Laboratory Studies of Post-combustion CO₂ Capture by Absorption with MEA and AMP Solvents

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Abstract With regard to the climate policy that has been adopted, the implementation of the carbon capture and storage and carbon capture and utilization technologies seems to be the unavoidable solution to reduce emissions from the energy sector. As most Polish energy is derived from coal, the government has taken steps to reduce CO₂ emissions by initiating a strategic research program—“Advanced technologies for energy generation: Development of a technology for highly efficient zero-emission coal-fired power units integrated with CO₂ capture.” This paper presents the results of an investigation of a process for continuous removal of CO₂ from the gas stream on a laboratory unit. This study was conducted at the Institute for Chemical Processing of Coal in Zabrze, Poland. The purpose of the tests that were conducted was to establish the effect of the sorption column load on CO₂ recovery and on the process regeneration heat duty. In the second part of the paper, a simple comparison of three amine solvents is presented as a part of the solvent selection process for further pilot plant tests.

Keywords CO₂ · Absorption · MEA · CCS · L/G ratio

Abbreviations

MEA Monoethanolamine
AMP 2-Amino-2-methyl-1-propanol
PZ Piperazine

1 Introduction

The carbon dioxide concentration in the Earth's atmosphere has oscillated at approximately 280 ppm for more than ten thousand years. However, since the second half of the nineteenth century, a significant rise in the carbon dioxide concentration in the atmosphere at the rate of 1.2 ppm per year has been observed, and today, the concentration of carbon dioxide equals approximately 380 ppm. The analyses conducted on the subject by the Intergovernmental Panel on Climate Change, among others, prove that human activity contributes significantly to the increase in the CO₂ concentration in the atmosphere and that anthropogenic carbon dioxide consequently leads to climate change [1–3]. Remaining at the current level of utilization of fossil fuels will increase climate change [4].

To counteract climate change, low emissions and high-efficiency technologies should be implemented. These “clean carbon technologies” should allow the burning of fossil fuels without a negative environmental impact. One of the solutions to reduce large carbon dioxide emissions (occurring during fossil fuel combustion) may be found in the implementation of post-combustion technologies. Almost 90 % of the energy in Poland is derived from coal, so the government also has taken steps to develop clean coal technologies that can reduce CO₂ emissions by initiating a strategic research program—“Advanced technologies for energy generation: Development of a technology for highly efficient zero-emission coal-fired power units integrated with CO₂ capture” [5,6].

Most of the carbon capture technologies are known at present, but they are used on a limited scale (mainly in the chemical, petrochemical and food processing industries), and these carbon capture technologies are not presently sufficient for application in full-scale power plants [7]. A vast number

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of post-combustion technologies for carbon dioxide removal exist in industry: physical and chemical absorption, adsorption, membrane processes, cryogenic and electrochemical processes [1]. Among these, chemical absorption is the most developed and most commonly used method to remove CO₂ from large gaseous streams. As the most advanced method, chemical absorption was selected because of the gas stream properties: temperature, pressure, CO₂ concentration and gas flow rate (Table 1). Flue gas from the power plant is emitted to the atmosphere at a pressure close to the atmospheric pressure, which eliminates the possibility of applying the physical absorption methods widely used in the chemical and refinery industries. The remaining post-combustion technologies (membrane, cryogenic and electrochemical processes) can be applied for treating moderate gas streams and are not suitable for application in the professional power industry.

Absorption processes based on amines were developed over 80 years ago for the removal of H₂S and CO₂ from natural gas [9]. Thereafter, this absorption process has been adapted for crude oil mining. Separated CO₂ from the process was further utilized for carbonating brines, production of dry ice, saturating drinks and to enhance production of oil until 1970 [10]. In the 1970s, Dow Chemical and Union Carbide companies started to use the amine absorption processes in the industry based on 30 wt% monoethanolamine (MEA) for the intensification of oil production (Econamine process) [11]. In practice, the 30 wt% of MEA solution found the biggest application in the cleaning process for gases resulting from the combustion of natural gas [10]. Currently, the majority of the commercial installations for CO₂ removal from the process gases are based on solutions containing MEA because of its advantages: low molecular weight resulting in high solution volume capacity, a relatively high rate of reaction with CO₂ and the high pH of the solution (pH 12–13). Unfortunately, MEA exhibits many disadvantages, such as high heat of absorption and the necessity of supplying an appropriate amount of energy to break the bonding in the MEA-CO₂ complex in the desorption process, as well

