



Enzyme-enhanced CO₂ absorption process in rotating packed bed

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Abstract

Carbon dioxide is considered the most important contributor to the global warming effect. To reduce greenhouse gas emissions, CO₂ should be separated from the exhaust gas stream in a selective way. The most often applied technology to capture CO₂ from exhaust gases is the reactive absorption in aqueous amine solutions, which is currently widely used in different industrial applications. The efficiency of this technology could be improved by applying high-gravity technologies that intensify mass transfer and can enable substantial equipment size reduction compared to the traditionally used packed columns. Rotating packed bed (RPB) technology meets these requirements very well. Applying innovative materials such as the highly efficient enzyme carbonic anhydrase can further improve the efficiency of the CO₂ absorption process. This combination of intensified technology together with new solvents is expected to improve the total efficiency of CO₂ absorption. In this study, we present our experimental results of CO₂ absorption using 30 wt% *N*-methyldiethanolamine (MDEA) solution in water in an RPB unit with and without carbonic anhydrase for different gas and liquid flow rates. The results indicate significantly improved performance of CO₂ absorption, up to 18 times compared to the solvent without enzyme.

Keywords Rotating packed bed · RPB · CO₂ absorption · Carbonic anhydrase · MDEA

Symbols

a	Specific surface area of the packing m ² m ⁻³
A	Cross-section area m ²
$c_{\text{MDEA}}\%$	MDEA concentration in water solution %
F_{factor}	F factor Pa ^{0.5}
M_{MDEA}	Molar mass of MDEA kg kmol ⁻¹
$\dot{n}_{\text{CO}_2,\text{abs}}$	Molar flow rate of total absorbed CO ₂ mol h ⁻¹
$\dot{n}_{\text{CO}_2,\text{abs,packing}}$	Molar flow rate of absorbed CO ₂ inside the packing mol h ⁻¹

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$\dot{n}_{\text{CO}_2,\text{abs,case}}$	Molar flow rate of absorbed CO ₂ in the casing mol h ⁻¹
$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{in}}}$	Molar flow rate of CO ₂ in gas inlet mol h ⁻¹
$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{inside}}}$	Molar flow rate of CO ₂ in the casing mol h ⁻¹
$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{out}}}$	Molar flow rate of CO ₂ in gas outlet mol h ⁻¹
p	Pressure Pa
R	Gas constant J mol ⁻¹ K ⁻¹
T	Temperature K
u_L	Specific liquid load m ³ m ⁻² h ⁻¹
$v_{\text{CO}_2}^{\text{in}}$	Volume fraction of CO ₂ in gas inlet m ³ m ⁻³
$v_{\text{CO}_2}^{\text{inside}}$	Volume fraction of CO ₂ in gas m ³ m ⁻³
$v_{\text{CO}_2}^{\text{out}}$	Volume fraction of CO ₂ in gas outlet m ³ m ⁻³
V_G	Volumetric gas flow m ³ h ⁻¹
V_L	Volumetric liquid flow m ³ h ⁻¹
Y_{CO_2}	CO ₂ loading mol mol ⁻¹

Greek letters

ε	Porosity of the packing m ³ m ⁻³
ρ_G	Density of gas kg m ⁻³
ρ_{MDEA}	Density of MDEA kg m ⁻³

Abbreviations

CA	Carbonic anhydrase
CCU	Carbon capture and utilization
DEA	Diethanolamine
MEA	Monoethanolamine
MDEA	<i>N</i> -Methyldiethanolamine
RPB	Rotating packed bed

Introduction

Carbon capture and utilization (CCU) has become crucial because CO₂ is recognized as the main driver of global warming. The main part of CO₂ is emitted in fossil fuel combustion (Friedlingstein and Prentice 2010). To reduce CO₂ emission into the atmosphere, it is necessary to develop more efficient and effective carbon capture technology. The most suitable for industrial application is currently the post-combustion capture based on a reactive absorption process with amine solvent, which is also the state-of-the-art technology (Yang et al. 2008). To improve reactive absorption efficiency, better process design and improved solvents are needed.

A variety of amine solvents have been used extensively for CO₂ absorption processes for years (Yildirim et al. 2012). Tertiary amines are the most promising since they have higher maximum CO₂ loading and lower regeneration energy in comparison to primary and secondary amines (El Hadri 2017). However, tertiary amines have very low reaction rates, so a long residence time in the apparatus is required (Bougie and Iliuta 2012; Quang et al. 2013; El Hadri 2017). To improve the reaction rates, it is possible to use innovative materials such as different types of activators or, more recently, enzymes. Carbonic anhydrase is considered the fastest and most selective biocatalyst known that performs hydration of CO₂ (Collett et al. 2011; Kunze et al. 2015).

