. As can be seen from Figure 5 all experiments follow a linear trend which is very well in accordance to equation (11). The liquid phase is the same, just the gas velocity and thus the gas side resistance is changed throughout these experiments. We can observe that for velocities above 0.34 m/s the absorption rate does not change showing that the mass transfer in this region is limited just by the liquid side mass transfer. All further experiments were conducted at a gas velocity of 0.34 m/s which corresponds to a total gas volume stream of 3 nl/min.

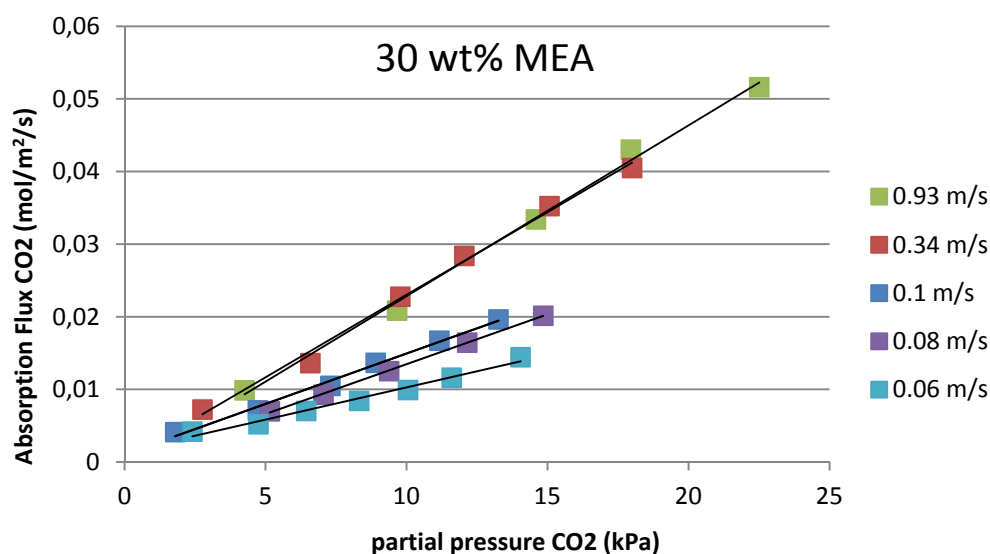


Figure 5: Absorption Flux of CO₂ into 30 wt% MEA at 313 K for different gas velocities

3.1.2. Effect of the enzyme

In Figure 6 the absorption fluxes of CO₂ for the solvents MDEA, and K₂CO₃ are shown with 0 and 0.5 wt% enzyme solution, both solvents demonstrated a clear increase in absorption rate when the enzyme carbonic anhydrase was present. The effect of adding enzyme to AMP and MEA is shown in Figure 7. AMP shows a slight increase whereas a decrease in MEA absorption can be seen. The effects causing this decrease need to be investigated in future experiments. This is very well in accordance to the expectation for the bicarbonate forming solvents MDEA and K₂CO₃. The small effect of absorption rate enhancement for AMP could be explained with the different reaction mechanism with CO₂ as it does not follow a base catalysis mechanism as MDEA and K₂CO₃. The decrease for MEA is somehow surprising, as there should be rather no effect than a negative effect.

