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A New Absorption-Desorption Pilot Plant for CO_2 Capture

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A recently built multi-purpose pilot plant for the investigations of (reactive) absorption processes is presented. It consists of two glass columns with an inner diameter of 0.1 m and 0.3 m. The plant can be used for fluid dynamic studies of packings and other internals as well as for absorption and desorption experiments. Above all, CO_2 capturing by aqueous amine solutions should be examined. Validation experiments on fluid dynamics showed that the bigger column can be used for pressure drop measurements of dry and irrigated packings. Furthermore, the results of first absorption and desorption runs performed with the widely used solvent monoethanolamine give evidence that the plant is applicable for thorough experimentation.

1. Introduction

Amine scrubbing appears to be the most suitable technology for capturing carbon dioxide (CO_2). It will probably be the dominating technology for CO_2 capture from coal-fired power plants in 2030 (Rochelle 2009). Usually, the scrubbing process is carried out in a closed loop consisting of an absorption and a desorption column. In particular, the regeneration of the solvent in the desorption column requires large amounts of energy. In industrial applications, mostly the state-of-the-art solvent monoethanolamine (MEA) is applied, which leads to a loss of around 11% of power plant efficiency (Neveux et al. 2013). On the other hand, several amine-based solvents (e.g. amine blends, activated amines, sterically hindered amines) are reported to be superior to MEA regarding reduced energy consumption for the desorption step.
However, up to now, only few of these solvents have been tested in an entire absorption-desorption loop, e.g., 2-amino-2-methyl-1-propanol (AMP) blended with piperazine (PZ) by Artanto et al. (2014) and blended with 1,2-ethandiamine (EDA) by von Harbou et al. (2013). Our group has been actively working in this area in order to close this gap. Newly proposed promising solvents from the literature will be tested in the recently built absorption-desorption pilot plant. This paper describes the characteristics and functionality of this plant and presents results of the first validation experiments carried out with MEA.

2. Set-up

The pilot plant (Figure 1) is designed as a multi-purpose set-up. Experiments can be carried out in different operation modes, namely, in the absorption, desorption and in the closed loop mode. Furthermore, fluid dynamic studies can be performed. In order to enable these operation modes, the plant consists of two glass columns, one with an inner diameter of 0.1 m and another with 0.3 m. Both columns are about 5 m high including a packed section of about 3 m. The smaller column is predominantly used for absorption; due to the small diameter and consequently considerable wall effects, this column is less suitable for fluid dynamic experiments. On the contrary, the bigger column can be used either for desorption or for fluid dynamic studies. In the closed loop mode, both columns are to be coupled.

2.1 Detailed description

The simplified process flow sheet of the absorption/desorption process is shown in Figure 2. Fresh air is fed into a pre-washer column by a blower, to ensure that the air is saturated with water. The gas flow rate can be set between 20 and 100 m³/h resulting in F-factors varying between 1 and 4 $Pa^{0.5}$. Before entering the absorptions column, a CO_2 stream is injected into the air stream by a mass flow controller. The maximum CO_2 mass flow is 20 m³/m²h, enabling a wide range of CO_2 partial pressures.

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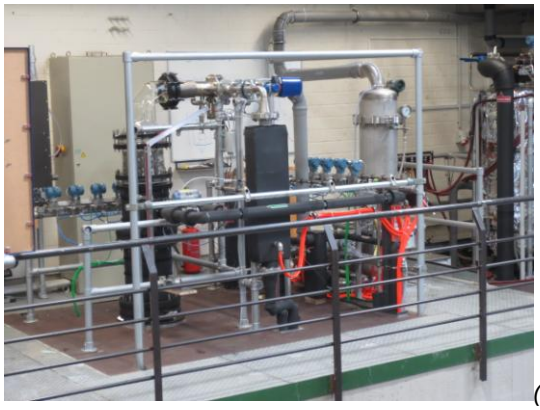
(a)



(b)

Figure 1: Photos of the pilot plant: middle part (a); upper part (b)

The absorber is built of several glass sections equipped with Montz B1.250 (Julius Montz GmbH) structured packing, one liquid-phase distributor at the top and one liquid-phase redistributor in the middle. The packing height is 1,470 mm below the redistributor and 1,372 mm above it (overall packing height: 2,842 mm).

The lean amine solution flow rate can be set between 100 and 500 kg/h resulting in liquid loads between 10 and 60 m³/m²h. For steady-state operation, the liquid level in the absorber bottom is controlled by a valve keeping the differential pressure constant. The CO_2-free air leaving the absorber is directed to a washing column to avoid amine emissions to the environment.