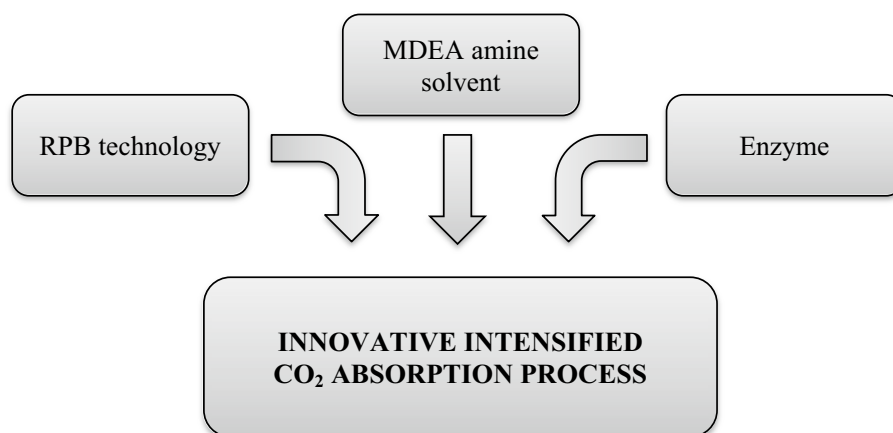
CO₂ absorption process can be enhanced, not only using better solvents, but also by intensified processes. Rotating packed bed (RPB) technology exploits a high centrifugal force to increase the surface renewal of phases in the packing and fosters the use of highly specific area packing, so that the combination significantly increases overall mass transfer. The RPB is operated in the countercurrent regime, in which the liquid is distributed into the eye of the rotor flows towards the outer rotor diameter. Overcoming the gravitational force, which is the limiting parameter in packed columns, allows for a significant volume reduction of the RPB unit to significant reduction of magnitude for the same gas and liquid throughputs (Rao et al. 2004) (Fig. 1).

Whereas RPB technology has been applied to CO₂ absorption, few studies have investigated the combination of RPB with an enzymatic-catalyzed solvent system. Leimbrink et al. reported the possible advantages of a reactive CO₂ absorption process with the use of the efficient enzyme carbonic anhydrase in different contactors, showing that an RPB allows for improved performance compared with a packed column (Leimbrink et al. 2017b). Such a combination of technology, materials and packing is expected to reduce total operating costs of the investigated process (Penders-van Elk et al. 2012; Gladis et al. 2017; Neumann et al. 2018). The current study builds on the results of Leimbrink et al. (2017a), extends the investigations beyond the common operating window of the packed bed absorber and the RPB and further investigates the applicability of printed packing structures for the considered solvent system, based on a recently introduced additive manufacturing approach (Gładyszewski and Skiborowski 2018).

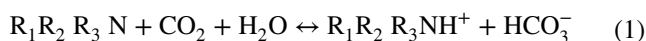
Theoretical

The tertiary *N*-methyldiethanolamine (MDEA) has great potential among the amines used as solvents in CO₂ reactive absorption processes thanks to its high absorption

Fig. 1 Schematic presentation of the work objectives



capacity, low heat of reaction and low regeneration energy. In addition, MDEA is less corrosive and less volatile and has decomposition products with lower toxicity, especially in comparison to primary and secondary amines such as monoethanolamine (MEA) and diethanolamine (DEA) (Bougie and Iliuta 2012; Quang et al. 2013; Afkhamipour and Mofarahi 2014; de Ávila et al. 2015; El Hadri 2017). Low regeneration energy is the essential factor for the implementation of carbon capture processes, as 70–80% of the energy of the absorption–desorption process can be consumed by the regeneration process (Nwaoha et al. 2016). What should also be noticed is that the tertiary amines react with CO₂ only in the presence of water as a result of reaction (1), in which carbonate product is formed (El Hadri 2017). Figure 2 presents the scheme of absorption and desorption processes with enzymatically catalyzed amine solvent. The water content is crucial here, as most enzymes can only work efficiently in aqueous solutions:



Acting as a catalyst, compared to activators such as piperazine, carbonic anhydrase has no influence on the physical or chemical properties of the amine solvent (Penders-van Elk et al. 2016), but it significantly enhances the reaction rate. The blended solution of CA-MDEA solvents provides a significant potential to decrease the energy consumption in reactive CO₂ absorption (Leimbrink et al. 2017a). The dominant mechanism of CO₂ hydration by CA depends on the solution pH. Two reaction mechanisms can be found in the literature. For pH < 8, reaction (2) is dominant. This is a two-step reaction whose first step is hydration of CO₂ and whose second step is rapid dissociation of bicarbonate ion. For pH > 10, reaction (3) is dominant. A bicarbonate ion is formed from CO₂ and hydroxyl ion (Kunze et al. 2015). Furthermore, reactions (1), (2), and (3) can occur simultaneously (Penders-van Elk et al. 2016).



