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## CO<sub>2</sub> absorption efficiency and heat consumption measured at high gas to liquid ratios in laboratory rig

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### Abstract

A laboratory rig for CO<sub>2</sub> absorption into amine solvents including desorption has been in operation at University College of Southeast Norway for several years. Measurements of CO<sub>2</sub> removal efficiency and heat consumption at different process conditions and varied gas concentrations, gas flows and liquid flows have been performed. In earlier work, the gas to liquid ratio has been lower than in a normal large-scale absorption column to achieve high CO<sub>2</sub> removal efficiencies, and the heat consumption has been very high. In this work, experiments with a higher gas to liquid ratio have been performed to reduce the heat consumption per kg CO<sub>2</sub> removed.

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*Keywords:* CO<sub>2</sub>; absorption; amine; experiments

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### 1. Introduction

The project with a CO<sub>2</sub> absorption and desorption rig at University College of Southeast Norway (earlier Telemark University College) has been developed since 2007, and since then there have been student projects every year to design, improve and operate the CO<sub>2</sub> rig. The first experimental results of CO<sub>2</sub> removal efficiency as a function of

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different parameters were presented in 2012 [1]. In 2014, the steam consumption (as a measure of reboiler heat) as a function of different parameters was also measured [2]. Due to the low absorber height, the heat consumption necessary to achieve a high removal efficiency was found to be very high compared to what is expected in a large-scale column.

This work has emphasis on updated experimental data from 2015 for CO<sub>2</sub> removal efficiency and heat consumption as a function of gas flow, gas composition and liquid flow especially for a high gas to liquid ratio. It is expected that this will reduce the heat consumption per kg CO<sub>2</sub> removed compared to earlier experiments. A photograph of the rig is shown in Fig. 1.

## 2. Process and system description

### 2.1. Process description

Fig. 2 shows a Piping and Instrumentation Diagram (P&ID) of the laboratory rig. The number in parenthesis refer to the equipment shown in Fig. 1. In the absorption column (1) air with a fraction of CO<sub>2</sub> is mixed countercurrently with a circulating solvent. The solvent from the absorption column (absorber) flows to a buffer tank (2) and is pumped (by 3) through a heat exchanger (4) to the desorption column (desorber). The desorption column (5) is heated with steam by the reboiler and is cooled by cooling water in the condenser. The CO<sub>2</sub> flows from the top of the desorber, and regenerated solvent flows out of the bottom of the desorber. The regenerated solvent is heat exchanged (in 4) and is pumped back (by 3) through a cooling heat exchanger (6) to the absorber.

