DE GRUYTER OPEN
Chemical and Process Engineering 2015, 36 (1), 39-48
DOI: 10.1515/cpe-2015-0003

PDU[1]-SCALE EXPERIMENTAL RESULTS OF CO_2 REMOVAL WITH AMP/PZ SOLVENT

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This paper provides a discussion concerning results of CO_2 removal from a gas mixture by the application of aqueous solutions of ethanoloamine (MEA) and 2-amino-2-methyl-1-propanol (AMP) promoted with piperazine (PZ). The studies were conducted using a process development unit. Research of such a scale provides far more reliable representation of the actual industrial process than modelling and laboratory tests. The studies comprised comparative analyses entailing identical energy supplied to a reboiler as well as tests conducted at similar process efficiencies for both solvents. The results thus obtained imply that using AMP/PZ enables reduction of the solvent heat duty. Moreover, while using AMP/PZ temperature decrease was also observed in the columns.

Keywords: CO_2 capture, MEA, AMP, PZ

1. INTRODUCTION

CO_2 removal by the absorption method using amines seems to be the simplest solution to apply in coal-fired power plants (Dreszer and Więcław-Solny, 2008). Unlike other CO_2 emission reduction methods, such as oxy-fuel combustion, the amine-based process of CO_2 removal may be applied while erecting new power units as well as in existing power generation facilities. The possibility to deploy a CO_2 removal section in the existing facilities is actually one of the most significant factors taken into account while making the choice of technology. It allows for the process to be implemented without the need for considerable structural modifications in the power plant's technological line. The capability of using any of the technologies currently offered by the chemical industry (Chen et al., 2013; Farla et al., 1995) is limited due to characteristic flue gas parameters, such as low flue gas pressure, low partial pressure of CO_2 present in flue gas released from typical coal-fired units or high moisture content in the flue gas.

In the amine-based process of removing acidic components from gas mixtures, ethanolamine (MEA) has been used for years (Barchas and Davis, 1992; Sander and Mariz, 1992). It is characterised by a high reaction rate and a high CO_2 absorption capacity on low partial CO_2 pressures (Wilk et al., 2013b). Moreover, ethanolamine is a relatively cheap raw material As far as the disadvantages of ethanolamine are concerned, one should mention high heat of CO_2 absorption, which affects the energy that must be delivered to regenerate the solvent (Kim and Svendsen, 2007). It is also susceptible to considerable degradation, both thermal (Davis and Rochelle, 2009) and oxidation (Fredriksen and Jens, 2013) induced, due to the oxygen content in flue gas. The aqueous solution of ethanolamine shows corrosive properties, particularly at high temperatures and on high CO_2 saturation (Kittel et al., 2009). Hence the

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[1] PDU – Process Development Unit

search for amines characterised by absorption capacity and reaction rate comparable to MEA, but not displaying negative features of MEA. One of the available options in the search for efficient solvents is using mixtures of amines of diversified properties. Primary and secondary amines are known for high reaction rate (Vaidya and Kenig, 2007), Tertiary amines, such as N-methyldiethanolamine (MDEA) display lower reaction energy compared to lower amines (Kim and Svendsen, 2007; Svensson et al., 2013). However, the CO_2 absorption process rate is far lower with tertiary amines (Wilk et al., 2013a). Similar properties can be observed in amines with steric hindrance, such as 2-amino-2-methyl-1-propanol (AMP). In the reaction of AMP and CO_2, transient carbamate is formed, whereas CO_2 mainly reacts to form carbonate and bicarbonate ions. AMP is characterised by low energy demand for regeneration compared to MEA (Wilk et al., 2014; Xie et al., 2013). In order to increase the reaction rate of amines with steric hindrance and tertiary amines, primary and secondary amines are used as activators. Such a mixture retains high absorption capacity, but its reaction rate increases considerably. The amine used to activate both MDEA and AMP is piperazine (PZ). It is a heterocyclic amine featuring a ring of two secondary amine groups. Aqueous mixtures of AMP/PZ and MDEA PZ are characterised by a higher reaction rate than solutions not containing PZ (Wilk et al., 2013b). It is also possible to use mixtures of different amines and water-soluble organic fluids. Owing to the lower content of water, such mixtures are characterised by lower specific heat, which makes it possible to reduce the energy consumption involved in the solvent regeneration process.