as high corrosivity relative to carbon steel, which translates to high purchase costs for the corrosion inhibitors [10, 12]. The main world trend in research on CO₂ removal from the flue gases by the amines, targeted to overcome MEA disadvantages, has been to find a solution that will have an energy demand as low as possible to remove one unit of CO₂ from the flue gas and a high sorption capacity resulting in a high removal efficiency (>85 %—requirements of the European Commission [13, 14]). However, MEA is an ideal solvent to use as a benchmark to explore other new solvents because of its stable performance, as well as extensive results from the research at different scale installations [12, 15–18]. Similar work directed at finding new amine solvents and other technological solutions that can reduce CO₂ removal energy consumption from the flue gas [19–22] is also conducted in Poland within the strategic research program.

2 Experimental

2.1 Laboratory Setup

For continuous CO₂ removal from flue gas, a special laboratory unit was constructed. The most important parameters of the unit are given in Table 2.

The basic elements of the setup were absorber, stripper and main heat exchanger. The columns were built from metal and glass and were filled with glass Raschig rings 6 × 6 × 0.5 mm (Raschig GmbH) with an option to replace with another type of packing [23]. Glass walls enabled the observation of distributor liquid sprinkling and liquid flow distribution over the packing. The main heat exchanger was necessary to exchange heat between the hot lean solution and the cooler rich solution. The main heat exchanger was used to reduce the overall stripper size. When necessary, the rich solution could be cooled to a lower temperature in the final water cooler. The solution was additionally filtered through an activated carbon bed to remove solid contaminants and some of the products of amine degradation. The heat needed for the CO₂ desorption from the solution was delivered to the system using an adjustable electric heater. That solution is convenient under

Table 1 Typical flue gas composition from a coal-fired unit in Poland [8]

Component	Composition
CO ₂ (vol%)	13.14
H ₂ O (vol%)	6.87
O ₂ (vol%)	7.69
N ₂ (vol%)	71.42
SO ₂ (mg/m ³)	200–400
NO _x (mg/m ³)	250–350
Dust (mg/m ³)	30
Temperature (°C)	40
Pressure (bar)	1.45

Table 2 Main laboratory setup parameters

Parameter	Value	Unit
Packing surface area [23]	805	m ² /m ³
Packing void fraction [23]	0.807	m ³ /m ³
Absorber diameter	0.100	m
Total absorber packing height	1.2	m
Stripper diameter	0.100	m
Total stripper packing height	1.2	m
Main heat exchanger area	0.5	m ²

laboratory conditions and simplifies the calculations. Despite having the column stripper and piping insulated, the ambient heat loss was so high that the heat loss strongly affected the value of the reboiler heat duty. Transferring the heat duty values directly to the industrial process would be inappropriate but still sufficient to allow the comparison of different solvents.

The laboratory setup described here has been equipped with approximately 30 measuring points and controlled by a supervisory control and data acquisition system (SCADA). The installation has the possibility of controlling parameters of the gas and solvent solution by *Intergaz* gas meters and *Tecfluid M21* flowmeters. The temperature is controlled by *Limatherm* PT-100 sensors and the pressure by pressure transducers from *Wika*. The amount of electricity to the stripper heater is regulated by *Aplisens PMS-200* electric current. The CO₂ volume fraction both at the inlet and at outlet is measured with *Siemens Ultramat 23* and *Sick-Maihak S700* gas analyzers.

2.2 Process Flow Description

A schematic diagram and a picture of the laboratory setup to investigate the CO₂ removal process are presented in Fig. 1. According to the schematic diagram, air is compressed in an air blower to the discharge pressure of 130–150 kPa_{abs}. After drying, air is directed to the gas mixer where the gas mixture is combined with carbon dioxide. The composed gas mixture is next directed to the absorber where the CO₂ absorption process takes place. Cleaned gas is evacuated from the column through the gas meter and discharged outside.

In the absorption column, the packing gas mixture comes into contact with the lean amine solution from stripper in a countercurrent way. The rich solution with the absorbed carbon dioxide is pumped from the bottom of the absorber and passed through the main heat exchanger to the top of the stripper. In the stripper, thermal carbon dioxide regeneration takes place. In next step, the lean solution is pumped back to the absorber top from the bottom of the stripper.

Losses of water/sorbent are supplemented periodically from the solvent tank.