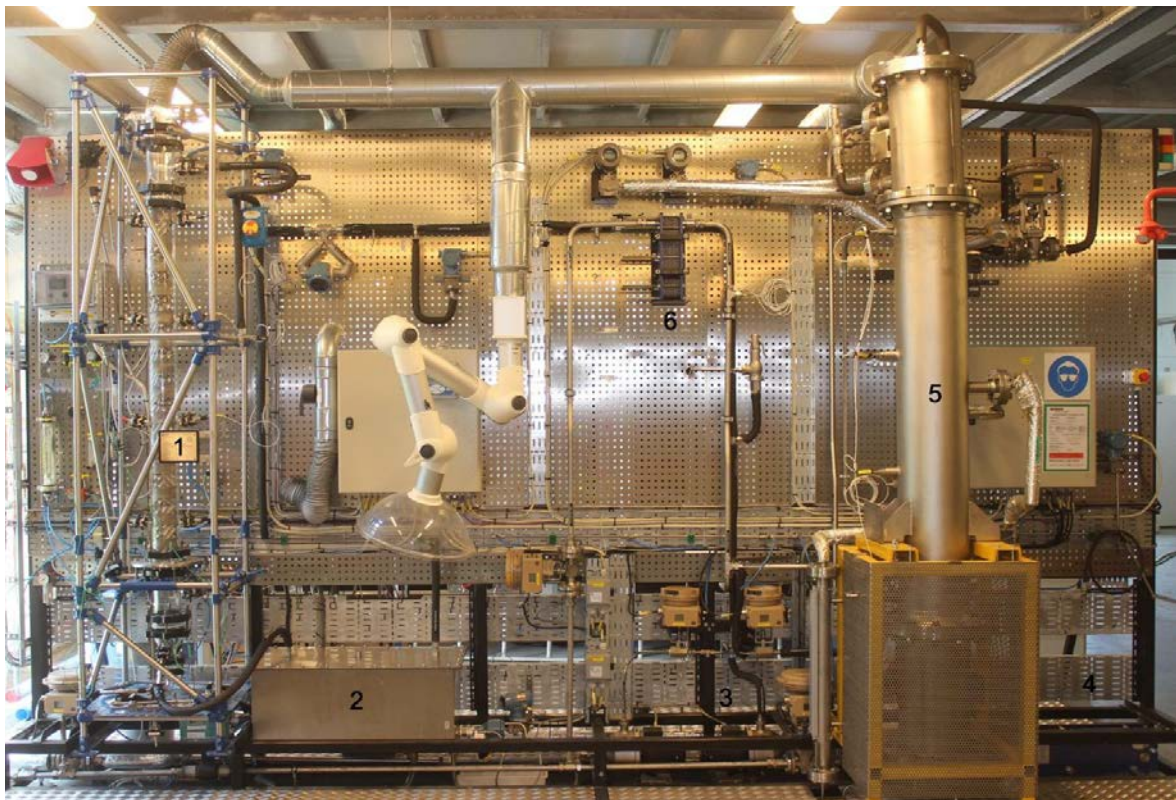


Fig. 1. Photograph of the CO<sub>2</sub> rig from spring 2016 showing 1. Absorption column, 2. Buffer tank 3. Pumps, 4. Crossflow heat exchanger, 5. Desorption column with reboiler and condenser, 6. Cooling heat exchanger