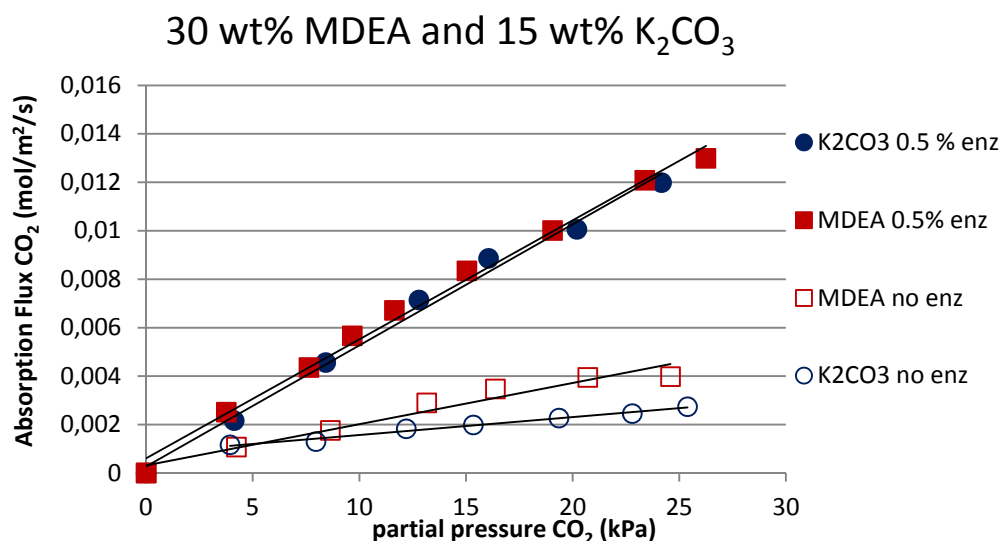


Figure 6: Absorption flux of CO₂ into 15 wt% K₂CO₃ and 30 wt% MDEA at 313 K with 0 and 0.5 % (w/w) enzyme solution.

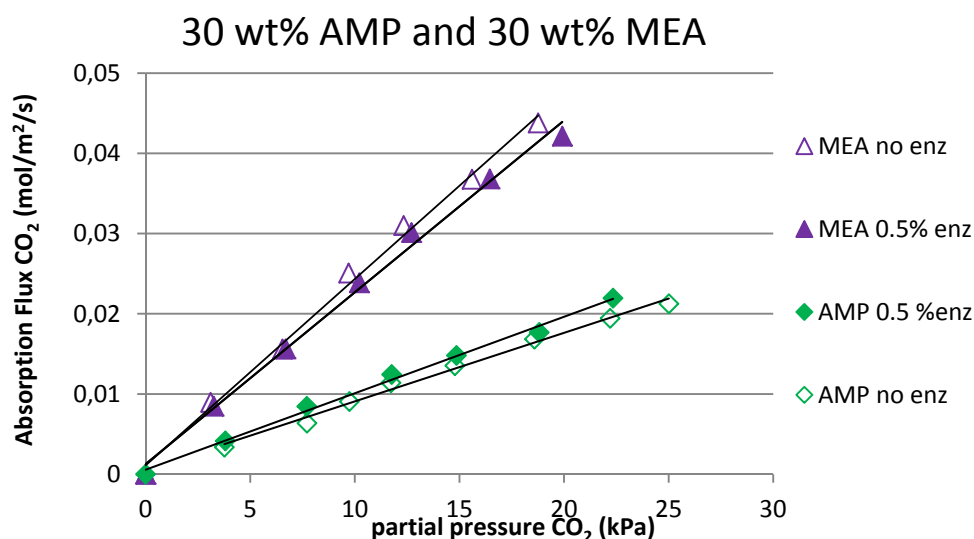


Figure 7: Absorption flux of CO₂ into 30 wt% AMP and 30 wt% MEA at 313 K with 0 and 0.5 (w/w) enzyme solution.

3.1.3. Absorption fluxes relative to 30 wt% MEA

An overview how the different solvent types can absorb CO₂ compared to unloaded 30 wt% MEA can be seen in Figure 8. This figure reveals the fast the absorption rate of MEA. It should be noted, that the absorption rate of MEA decreases when CO₂ is dissolved in the solution, the grey shaded bars represent 30 wt% MEA solution with already dissolved and reacted CO₂ in it, the term loading describes how much mole of CO₂ compared to mole of total amines are in the solution including all reacted CO₂.

In Figure 9 the effect of adding 0.5 % enzyme to the solvent for all different investigated solvents is shown compared to unloaded 30 wt% MEA. The high absorption rate enhancement for MDEA and K₂CO₃ do still not result in absorption rates comparable to 30 wt% MEA.

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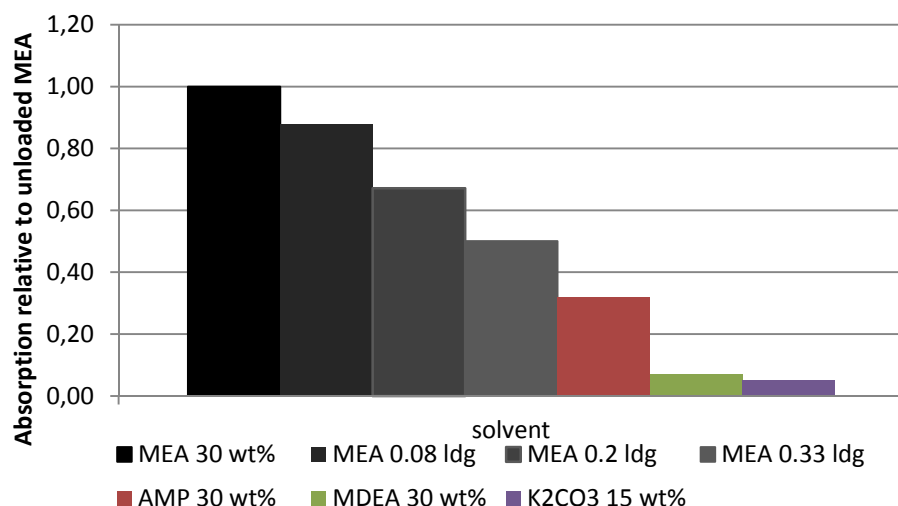