The rich amine solution is pumped into the desorber through the lean-rich heat exchanger. The desorber has the same design as the absorber except for the larger diameter and the height of the upper packed section (1,470 mm; overall packing height: 2,940 mm). At the liquid-phase outlet of the desorber, a natural recirculation evaporator is installed for partial evaporation of the solvent. The CO_2 stream leaving the desorber can be directed to the washing column and released to the atmosphere or sucked out of the column for the reuse in the absorber (the latter option is not shown in the flow sheet).

As described above, the desorber is also used for fluid dynamic studies. Fresh air is fed into the column by another blower. The flow rate can be set between 200 and 1,000 m³/h resulting in F-factors between 1 and 3.5 $Pa^{0.5}$.

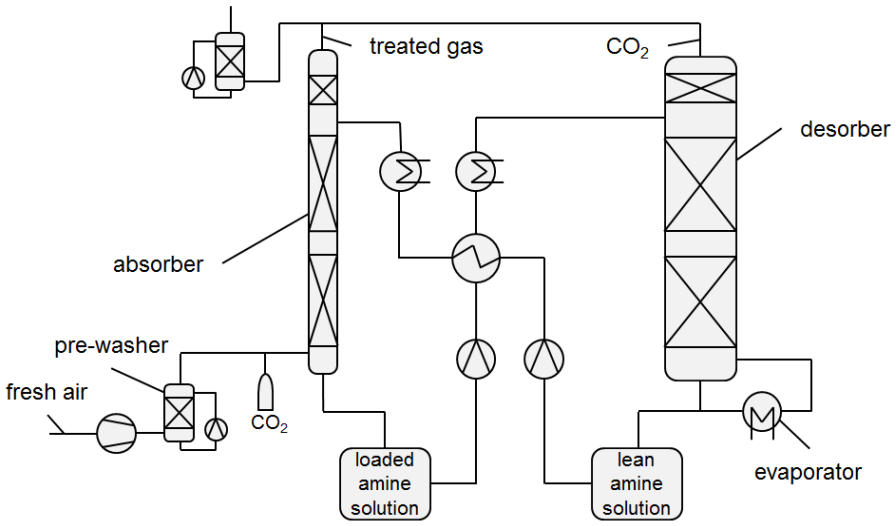


Figure 2: Simplified flow sheet of the closed-loop process

2.2 Sampling and analytics

The two pilot plant columns are designed to measure axial temperature and concentration profiles. Both gas-phase and liquid-phase samples are taken with the aid of specially developed sampling flanges (Figure 3) placed at 0.5 m intervals along the packing height. These flanges are installed between two glass sections, each flange has two bore holes to insert devices for liquid and gas sample taking. The liquid-phase device works as follows: The liquid trickles into the upper tube at the tip of the device, flows through the chamber and trickles out of the lower tube; thus, liquid holdup in the chamber is always renewed. With a syringe, the liquid can be taken from the chamber. The liquid samples are collected and analysed off-line.

For gas-phase sampling, a tube is inserted into the flange. This tube is connected to the gas chromatograph via a system of heated pipes and a vacuum pump, so that the gas samples can be analysed online. A Pt100 resistance thermometer is inserted into the tube to measure the temperature at each sampling point. For measuring the pressure drop along the columns, the devices at the top and bottom are also equipped with a small tube connected to pressure drop transmitters.

The analysis of both gas and liquid samples is performed with a gas chromatograph integrated into the plant. The chromatograph is equipped with a system of three columns and two detectors. To analyse the gas phase, a Molsieve and a Porapak Q column will be used in combination with a thermal conductivity detector (TCD). The liquid-phase samples are vaporized in the chromatograph and run over a special column for amine solutions (COL-ELITE-5-AMINE 30 m X 0.25 mm X 0.50 µm). In order to make a qualitative and quantitative analysis of the amine, a mass spectrometer is used as detector.

3. Experimental Results

First fluid dynamic, absorption and desorption experiments were carried out. The results were compared to the literature data for the sake of the pilot plant validation.

3.1 Fluid dynamic experiments

Fluid dynamic experiments were performed to measure the pressure drop of the dry and irrigated packed column and hence to validate the measuring system. The obtained results were compared to the data measured at the packing manufacturer Julius Montz GmbH (column diameter: 600 mm) as well as to the values published by Olujic (1999) (column diameter: 200, 450 and 800 mm).