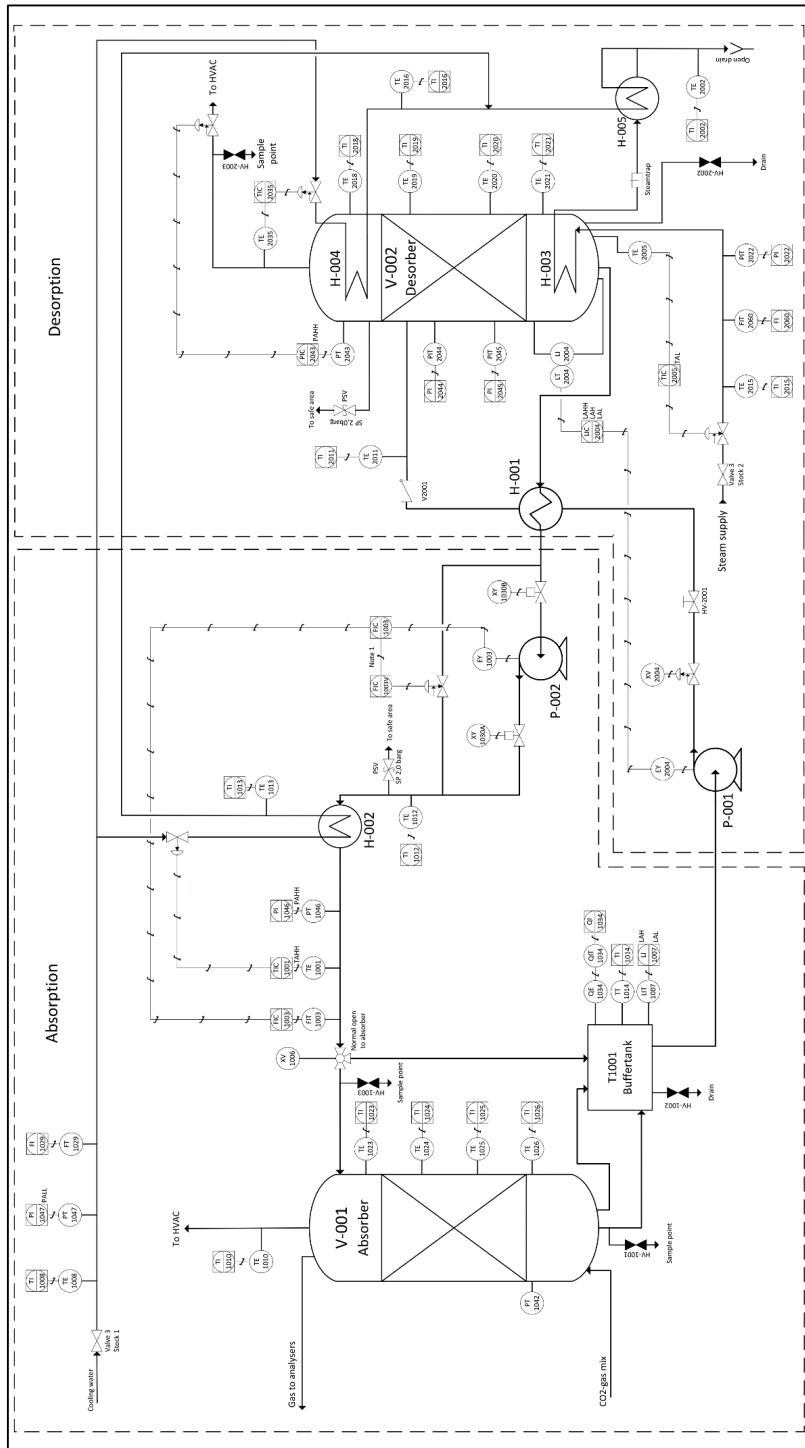


Fig. 2. Piping and Instrumentation Diagram (P&ID) of the laboratory rig

The absorption column which is made of glass, has a structured packing height of 1500 mm, a diameter of 100 mm and a total height of 2500 mm. The desorption column has a packing height with P-rings of 1000 mm, a diameter of 265 mm and a total height including reboiler and condenser of 3000 mm. A more detailed process description including description of the main equipment is given in an earlier report [1].

## 2.2. Description of control and instrumentation

The rig has a distributed PLC (Programmable Logic Controller) system with instruments for temperatures, pressures, flows and levels. The CO<sub>2</sub> concentration in a gas sample stream from the inlet or the outlet of the absorber is measured with NDIR (Non-Dispersive InfraRed). The CO<sub>2</sub> content in the liquid is analyzed by titration in some experiment series to check the material balance. The flows of air and CO<sub>2</sub> to the absorber are measured by rotameters. The flow of amine to the absorber column is controlled by a variable speed pump, the temperature of the amine flow to the absorber is kept constant using a heat exchanger with cooling. A steam valve controls the temperature in the reboiler, and a pressure reduction valve on the gas flow outlet of the desorber controls the desorber pressure. The PID (proportional, integral and derivative) controllers have been described in more detail earlier [1,2].

## 3. Experiments

Operation of the plant in earlier experiments has been described earlier [1,2]. Detailed operation conditions are described in detail in the project reports documenting the original experiments [3,4,5,6,7,8]. Start-up, shut-down and operation procedures have been updated every year. The experiments from spring 2015 are described in the project report [8]. In this work, measurements of CO<sub>2</sub> removal efficiency and heat consumption were measured as a function of gas flow, gas concentration and liquid flow. Absorption and regeneration experiments using about 30 wt-% monoethanol amine (MEA) as the solvent have been performed with the desorber part including the reboiler and the condenser in operation. In the first experiment series the wt-% MEA was 30 %, but due to evaporation of water, the concentration of MEA increased gradually. The rig has been operated under stable conditions with absorption of CO<sub>2</sub>, desorption at approximately 1.8 bar(a), steam heating to 120°C and amine recirculation. After a change in operating conditions, the rig spends in order of magnitude 10 minutes for stabilization of the continuously measured parameters. Parameters which have been varied in the experiments in this work, are the gas flow, CO<sub>2</sub> concentration in inlet gas and the liquid circulation rate.