The complete reaction mechanism of carbonic anhydrase with CO₂ is a closed cycle, which is presented in Fig. 3

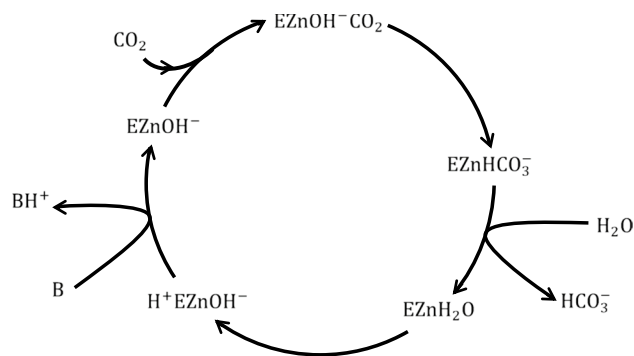


Fig. 3 Wheel of carbonic anhydrase reaction (Penders-van Elk et al. 2016). *B* base, *Zn* zinc atom in active site of CA, *E* CA enzyme

(Penders-van Elk et al. 2016). The “B” represents the base that is used for enzyme regeneration, e.g., hydroxide ion, carbonate ion, bicarbonate ion or alkanolamine (Penders-van Elk et al. 2016).

Experimental

Materials

The solvent used in this study was a tertiary amine [*N*-methyldiethanolamine (MDEA)] solution, provided by BASF (purity ≥ 99%), diluted with deionized water to concentration of 30 wt% of MDEA. The carbonic anhydrase (CA) enzyme (EC 4.2.1.1) used in this study was provided in dissolved form. CA was produced as an extracellular protein of microbial origin and was supplied by Novozymes A/S (Bagsvaerd, Denmark). In all experiments with enzyme, the concentration of 0.2 wt% CA was kept constant.

In this study, a foam-like packing was manufactured by means of rapid prototyping. The additive manufacturing enables the fast and low-cost production of packing prototypes and allows for the development of tailor-made packings for the use in RPBs. For this purpose, Titan 2, a 3D printer from Kudo 3D Inc., was used to produce a packing from the photosensitive resin Spot-HT. The packing morphology was a replica of metal foam NCX1116 produced

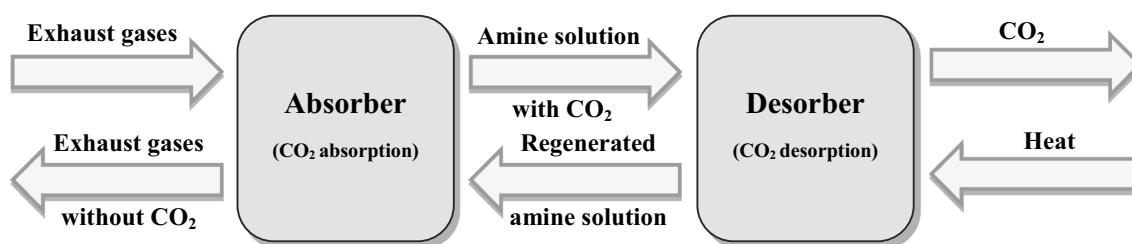


Fig. 2 Scheme of absorption and desorption processes with amine solvent (Source: Wojtasik 2017)

by RECEMAT B.V. Netherlands. The general idea of additive manufacturing for rapid prototyping of RPB packings was recently introduced by Gladyszewski and Skiborowski (2018). The structure of the used packing is presented in Fig. 4. The most important packing parameters are average pore diameter (here, 1.4 mm), average strut side length (0.27 mm), specific surface area ($a = 1136 \text{ m}^2 \text{ m}^{-3}$) and porosity ($\epsilon = 0.912$) (Gladyszewski and Skiborowski 2018).

While polymeric packings are not considered viable for a long-term application, the current study also investigated and validated the chemical and mechanical stability of the printed packing within the conducted experimental investigations with the considered chemical solvent system.

The experimental investigations were conducted for a 30 wt% aqueous MDEA solution without enzyme and with 0.2 wt% CA enzyme. For each run, the initial pH of the solution was between 10.0 and 10.3. Maintenance of pH range is important to avoid negative effects on enzyme stability (Gundersen et al. 2014).