First, the dry pressure drop was measured in both columns. The F-factors were varied between 1 and 4 $Pa^{0,5}$. As can be seen from Figure 4a, our measurements made at the bigger column (diameter of 300 mm) agree well with the experimental values of Montz. The pressure drop per meter measured at the small column is higher; this can be explained by a more significant wall effect.

Due to the pronounced wall effect in the small column, the pressure drop of the irrigated packing was measured in the bigger column only. Experiments were carried out at liquid loads of 10, 20 and 50 $m^3/m^2/h$ and at F-factors varying between 1 and 4 $Pa^{0,5}$. For low liquid loads, our measurements showed good agreement with the values of Montz (see Figure 4b). The slight deviation can be explained by clearly smaller diameter of our column compared to the column at Montz. Additionally, in Figure 5, we compared the results obtained at a liquid load of 10m^3/m^2h with the values measured by Olujic (1999). Good agreement is achieved with the data for the column with 450 mm diameter. A higher deviation in Figure 4b at a liquid load of 50 m^3/m^2h is most likely due to water accumulation in the pressure measuring system

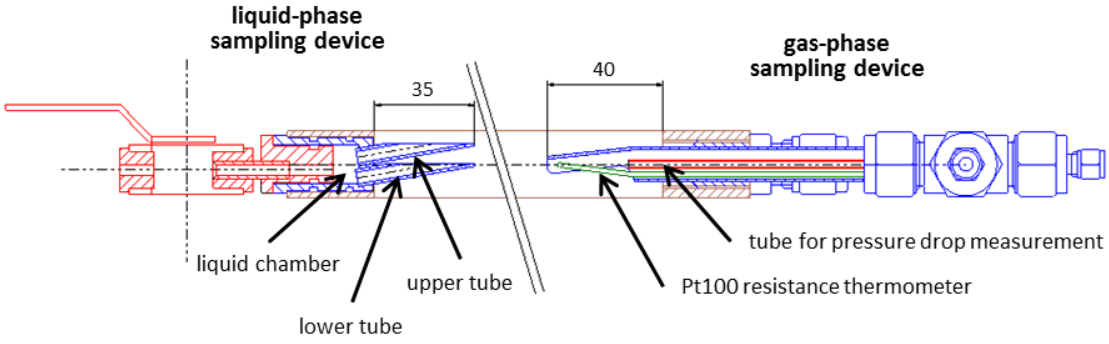


Figure 3: Drawing of the flange including the liquid-phase sampling device (left) and gas-phase sampling device (right)

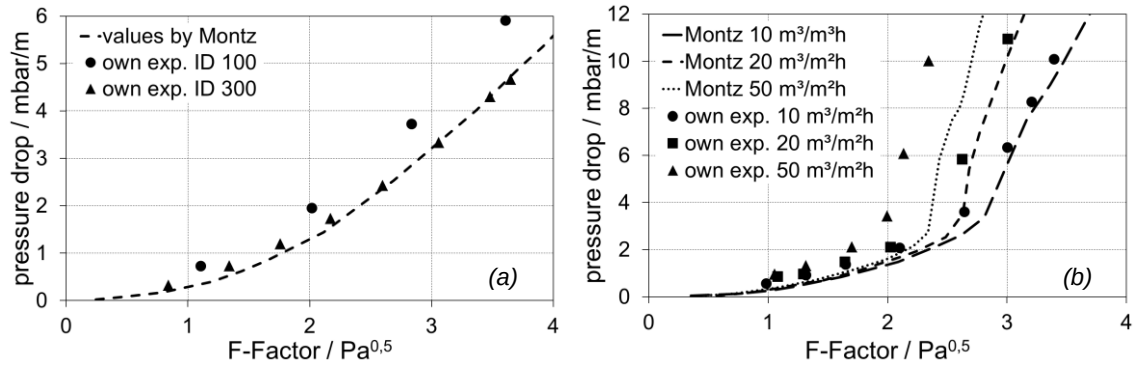


Figure 4: Results of the pressure drop measurement compared to values by Julius Montz GmbH: Dry pressure drop at both columns (a); pressure drop of irrigated packing at the thicker column (b)