Figure 8: Absorption relative to unloaded MEA 30 wt% without enzyme, the dark grey shade represents the absorption into MEA 30 wt% with different loadings (mol CO₂/mol Amine)

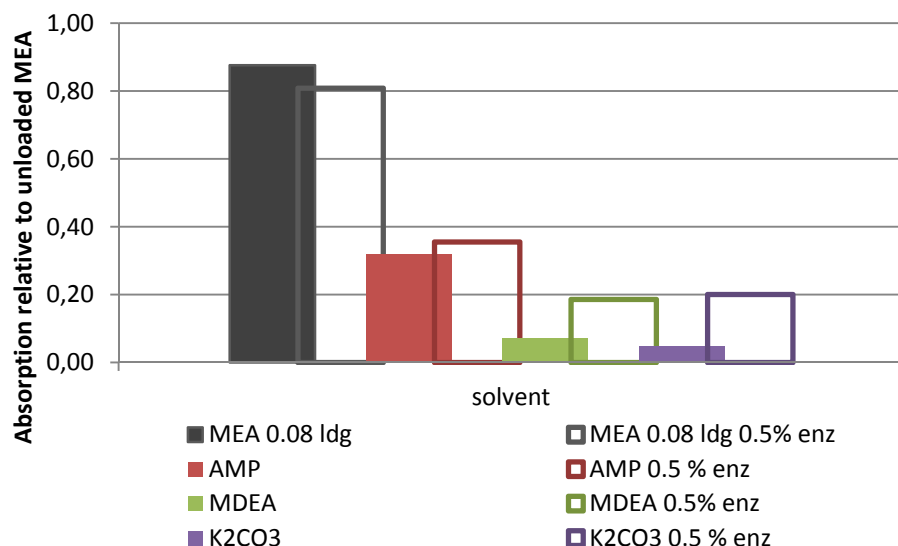


Figure 9: The effect of adding 0.5% enzyme to the solvents

3.1.4. Enzyme kinetics

The enzyme effect and the relative reaction rate constant shown as a function of the enzyme concentration for the 15 wt% K₂CO₃ at 313 K can be seen in Figure 10 and Figure 11. The enzyme effect is the ratio of absorbed flux with enzyme to absorbed flux with just the solvent. The relative overall reaction rate constant is calculated with equation (15). The linear trend in Figure 11 suggests that the relative overall reaction rate is proportional to the enzyme concentration in a range of 0-1 wt%. In order to confirm this theory, more experiments are needed for validation. It may give a hint of what to expect from the enzyme or which enzyme concentration is required at least to meet some absorption rates.



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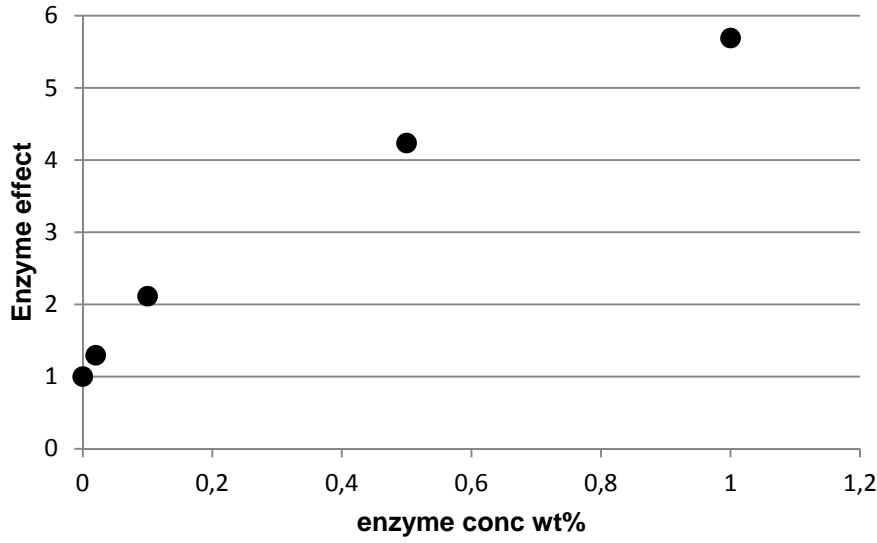