Experimental setup

In this study, a single-stage RPB unit was investigated. A rotor with an outer diameter of 400 mm, an axial height of 10 mm and an inner packing diameter of 146 mm was used. To avoid bypass of gas, a hydrodynamic seal was used between the upper plate of the rotor and the casing of the RPB. As a barrier fluid, deionized water was used. The shaft was sealed by a mechanical seal with propylene glycol as a barrier liquid. Three nine-hole nozzles were used, which allowed us to distribute the liquid onto the inner periphery of the packing as a full jet, before entering the liquid into the packing. The holes in the nozzle had a diameter of 0.9 mm, and the angle between them in the spraying plane was 90° .

Experiments in this study were conducted according to the operating conditions specified in Table 1.

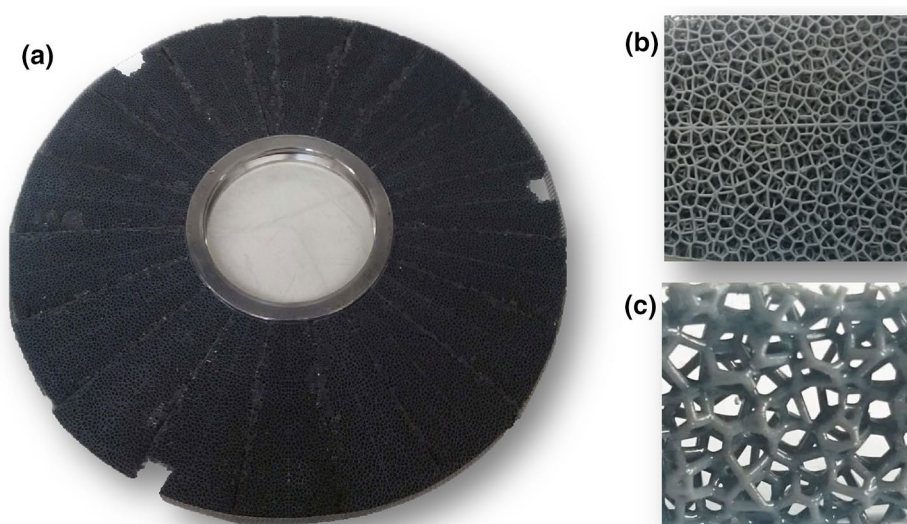
Experimental methodology

Figure 5 depicts the experimental device for reactive absorption of CO_2 . In the gas stream, air and CO_2 were mixed in the static mixer and humidified (8) before entering the RPB unit. The gas stream provided to RPB contained 15 vol.% CO_2 . During operation, the gas mixture was fed through a gas inlet (10) and flew to the outer diameter of rotor (5). The solvent was introduced from the liquid storage tank (1) by a gear pump (2) to the top of the RPB unit and then to the center of the rotor. The liquid was distributed by 3 stationary nine-hole nozzles (4) onto the inner periphery of packing. The solvent moved to the outside of the packing due to the centrifugal force. The temperature of the solvent was constant at 20°C for all experiments because this showed favorable properties

Table 1 Experimentally investigated operating points

Parameter	Values
Gas flow rate (m^3/h)	20
	40
	60
Liquid flow rate (m^3/h)	0.18
	0.3
Rotational speed (rpm)	600
	1200
Presence of carbonic anhydrase	None
	0.2 wt% in solution

Fig. 4 **a** Assembled printed packing. The three visible cavities at the outer rim of the packing were implemented on purpose, since they were required to fit the necessary rotors spacers; **b**, **c** packing structure