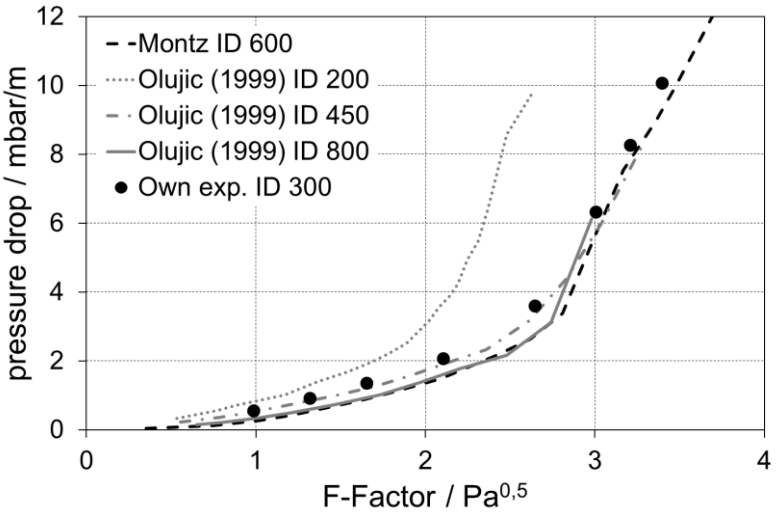


Figure 5: Comparison of our measured pressure drop of irrigated packing at a liquid load of 10 m³/m²h with the data of Montz and of Olujic (1999)

resulting in measurement errors. Anyway, as the deviation at 10 and 20 m³/m²h is relative low, our column proves to be applicable for studying fluid dynamics of different packings as well as other internals.

3.2 Absorption experiments

First absorption measurements were performed with the commonly used solvent monoethanolamine (MEA). Three experiments (summarized in Table 1) were carried out, with liquid loads of 17 m³/m²h and F-factors of about 1.6 $Pa^{0.5}$. The solvent was a 14 wt% aqueous MEA solution. The inlet CO_2 concentration was varied between 0.045 and 0.091.

The start-up of the experiments was as follows: The solvent and the fresh air were fed to the absorber and then the CO_2 injection started. As soon as absorption began, the temperatures in the column started

Table 1: Process conditions of experiments A1 to A3.

	$\dot{m}_{solvent,in}$	$T_{solvent,in}$	$w_{MEA,in}$	$\dot{V}_{Gas,in}$	$T_{gas,in}$	$y_{CO_2,in}$
	kg/h	°C	g/g	m³/h	°C	mol/mol
A1	130.0	22.8	0.14	42.2	22.8	0.045
A2	130.0	22.3	0.14	43.6	22.8	0.091
A3	130.0	21.5	0.14	43.0	22.6	0.070