2.3 Materials and Media

The gas mixture was prepared from ambient air and carbon dioxide (purity 4.0) delivered by Linde AG. Carbon dioxide in gas composition oscillated at 13 vol% level. To simplify the experiment, ambient air instead of nitrogen was used. This simplification brought the experiment to more real conditions. Due to the relatively short time tests, solvent oxygen degradation was negligible.

Baseline studies were conducted using 30 wt% monoethanolamine solution. Concentrated monoethanolamine

(MEA, CAS #000141-43-5) was obtained from Acros Organics.

For solvent comparison studies, the following solutions were also used:

- 30 wt% 2-amino-2-methyl-1-propanol with 10 wt% piperazine. Both reagents—2-amino-2-methyl-1-propanol (AMP, CAS #000124-68-5) and piperazine (PZ, CAS #000110-85-0)—were obtained from Sigma-Aldrich.
- Multicomponent solution 2—developed at the Institute for Chemical Processing of Coal—contains 15 wt% of first-order amine, 20 wt% of AMP, 2 wt% of activator, 63 wt% of organic solution and water [25].

Distilled water was used during solvent preparation.

2.4 Testing Procedure

The performance of the 30 wt% MEA was evaluated to estimate its ability to capture carbon dioxide and to minimize the reboiler heat duty under laboratory conditions as well as to obtain baseline performance for solvent comparison. Table 3 describes the range of process conditions applied in the laboratory setup during the studies that we conducted.

The measurements presented in this paper were selected from the database of trends recorded in the SCADA system. The tests were considered of value only when the period of the steady state lasted at least 1 h. For better balancing or performance estimation, the average of the chosen parameters from the steady-state period was used for further calculations.

The lines at presented figures were used to connect experimental data, and these lines serve only to join the data points.

2.5 Error Determination

After conducting the appropriate calculations, which included inlet gas parameters and mass balance, an estimation of the maximum absolute error was made (Table 4). Based on several measurement cycles, the precision of the test method was also established. Repeatability of the results was determined within the range of the measurement errors.

3 Results and Discussion

3.1 MEA Baseline Performance: The Effect of Liquid-to-Gas (L/G) Loading Ratio on CO₂ Recovery and Reboiler Heat Duty

The effect of varying the L/G ratio on CO₂ recovery and gross reboiler heat duty for three different stripper heating powers is illustrated in Figs. 2, 3 and 4.

Fig. 1 Schematic diagram and a picture of the laboratory unit to investigate the CO₂ capture process [24]

At constant heater power, within L/G ratio changes, the CO₂ recovery curves create reversed parabolic trends. Standard parabolic regularity can be observed for reboiler heat duty curves where some visible minimum at a specific L/G ratio can be observed. This regularity is especially visible for lower stripper power adjustments—2000 W and Fig. 2. The reboiler heat duty parabolic trends as a function of L/G ratio

were also reported by several authors: Han [12], Knudsen [15], Artanto [16], Mangalapally [26]. The figures show that to obtain higher CO₂ recovery and the lowest energy consumption, a specific liquid-to-gas ratio is required. Further, for a given gas flow rate, there is a specific or optimal solvent flow rate to operate the CO₂ absorption process. This regularity can be explained because for a liquid ratio that

Table 3 Process conditions for laboratory unit trial

Parameter	Value	Unit
Gas flow rate	5.5 ± 0.2	m^3/h
CO_2 in gas composition	13 ± 1	vol%
Absorption temperature	40 ± 3	$^\circ\text{C}$
Absorber pressure	1.2	bar_a
Stripper pressure	1.15	bar_a
Liquid/solvent flow rate	various: $(20\text{--}60) \pm 1$	dm^3/h
Heater power set for MEA performance evaluation	various: 2000, 2500, 3000	W
Heater power set for solvent comparison	2000	W

Table 4 Maximal absolute error for selected parameters

Parameter	Error	Unit
<i>Instrumentation error</i>		
Gas flow rate	0.36	m^3/h
CO_2 in inlet gas composition	1	vol%
CO_2 in outlet gas composition	0.5	vol%
Liquid/solvent flow rate	2.4	dm^3/h
Temperature	1	$^\circ\text{C}$
Pressure	0.002	bar_a
<i>Proportional error</i>		
CO_2 recovery	1.2	%
Reboiler heat duty (gross)	0.08	$\text{MJ}/\text{kg}_{\text{CO}_2}$

Fig. 2 The effect of L/G ratio on CO_2 recovery and reboiler heat duty for stripper heater power set at 2000 W for the MEA solution

is too low, there is not enough free amine in the small circulating volume of solvent to absorb a constant amount of carbon dioxide in the absorption column, despite the low solvent loading leaving the stripper. However, a liquid flow

rate that is too high results in a higher solvent loading leaving the stripper, so the circulating solvent cannot absorb the same amount of carbon dioxide in the absorption column. Thus, there is also insufficient free amine to absorb the CO_2 from the gas. The effect on the decrease in CO_2 removal is similar.