## 4. Results and discussion

### 4.1. CO<sub>2</sub> removal efficiency as a function of gas flow

The gas flow to the absorber with 10 vol-% CO<sub>2</sub> has been varied with a constant liquid flow of approximately 150 l/h. The removal efficiency was reduced from close to 100 % at 5 Nm<sup>3</sup>/h down to about 30 % at 35 Nm<sup>3</sup>/h (which is equivalent to a gas velocity of about 1.0 m/s through the absorber). The results from spring 2015 are compared with three experiment series from autumn 2012, spring 2013 and autumn 2013 in Fig. 3.

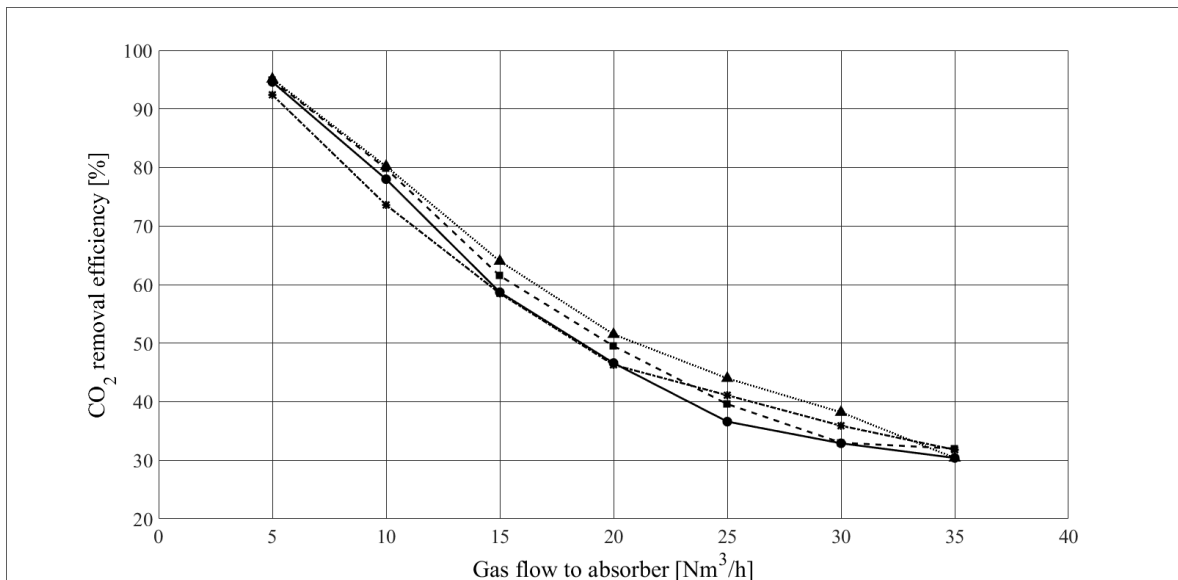


Fig. 3. Measured CO<sub>2</sub> removal efficiency in 30 wt-% MEA as a function of gas flow at liquid flow 140 l/h (■ - dashed line [5]), (\* - dash-dot line [6]) and 150 l/h (▲ - dotted line [7]), (● - solid line [8])

As in earlier experiments, the removal efficiency in % CO<sub>2</sub> removal is reduced as the gas flow increases. The retention time of the gas and then the absorption efficiency from the gas is expected to be reduced when the gas flow increases.