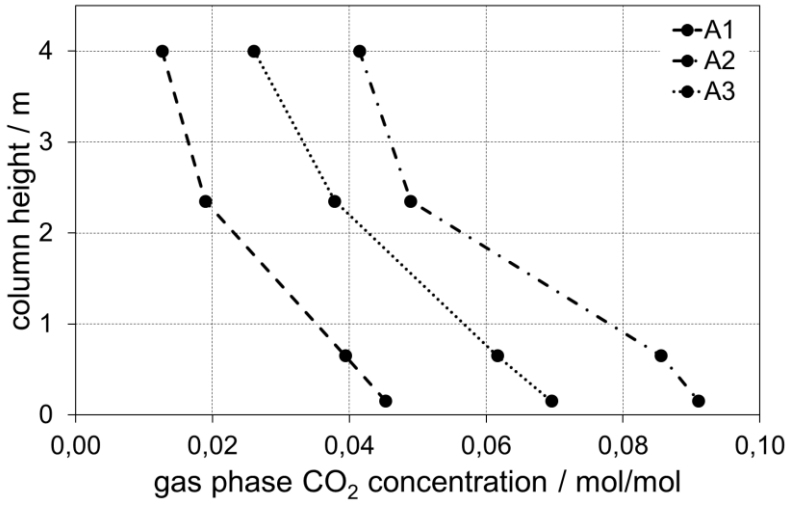


Figure 6: Measured CO_2 concentration profiles

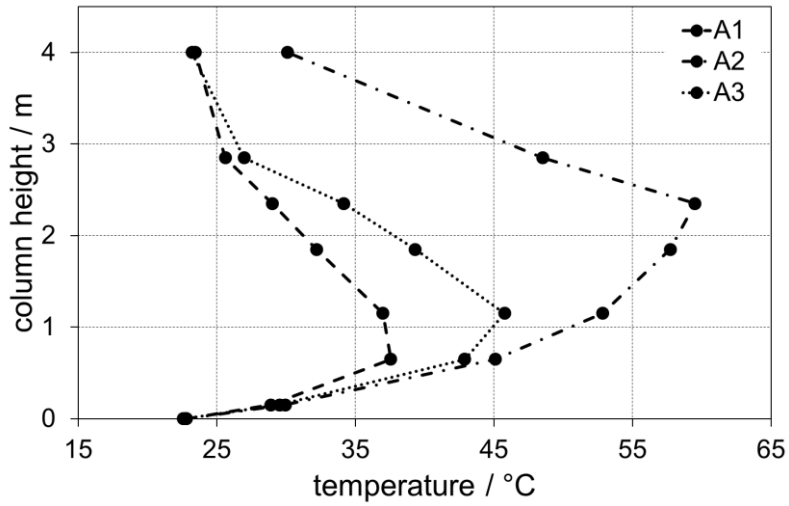


Figure 7: Measured CO_2 temperature profiles

raising, due to the exothermic reactions. The measurements were performed after reaching the steady state.

Figure 6 shows the gas-phase CO_2 concentration profiles and Figure 7 the temperature profiles along the column height. As described above, all experiments were carried out under the same conditions except the initial CO_2-concentration.

A similar trend in the CO_2 concentration profiles in Figure 6 indicates that approximately the same amount of CO_2 is absorbed in all experiments. Taking into account that MEA concentration and mass flow rate were the same, we can conclude that the plant worked reasonably well. As can be seen from Figure 7, a temperature bulge appears near the bottom of the column for experiment A1, somewhat higher for A3 and near the middle of the column for A2. Such behaviour was reported by Kvamsdal and Rochelle (2008), who analysed the location of the temperature bulge in CO_2 absorption from flue gas by aqueous MEA solutions. This provides an additional proof that the measured values are correct.

3.3 Desorption experiments

One desorption study was carried out to check whether the evaporator is working well and CO_2 is desorbed. The experiment was conducted as follows: The loaded amine solution was circulated through the desorber and heated in the evaporator. Over the whole period of the experiment, gas-phase samples were analysed. Figure 8 shows the change of the gas-phase temperature at the top as well as the solvent temperature at the bottom of the column with time. Additionally, the increase of the dry CO_2-concentration

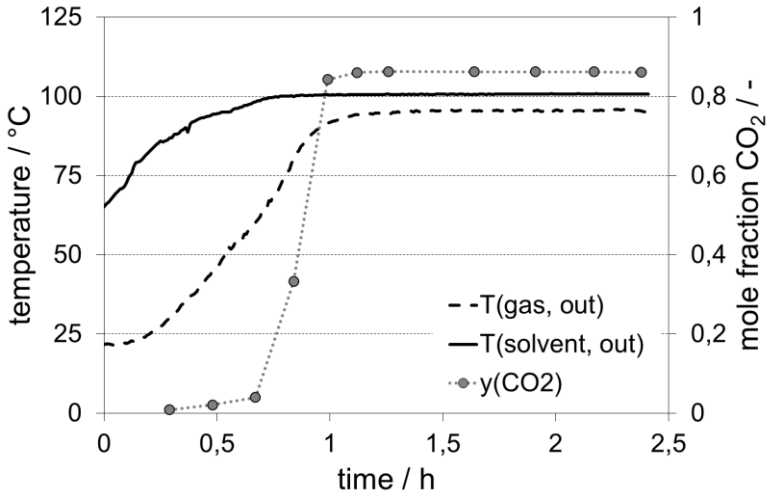


Figure 8: Gas-phase temperature and CO_2-concentration at the top and liquid-phase temperature at the bottom of the column as functions of time.

is visualized. After approx. 1 h, a steady state was reached. At this point, the outlet temperatures of the gas and liquid phase as well as the CO_2-concnetration at the top of the column approached their highest values. Generally, we may conclude that, similar to absorption, the desorption experiment shows reasonable tendencies, so that the closed loop investigations can be started.

4. Conclusions

In this work, we presented the recently build pilot plant for the investigations of (reactive) absorption processes. The plant was run in the fluid dynamic mode, followed by absorption and desorption experiments with the system CO_2/air - aqueous MEA solution. The measured pressure drop, temperatures and concentrations were compared to literature data. In all three operation modes, good performance could be achieved. Thus, the pilot plant can be used with confidence for thorough experimentation in the field of packing and solvent design.

Acknowledgements

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