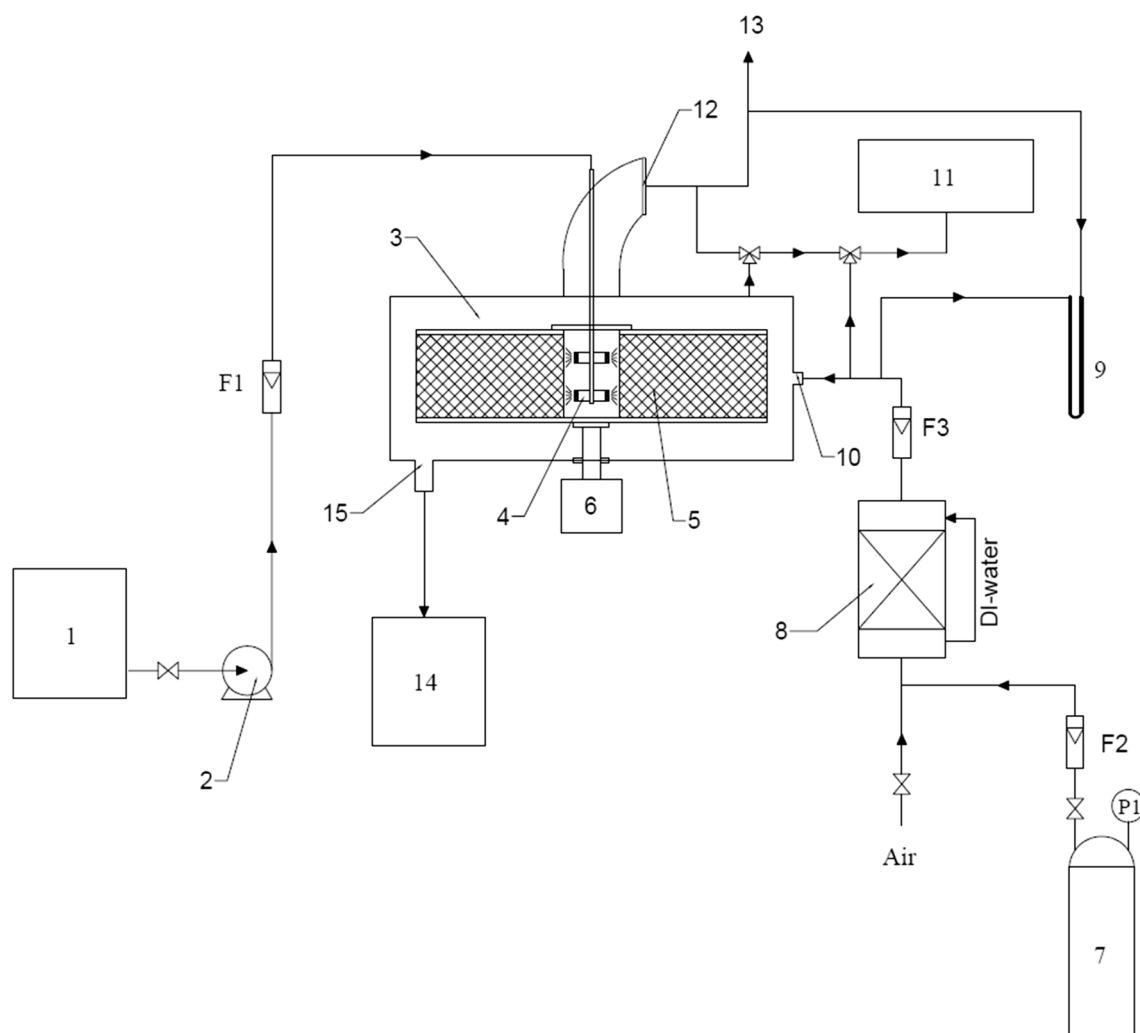


Fig. 5 Experimental setup of RPB equipped with printed packing for CO₂ absorption process. 1-liquid storage tank, 2-gear pump, 3-RPB unit, 4-nozzles, 5-packing, 6-motor, 7-CO₂ cylinder, 8-humidifier,

9-U-tube manometer, 10-gas inlet, 11-CO₂ analyzer, 12-gas outlet, 13-inlet to ventilation system, 14-waste storage tank, 15-liquid outlet, F1-liquid flowmeter, F2, F3-gas flowmeter (Source: Wojtasik 2017)

in previous investigations in packed beds (Leimbrink and Tlatlik 2017c). Solvent with absorbed CO₂ was collected at the walls and at the bottom of casing and then flowed out by 4 liquid outlets (15) to the waste storage tank (14). Outlet gas steam was led away by a ventilation system (12).

CO₂ concentration was measured by a Guardian NG infrared gas monitor (detection limit of 30% CO₂) (11) in 3 sampling points: the gas inlet, inside the RPB casing and at the gas outlet. The value was recorded as a result when the analyzer presented a constant value for 5 s. Furthermore, the pressure drop was measured with a U-tube manometer connected with the RPB unit at the inlet and outlet of the gas stream. Waste liquid collected in the tank (14) after the process was regenerated at 50 °C till the time when the pH was acceptable and then was reused to conduct next experiments.

Evaluation of experimental results

The total amount of absorbed CO₂ was calculated according to Eq. 4:

$$\dot{n}_{\text{CO}_2, \text{abs}} = \dot{n}_{\text{CO}_2}^{\text{G}_{\text{in}}} - \dot{n}_{\text{CO}_2}^{\text{G}_{\text{out}}} \quad (4)$$

Here, the amount of absorbed CO₂ is the molar difference between CO₂ in gas inlet ($\dot{n}_{\text{CO}_2}^{\text{G}_{\text{in}}}$) and outlets ($\dot{n}_{\text{CO}_2}^{\text{G}_{\text{out}}}$). To calculate the molar absorption of CO₂, the ideal gas law was applied:

$$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{in}}} = \frac{V_{\text{G}} \cdot v_{\text{CO}_2}^{\text{in}} \cdot p}{R \cdot T} \quad (5)$$

$$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{inside}}} = \frac{V_{\text{G}} \cdot v_{\text{CO}_2}^{\text{inside}} \cdot p}{R \cdot T} \quad (6)$$

$$\dot{n}_{\text{CO}_2}^{\text{G}_{\text{out}}} = \frac{V_G \cdot v_{\text{CO}_2}^{\text{out}} \cdot p}{R \cdot T} \quad (7)$$