The comparison of the experiment series shows that the maximum deviation in the measured CO<sub>2</sub> removal efficiency is approximately 5 %-points. The corresponding maximum deviation in measured gas flow is approximately 5 Nm<sup>3</sup>/h or 25 % relative at low gas flow. This gives an indication of the uncertainty of the measurements. Within each experiment series, the curve is very smooth with deviations from a fitted line well below 5 %-points. This may indicate that the conditions vary slightly between the series. The gas inlet temperatures and the MEA concentration may have varied slightly between the series. The new experimental values from spring 2015 lie between earlier values except for one point which is slightly lower. The maximum deviation is still approximately 5 %-points.

To obtain results for a high gas to liquid ratio, the gas flow was varied when the liquid flow was 20 kg/h and 40 kg/h. The results are shown in Fig. 4. The removal efficiency is order of magnitude twice as large when the liquid flow is increased from 20 to 40 l/h. When the gas flow is low, the removal efficiency increases about proportionally with liquid flow. When the gas flow is high, the removal efficiency increases less than proportionally with liquid flow. This indicates that the absorption liquid tends to be more saturated when the gas flow increases.

In earlier experiment series, the liquid flows in similar series have been higher. Removal efficiencies for 15 Nm<sup>3</sup>/h and 40 l/h have been measured earlier [2], and the results are similar in these experiments.

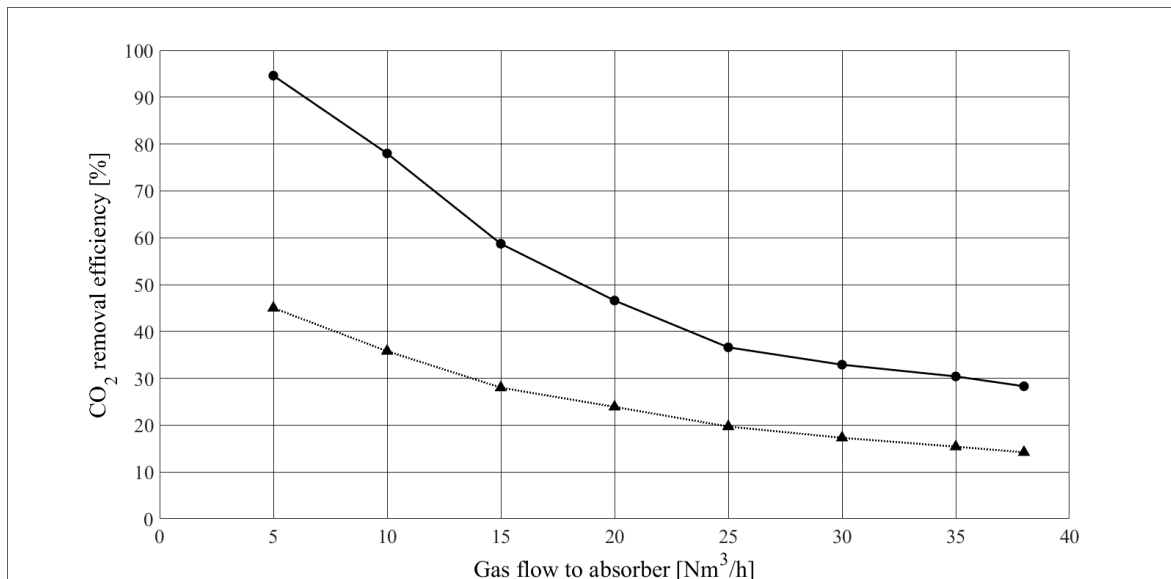


Fig.4. Measured CO<sub>2</sub> removal efficiency in 30 wt-% MEA as a function of gas flow at liquid flow 40 l/h (● - solid line), and 20 l/h (▲ - dotted line)

4.2. CO<sub>2</sub> removal efficiency as a function of liquid flow at different CO<sub>2</sub>-concentrations in inlet gas

The liquid circulation flow with temperature 40 °C at the absorber inlet was varied with a constant gas flow at 30 Nm<sup>3</sup>/h (which is equivalent to a gas velocity close to 1 m/s). The inlet concentration of CO<sub>2</sub> was 5, 10 and 15 vol-% respectively. The results are shown in Fig. 5.