2. PLANT CHARACTERISTICS

The process development unit (PDU) used to study the process of CO_2 removal by means of amines is one of three units of this type used by the Institute for Chemical Processing of Coal. It has been installed at the Centre for Clean Coal Technologies in Zabrze (Lajnert and Latkowska, 2013). Besides that, the Institute also uses a laboratory-scale installation (Krótki et al., 2012) and mobile pilot plant (Więcław-Solny et al., 2014) for post-combustion carbon capture research.

The testing unit presented in this paper is a transition-scale unit. However, unlike other plants, it can be used to study CO_2 removal from flue gas, process gas and mixtures of technical gases. The testing unit overview and its schematic diagram are shown in Fig. 1.

The gas mixture is delivered to a pre-treatment scrubber where the gas becomes water saturated, and the mixture is cooled down and cleaned of all particulate. Then it flows through a solid adsorption bed where SO_x are removed. Once it is free of SO_x, the gas flows to an absorber where CO_2 absorption takes place by means of an aqueous amine solution. The system draws benefits from a split flow arrangement of regenerated solution streams (Szczypiński et al., 2013). It is for this modification that energy consumption of the regeneration process can be reduced. Semi-lean amine is delivered to the lower absorber section, whereas lean amine is healed to the column top. Purified gas flows through an additional packing layer at the top of the absorption column, where water condensate is fed from a separator behind the stripper. The purpose of this section is to wash out droplets of solvent carried together with a purified gas from the column and cool down the gas leaving the absorber at the same time. The semi-lean amine is collected from a mid-level tray and delivered to the absorber. The remaining part of the solvent flows off to the lower column section where it is regenerated in the regenerator reboiler heated by an electric heater. The lean amine solution is delivered to the absorber top. Streams of solvent are involved in mutual heat exchange in a system of plate heat exchangers. The composition of gas at the system inlet and outlet was monitored on-line by means of ULTRAMAT 23 gas analysers. The solvent was analysed using the KEM DA-640 densitometer and by titration. The amine content in the solvent was measured by means of the Dionex ion chromatograph by titration methods.

The most important parameters of the system have been collected in Table 1.