Molar flow rates of CO₂ at the gas inlet (Eq. 5) inside the RPB (Eq. 6) and at the outlet (Eq. 7) can be calculated for a known pressure (p), temperature (T) and total gas flow rate (V_G), where R is the ideal gas constant and volume fraction of CO₂ at the inlet ($v_{\text{CO}_2}^{\text{in}}$), inside the casing ($v_{\text{CO}_2}^{\text{inside}}$) and at the outlet ($v_{\text{CO}_2}^{\text{out}}$). Due to the absorption process, the volume fraction of CO₂ in the gas stream decreases as the gas flows through the RPB unit.

Thanks to the location of each measured point close to the rotor's outer periphery, it was possible to calculate separately the molar absorption of CO₂ inside the packing (Eq. 8) and inside the casing (Eq. 9):

$$\dot{n}_{\text{CO}_2, \text{abs, packing}} = \dot{n}_{\text{CO}_2}^{\text{G}_{\text{in}}} - \dot{n}_{\text{CO}_2}^{\text{G}_{\text{inside}}} \quad (8)$$

$$\dot{n}_{\text{CO}_2, \text{abs, case}} = \dot{n}_{\text{CO}_2}^{\text{G}_{\text{inside}}} - \dot{n}_{\text{CO}_2}^{\text{G}_{\text{out}}} \quad (9)$$

For quantification of the efficiency of the absorption, another important piece of information is the CO₂ loading Y_{CO_2} , which was calculated by Eq. 10:

$$Y_{\text{CO}_2} = \frac{\dot{n}_{\text{CO}_2, \text{abs}}}{V_L \times c_{\% \text{MDEA}} \times \rho_{\text{MDEA}} \times M_{\text{MDEA}}} \quad (10)$$

Here, V_L is the liquid flow rate, $c_{\% \text{MDEA}}$ is the MDEA wt% concentration in aqueous solution, ρ_{MDEA} is the MDEA density and M_{MDEA} is the molar mass of MDEA.

To characterize the operating window and enable the comparison of obtained results with other equipment, the F factor (Eq. 11) was calculated:

$$F_{\text{factor}} = \frac{V_G}{A} \sqrt{\rho_G} \quad (11)$$

Here, A is the cross-sectional area and ρ_G is the gas mixture density.

Another commonly used parameter is specific liquid load u_L , which is defined by Eq. 12:

$$u_L = \frac{V_L}{A} \quad (12)$$

According to the literature, recommendations for RPB units (Rao et al. 2004), the specific values for the F factor and the specific liquid load are reported with respect to the inner radius of the packing. This is the radius of the area between the center (axis) of the rotor and inner radius of the packing, which is called the eye of the rotor.

Results and discussion

Each set of experiments conducted in this study was performed 3 times. The initial temperature of the solvent was 20 °C. For the chosen gas and liquid flow rates, F factors and specific liquid load are presented in Table 2.

F factors and specific liquid load allowed us to compare the obtained CO₂ absorption rate results in the RPB with available data for packed columns. Leimbrink et al. already performed a comparison between the current RPB and a lab-scale packed column in a common operating window, which was limited in terms of the F factor to a range from 0.5 Pa^{0.5} to 1.5 Pa^{0.5} (Leimbrink et al. 2017b). While the current study also intersects this operating window (F factor 1.32 Pa^{0.5}), it specifically evaluates the range of higher gas capacities, which are only accessible with the RPB.

The maximum CO₂ absorption observed for a liquid flow rate of 0.18 m³ h⁻¹ was approximately 25 mol h⁻¹ for F factor 3.96 Pa^{0.5} and both rotational speeds. As expected from the higher driving force, the amount of CO₂ absorption also increased with increasing gas flow rate. It also should be noticed that more CO₂ was injected into the equipment with increased gas load. Doubling the rotational speed caused only a slight increase in CO₂ absorption rate, as the process was mostly limited by reaction in liquid phase. A similar situation was observed for experiments with a liquid flow rate 0.3 m³ h⁻¹. However, the maximum amount of absorbed CO₂ was higher by approximately 30 mol h⁻¹ for the gas flow rate of 60 m³ h⁻¹.