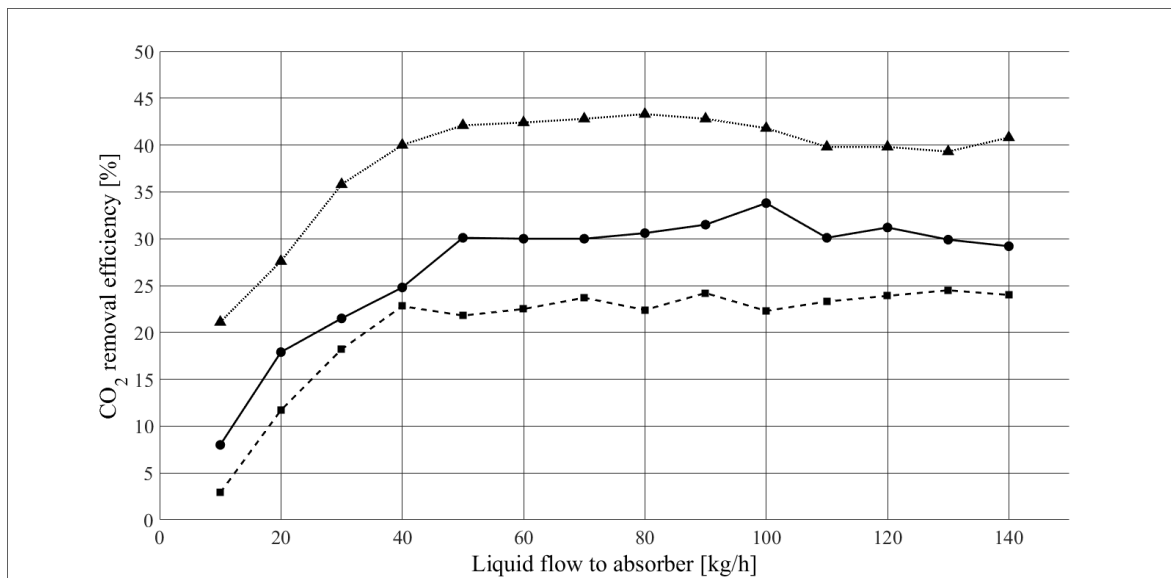


Fig. 5. Measured CO<sub>2</sub> removal efficiency at different gas inlet CO<sub>2</sub> concentrations with 30 Nm<sup>3</sup>/h gas flow, 5 % CO<sub>2</sub>, (▲ - dotted line) 10 % CO<sub>2</sub> (● - solid line [3], and 15 % CO<sub>2</sub> (■ - dashed line [7]).

The CO<sub>2</sub> removal efficiency increased gradually to a close to constant value when the liquid flow increased to above 60 kg/h at all inlet CO<sub>2</sub> concentrations. The removal efficiency was decreasing significantly when the inlet CO<sub>2</sub> content increased from 5 to 15 %. An explanation for this is that higher CO<sub>2</sub> concentration will increase the CO<sub>2</sub> concentration in the liquid towards saturation, and especially close to the surface.

The curve for 10 % CO<sub>2</sub> is very close to earlier experiments [1,2]. As in earlier experiments [1,2], the liquid flow achieves a maximum CO<sub>2</sub> removal efficiency at some value in most of the experiment series. The expected reason for a certain liquid load showing a maximum removal efficiency is that the absorption efficiency is expected to increase with liquid flow for low liquid flow rates, but that the efficiency decreases if the column is overloaded.

The curves for removal grade as a function of liquid flow is also in earlier experiments less smooth compared to the removal grade as a function of gas flow. A possible explanation is that the uncertainty in the liquid flow is larger because it is not completely stable under operation.