Figure 10: Absorption rate enhancement of 15 wt% K_2CO_3 at 313 K as a function of enzyme concentration.

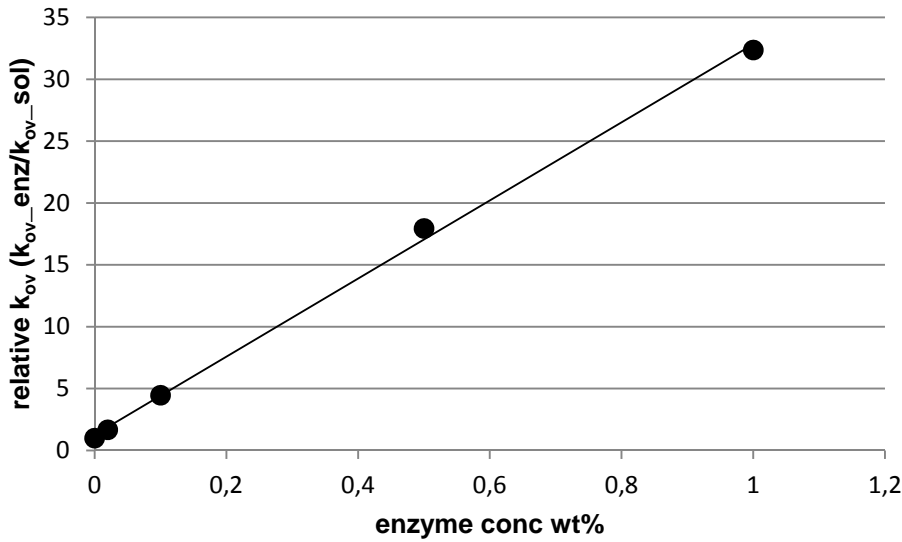


Figure 11: relative overall reaction rate k_{ov} of 15 wt% K_2CO_3 at 313 K as a function of enzyme concentration



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Table 3: Overview of the experiments

type	Solvent		Absorption performance at 313 K				
	name	conc wt%	CO ₂ loading (mol/mol)	enz solution wt%	Flux at 10 kPa CO ₂ mol/m ² /s	relativ to unloadedMEA -	Enzyme effect -
primary amine	MEA	30	0	0	0,0260	1,00	-
		30	0,08	0	0,0228	0,88	-
		30	0,2	0	0,0174	0,67	-
		30	0,33	0	0,0131	0,50	-
		30	0,08	0,5	0,0209	0,81	0,92
		30	0,2	0,5	0,0167	0,64	0,96
tertiary amine	MDEA	30	0	0	0,0018	0,07	-
		30	0	0,5	0,0048	0,19	2,61
sterically hindered amine	AMP	30	0	0	0,0083	0,32	-
		30	0	0,5	0,0092	0,36	1,11
Carbonate salt	K ₂ CO ₃	15	0	0	0,0013	0,05	-
		15	0	0,02	0,0017	0,06	1,30
		15	0	0,1	0,0027	0,10	2,11
		15	0	0,5	0,0054	0,21	4,24
		15	0	1	0,0073	0,28	5,69



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4. Conclusion

When conducting absorption experiments on a wetted wall column under conditions similar to the conventional absorption process in post combustion carbon capture for power plants, the solvent group of tertiary amines and carbonate salt solutions showed very promising results when adding enzymes. These solvents are right now not used for this application even though they can offer energy efficient desorption, because their kinetics are too slow and technical unit operations would need very big dimensions to capture the same amount of CO₂ as the faster solvents. Even though the absorption rate of these solvents could not match the reference solvent 30 wt% MEA they reveal that the application of carbonic anhydrase as an absorption rate enhancer is a promising technology as the absorption rate increased several-fold with the addition of small amounts of enzyme.