Previous results indicate the meaningful influence of the casing on the overall mass transfer process (Chen et al. 2005). Because we measured the CO₂ concentration in 3 points, it was possible to calculate the contributions of packing and casing to the overall absorption rate. The main part of CO₂ absorption occurred inside the casing and contributed up to 80% with 30 wt% MDEA and up to 60% with 30 wt% MDEA + 0.2 wt% CA. These results indicate that the resident time inside the packing was still too short. The detailed data are presented in Appendix A.

Evaluating the obtained results of CO₂ absorption for both investigated solvents in all configurations, we concluded that a diminutive addition of carbonic anhydrase

Table 2 Calculated values of F factors and specific liquid load

Parameter	Values (m ³ h ⁻¹)	F factor (Pa ^{0.5})	u_L (m ³ m ⁻² h ⁻¹)
Gas flow rate	20	1.32	—
	40	2.64	—
	60	3.96	—
Liquid flow rate	0.18	—	39.24
	0.3	—	65.41

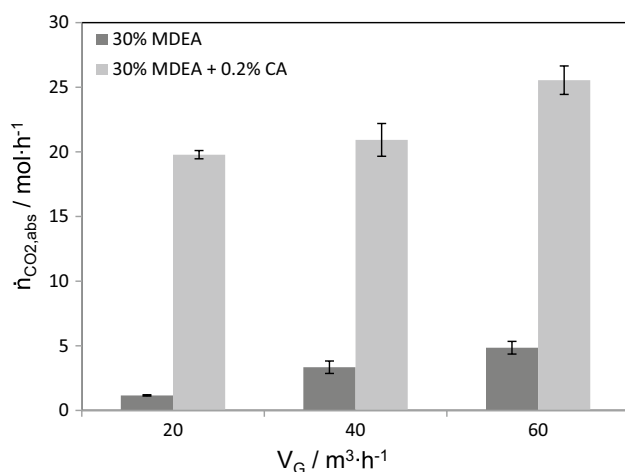


Fig. 6 Effect of gas flow rate on CO_2 absorption rate for liquid flow rate $0.18 \text{ m}^3 \text{ h}^{-1}$ and rotational speed 600 rpm

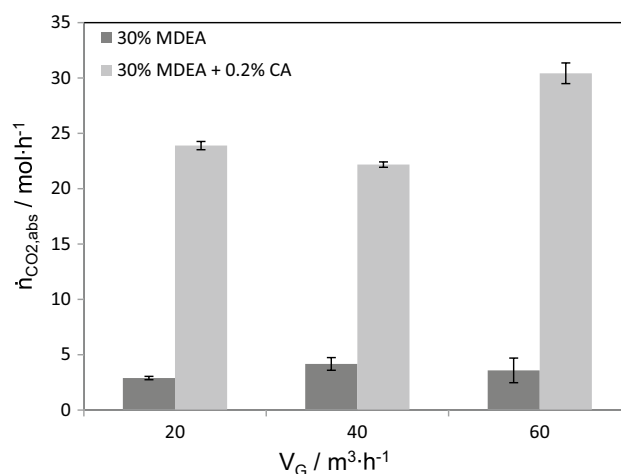


Fig. 8 Effect of gas flow rate on CO_2 absorption rate for liquid flow rate $0.3 \text{ m}^3 \text{ h}^{-1}$ and rotational speed 600 rpm

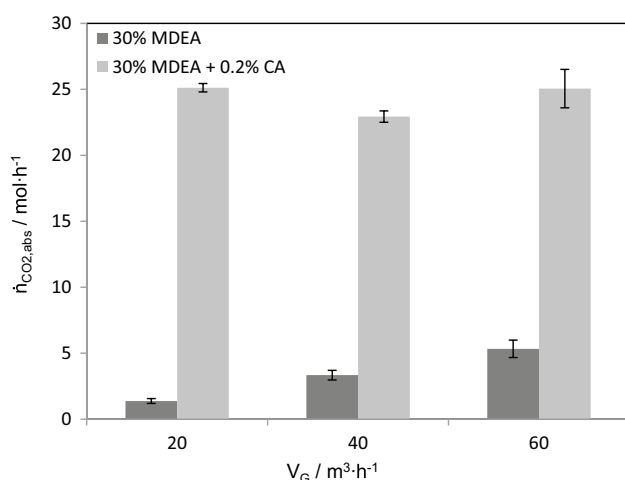


Fig. 7 Effect of gas flow rate on CO_2 absorption rate for liquid flow rate $0.18 \text{ m}^3 \text{ h}^{-1}$ and rotational speed 1200 rpm