#### 4.3. CO<sub>2</sub> removal efficiency and reboiler heat as a function of gas flow

The gas flow to the absorber with approximately 10 vol-% CO<sub>2</sub> was varied with the liquid flow constant at 40 kg/h. The results are shown in Fig. 6. The CO<sub>2</sub> removal efficiency is close to 100 % at 5 Nm<sup>3</sup>/h, and decreases to about 30 % at 30 Nm<sup>3</sup>/h. The steam consumption per kg CO<sub>2</sub> removed decreased significantly when the gas to liquid flow ratio increased by increasing the gas flow.

The heat consumption measured in earlier experiments have been very high (order of magnitude 30–60 MJ/kg CO<sub>2</sub>) compared to an order of magnitude 4 MJ/kg CO<sub>2</sub> in a large-scale capture plant. One explanation for this has been that it is probably not possible to achieve very low heat consumptions in a laboratory plant with an absorber packing height of only 1500 mm. Another explanation for the high heat consumption is that the high removal efficiency achieved in most of the earlier experiments is at a low gas to liquid ratio.

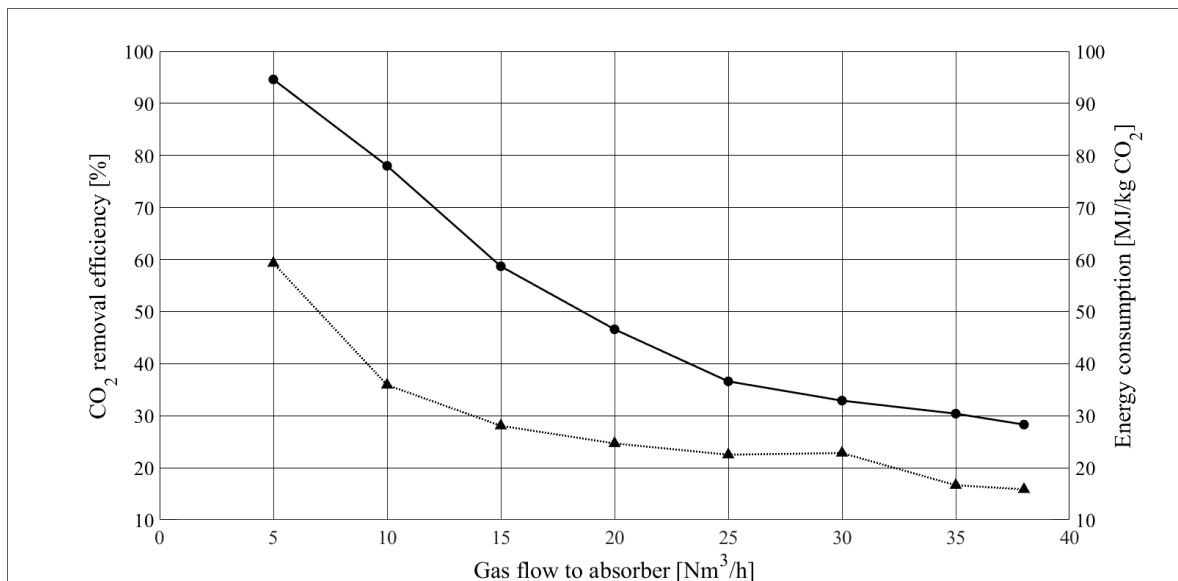


Fig. 6. Measured CO<sub>2</sub> removal efficiency (● - solid line) and energy consumption (▲ - dotted line) as a function of gas flow with liquid flow 40 kg/h.

The energy consumption per CO<sub>2</sub> removed in this work was reduced to 16 MJ/kg at high gas to liquid ratios. Experiments with 20 kg/h also achieved less than 20 MJ/Kg CO<sub>2</sub>, but these experiments did not show the smooth curve obtained with 40 kg/h. The CO<sub>2</sub> loading (mole CO<sub>2</sub> per mole amine) typically increased from 0.30 to 0.35 in the absorber compared to a typical increase of 0.27 to 0.47 in a large scale absorption column [1]. This difference is reasonable if we look at the conditions in the absorption rig column similar to the conditions in the upper section of a taller absorption column.