The reboiler heat duty is correlated with the energy supplied to the process and the mass of the CO_2 removed, so that both values are closely related to each other. The reboiler heat duty values do not include heat losses to the ambient air.

Figure 2 shows the effect on the CO_2 recovery of the L/G ratio associated with the reboiler heat duty. At this setting of the heater power, the lowest regeneration energy demand (approximately $7.70 \text{ MJ}/\text{kg}_{\text{CO}_2}$) can be observed within a $5.9\text{--}7.3 \text{ kg}/\text{kg}$ L/G ratio. The maximum achieved for the CO_2 capture efficiency is in the range of $77.6\text{--}78.3\%$. For the heat power setup at 2500 W (Fig. 3), the highest CO_2 recovery amounts to $7.1\text{--}8.8 \text{ kg}/\text{kg}$ L/G ratio. The lowest reboiler

Fig. 3 The effect of L/G ratio on CO_2 recovery and reboiler heat duty for stripper heater power set at 2500 W for MEA solution

Fig. 4 The effect of L/G ratio on CO₂ recovery and reboiler heat duty for stripper heater power set at 3000 W for MEA solution

Fig. 5 The effect of L/G ratio on CO₂ recovery for different stripper heater power settings for the MEA solution

heat duty can be observed in the range of 8.64–9.50 MJ/kg CO₂. Further increase in the setting of the heater power (3000 W), as shown in Fig. 4, increases the CO₂ recovery level (up to 90.7 %). Figure 4 might also suggest that with the L/G ratio, the carbon dioxide efficiency will trend to infinity, but according to the minimum reboiler heat duty obtained, we can assume that the maximum CO₂ recovery has been achieved. Further increasing the L/G ratio was not possible due to solvent flow meter limitations. The lowest reboiler heat duty can be seen in the 7.9–8.4 kg/kg L/G ratio range.

Fig. 6 The effect of L/G ratio on reboiler heat duty for different stripper heater power settings for the MEA solution

Comparing the effect of the L/G ratio on CO₂ recovery for every stripper power setting (Fig. 5), we can see from the graph that the stripper heater power set at 3000 W exhibits the highest capture efficiency. However, CO₂ recovery at the L/G ratio in the range from 2.6 to 5.5 is almost the same between 3000 and 2500 W heater power setting, implying that minimal solvent loading and excess heat utilized in the condenser and/or wasted to the ambient air have been achieved at 3000 W. For an L/G ratio higher than 5.5, the efficiency is decreasing. This figure also indicates that, for the stripper heater set at 3000 and 2500 W, CO₂ recovery is not significantly affected by L/G ratio at 2000 W because the lean solvent for 2000 W has a higher loading than the others, so with the different liquid flow rates the regeneration impact on the solvent loading is higher. From the graph, we can also see that for every stripper heater power setting, the optimal L/G ratio is different. An optimal L/G ratio increases with the stripper heater increase.

Low CO₂ recovery translates into a low amount of CO₂ separated from the gas stream, which is reflected in the regeneration heat duty related to the unit of mass. Comparing reboiler heat duties for different stripper heater power settings, the same regularity with optimal L/G ratio can be observed (Fig. 6). The lowest regeneration energy consumption and the optimal L/G ratio are naturally the same as in Fig. 5, due to the CO₂ removal rate. Direct comparison of the values of the reboiler heat duty shows clearly that stripper heat power set at 2000 W is superior to other heater power settings, giving the lowest heat duty.