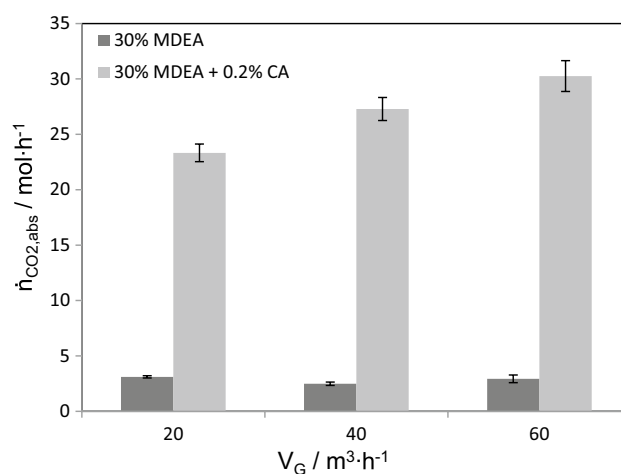


Fig. 9 Effect of gas flow rate on CO_2 absorption rate for liquid flow rate $0.3 \text{ m}^3 \text{ h}^{-1}$ and rotational speed 1200 rpm

(0.2 wt% CA) caused a significant increase in the total rate of CO_2 absorption. This result indicates an increase in CO_2 absorption of 5 to 18 times compared to the solvent without enzyme. Values measured by Penders-van Elk et al. (2012), Kunze et al. (2015), Gladis et al. (2017), Leimbrink et al. (2017c) were in the range of less than a factor of 10. The observed high increase of CO_2 absorption rate, up to 18 times the nominal value without CA, may be the result of the significantly higher F factor compared to the packed column and the increased surface area of the packing. Moreover, the impact of centrifugal force can be important (Sudhoff et al. 2014, Neumann et al. 2018).

The highest increase in CO_2 absorption rate for liquid flow rate $0.18 \text{ m}^3 \text{ h}^{-1}$ was recorded with gas flow rate $20 \text{ m}^3 \text{ h}^{-1}$ and both rotational speeds (Figs. 6, 7), whereas for

liquid flow rate $0.3 \text{ m}^3 \text{ h}^{-1}$, the highest growth was recorded with gas flow rate $60 \text{ m}^3 \text{ h}^{-1}$ and both rotational speeds (Figs. 8, 9). The total increase in CO_2 absorption rate was in the range 17.6 to 27.3 mol h^{-1} .

The literature reports that the value of maximum CO_2 loading for 30 wt% MDEA solution at 20°C is in the range $0.4\text{--}0.67 \text{ mol CO}_2 \text{ mol}^{-1} \text{ MDEA}$. In this study, we assumed that the maximum value was $0.5 \text{ mol CO}_2 \text{ mol}^{-1} \text{ MDEA}$ (Shen and Li 1992; El Hadri 2017). Addition of carbonic anhydrase as a catalyst should not influence the physical or chemical properties of the amine solvent (Penders-van Elk et al. 2016).

According to Table 3, the rich CO_2 loading obtained in this study was in the range of 10% of the maximum CO_2 capacity for 30 wt% MDEA solution. Hence, there was

Table 3 CO₂ loading (Y_{CO_2}) for 30 wt% MDEA + 0.2 wt% CA solvent

Liquid flow rate (m ³ h ⁻¹)	Rotational speed (rpm)	Gas flow rate (m ³ h ⁻¹)	CO ₂ loading mole CO ₂ mol ⁻¹ MDEA	Part of maximum CO ₂ loading (%)
0.18	600	20	0.0420	8.40
		40	0.0444	8.88
		60	0.0542	10.84
	1200	20	0.0533	10.66
		40	0.0487	9.73
		60	0.0532	10.63
0.3	600	20	0.0304	6.08
		40	0.0282	5.65
		60	0.0387	7.75
	1200	20	0.0297	5.94
		40	0.0347	6.95
		60	0.0385	7.70

potential to significantly improve the performance of CO₂ absorption in the RPB unit.

Conclusions

While a 30 wt% MDEA solution is unsuitable as a solvent for efficient CO₂ absorption in an RPB unit, the addition of carbonic anhydrase significantly improves the performance of CO₂ absorption. Due to the relatively low loading of CO₂ in 30 wt% MDEA + 0.2% CA solution, the total CO₂ absorption rate can be improved by an increase in the specific surface area or extension of the residence time of the solution inside the RPB unit.

Future work should extend the investigations of enzymatic absorption of CO₂ in RPB units. Different RPB setups and process parameters should be investigated. Additionally, a different approach of enzyme delivery into the RPB, such as enzyme immobilized in the form of small solid particles, should be considered. To use enzyme immobilized in the form of solid particles, a new type of packing needs to be designed and fabricated.

Our results also indicate that the current design and operating point are rather suboptimal in terms of utilizing the equipment.

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