The heat loss from the equipment may be significant when evaluating the heat demand in the process. In a large-scale process, the heat loss is expected to be very small compared to the heat consumption, but in a small laboratory rig, the heat loss is probably not negligible compared to the heat consumption. In the experiments, the steam consumption was measured to values between 8 and 13 kW (maximum effect is 36 kW). An order of magnitude estimate of the heat loss from the desorption column have been made based on an area of 1.3 m<sup>2</sup>, a temperature difference of 100 °C and an overall heat transfer number of 8 W/(m<sup>2</sup>·°C). The resulting heat loss of 1.04 kW is order of magnitude 10 % of the steam consumption. This indicates that the reboiler heat demand per kg CO<sub>2</sub> in a large-scale process will be slightly over-estimated. However, the heat loss in the laboratory rig is probably not important when searching for the process conditions giving the lowest reboiler heat demand.

The results show clearly that the energy consumption (in MJ/kg CO<sub>2</sub>) decreases drastically as the gas flow (or the gas to liquid ratio) increases. At high gas to liquid ratios, the heat consumption is well below 20 MJ/kg CO<sub>2</sub>.

#### 4.4. Material balances

The CO<sub>2</sub> removal efficiency was in this work calculated for each experiment based on the concentration difference in the gas in and out of the absorption column. The CO<sub>2</sub> concentration was in some cases analyzed based on BaCl<sub>2</sub> titration as explained in earlier reports [2]. The CO<sub>2</sub> absorption rate was for these cases calculated from both the gas side and the liquid side. The difference between the absorption rate calculated from the gas side and the liquid side was earlier calculated to 17 % [4], and to -24 % and -33 % [3]. In experiments from 2013 and 2015 [6,7,8], these differences are also in this order of magnitude.

These deviations are still too high. Possible reasons for these deviations have been discussed earlier [2]. There are considerable uncertainty in the measurements of the gas concentration and liquid concentrations of CO<sub>2</sub>. The small uncertainty in the measured removal efficiency calculated from the gas side in the experiments in this work, indicate that the main uncertainty is on the liquid side.

There is also a question whether the pilot plant runs at stable conditions. The continuously measuring instruments and especially the CO<sub>2</sub> gas analyzers become stable after about 10 minutes. When taking liquid samples for a total material balance, the pilot plant was normally stabilized for about 30 minutes. This might not be enough to stabilize liquid concentrations.

Work has now started to run experiments in the rig with an instrument for continuous measurements of the liquid composition. This will make it easier to be sure that the operating conditions are stable. It will probably also give more accurate liquid composition measurements so that the material balance can be improved.

#### 4.5. Evaluation of conditions in a part of a large-scale column

The order of magnitude CO<sub>2</sub> loading in the liquid is 0.3 after regeneration and 0.35 after absorption. This is close to the conditions in the upper part of a large-scale column. If the regenerated amine could have a higher CO<sub>2</sub> loading, the conditions in the absorption column would be closer to the lower part of a large-scale column. This is a possibility for later experiments.



## 5. Conclusions

The CO<sub>2</sub> absorption rig has been used successfully to measure the absorption efficiency and heat consumption also at high gas to liquid ratios as a function of gas flow, gas concentration and liquid flow at stable conditions. The measured heat consumptions are still high compared to expected large-scale operation, but they are not so high as in earlier experiments. There are possibilities for improvements, especially in the accuracy of measurements of the CO<sub>2</sub> concentrations in the gas and the liquid. The measurements can be used to validate process calculations and to find optimum process conditions.

The results indicate that with a low absorber height, it is not possible to achieve both high removal efficiency and low heat consumption. But when running at a low efficiency, the experiments can illustrate the conditions in the upper part of a large-scale absorption column. By increasing the CO<sub>2</sub> loading in the liquid to the absorber, also the conditions in the lower part of the column might be illustrated in future experiments.

## Acknowledgements

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