In the range of removal efficiencies (>85 %) that we are interested in, by comparison with other publications, the reboiler heat duty results that we report seem quite high

[15,16,26,27]. Our results can be explained by the significant stripper heat losses to the ambient air. An estimation of the stripper heat losses to the ambient air revealed that even 30 % of the electric heat that is supplied is wasted. A confirmation may be found in the literature [28,29], where similar laboratory-scale installations exhibit a similar range of reboiler heat duties. From our other research, we can see that with an increase in the process scale, the reboiler heat duty decreases. The lowest desorption heat requirement can be observed for the pilot plant test, where similar results were achieved for all units. As suggested, this result is related mainly to the unit heat exchangers. The comparison can be found in [30].

3.2 Solvent Comparison

The figure (Fig. 7) illustrates the effect of the L/G ratio on CO₂ recovery for three selected amine solutions: 30 wt% MEA—the standard reference solution, the amine most commonly used in the industry for removing acidic components; AMP/PZ solution, a system of an amine with steric hindrance with the addition of a polyamine; and multicomponent solution 2, which is a novel mixture of an amine with an organic compound [25]. As predicted, the novel solvents gave a higher CO₂ recovery than the standard MEA solution. Differences in CO₂ recovery between the solutions analyzed are dictated by the various sorption capacities, reaction kinetics (absorption rate) and some physicochemical properties (density, viscosity, surface tension, etc.) that were described in previous publications where new absorption solutions were found [25,31].

Fig. 7 The effect of L/G ratio on CO₂ recovery for MEA, AMP+PZ and multicomponent solution 2 for stripper heater power set at 2000 W

Multicomponent CO₂ recovery exhibits higher CO₂ recovery than MEA solution, mainly because of the higher sorption capacity and comparable absorption rate [25]. Thus, the solvent can absorb more carbon dioxide molecules at a similar liquid flow. CO₂ recovery for the multicomponent did not significantly change CO₂ recovery with a liquid-to-gas ratio compared to other solutions, so the multicomponent can be used over a wider L/G range, giving a more constant CO₂ separation efficiency (6.5–8.4 kg/kg). Among the others, the highest CO₂ efficiency at the conducted liquid flow is shown by AMP/PZ (93.4–97.0 %). This result can be explained by the higher sorption capacity of AMP/PZ with faster reaction kinetics. Therefore, the sorbent can absorb additional CO₂ particles faster [31].

The optimal CO₂ recovery for AMP/PZ solution was obtained for an L/G ratio of approximately 7.6 kg/kg, which was higher than MEA (ca. 6.5) and higher than multicomponent (ca. 7.2 kg/kg).

Figure 8 illustrates the effects of the L/G ratio on the reboiler heat duty for selected solvents. For MEA at an L/G ratio equal to approximately 6.5, we see the lowest reboiler heat duty. With the L/G ratio increasing, the optimal reboiler heat duty for both AMP/PZ and multicomponent decreases [32]. The reboiler heat duty in the solvent regeneration process for both AMP/PZ and multicomponent is lower than with the MEA solution. Therefore, the strong influence on the studied system arises from the relationship between the separated CO₂ from the gas stream and the reboiler heat duty as well as the kinetics of the absorption [31].

From Figs. 7 and 8, we can simply read that MEA requires a higher heater power setting to attain satisfactory efficiency

Fig. 8 The effect of the L/G ratio on the reboiler heat duty for MEA, AMP+PZ and the multicomponent solution 2 for stripper heater power setting at 2000 W

(>85 %). Thus, the reboiler heat duty will increase. Lowering the heater power setting for AMP/PZ solution can lower heat requirements in addition, so AMP/PZ solution will have the lowest reboiler heat duty among the solvents investigated and will give a perspective for investigating the CO₂ absorption process on a larger scale.

4 Conclusions

The results indicate how important the ability to operate CO₂ removal processes at the proper L/G ratio and heater power setting is. Further, for a given heater power setting, there is a specific or optimal L/G ratio to operate the CO₂ removal process. Increasing the heater power setting increases the optimal L/G ratio slightly. Unskillful running of the process can lead to excessive heat consumption in the desorption process, which is incommensurable to the effect of the CO₂ recovery. The reboiler heat duty results that we obtained compared to other publications seem quite high, caused by substantial heat losses at the small scale of the installation.

The results obtained for selected solvents show that it is possible to prepare solutions that feature higher CO₂ removal efficiency and lower regeneration energy consumption per kg of captured CO₂ compared to the 30 wt% solution of MEA, which is now the most widely used solvent for the process. The AMP/PZ solution gave the most perspective for investigating the CO₂ absorption process on a larger scale due to the highest carbon dioxide removal rate and almost the lowest decreases in regeneration energy consumption.

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