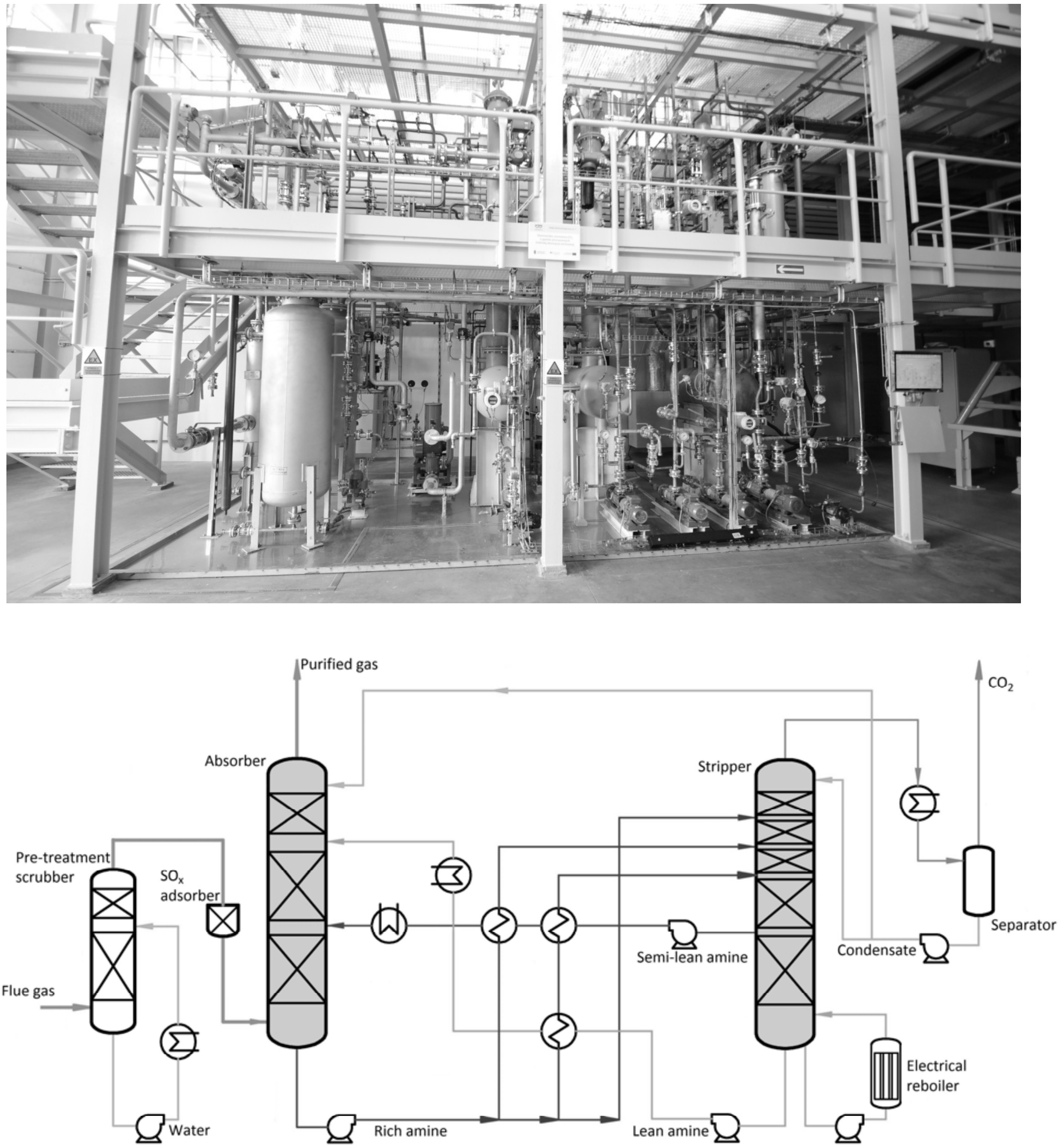

Fig. 1. Overview and process flow diagram of the testing unit

Table 1. Testing unit parameters

Parameter	Value	Unit
Nominal gas flow rate	100	[Nm^3/h]
Nominal solvent flow rate	0.515	[m^3/h]
Lean amine temperature	40	[°C]
Max. reboiler power	33	[kW]
Absorber pressure	130	[kPa]
Stripper pressure	145	[kPa]
Total absorber packing height	3.2	[m]
Absorber diameter	0.263	[m]

3. EXPERIMENTAL

The aim of this paper is to discuss energy consumption in the carbon capture process based on the experimental comparison of two different solvents. Laboratory scale tests of the carbon dioxide absorption alone are not sufficient to determine the behaviour of the solvent in industrial processes therefore it seems reasonable to test the solvent in PDU scale, being closer to a full-scale industrial process. Additionally, based on PDU scale results, it is possible to propose the solvent for a process scale-up, for example for a pilot plant. The results were compared with the literature sources. Heat duty for AMP/PZ solvent obtained during the research on our own laboratory rig and pilot plant were also presented. Furthermore, the temperature and CO_2 concentration profiles in the columns were also measured.

Under the experimental studies, comparative tests were conducted using the following aqueous solutions:

- 30% MEA
- 30% AMP 10% PZ

The ethanolamine solution was used as a reference for other solvents examined. In the course of the laboratory tests of the CO_2 absorption (Wilk et al., 2014), no formation of solid products of the CO_2 reaction with the amines present in the relevant solutions within the system's range of operating temperatures was observed. The Monoethanolamine (>98%), 2-amino-2-methyl-1-propanol (90%) and piperazine (>99%) were all purchased from Sigma-Aldrich. Aqueous solutions were prepared according to pre-defined concentrations. The gas examined was a mixture of CO_2 and N_2. Both gases were supplied by Messer. The gas composition was adjusted to match the average CO_2 concentration similar to the flue gas studied at the Tauron's pilot plant. The plant features more than 100 measurement points (temperature, pressure, fluid level, flow rate). Figure 2 below illustrates fluid and gas sample taking points as well as the arrangement of temperature measurement points within the absorption column.

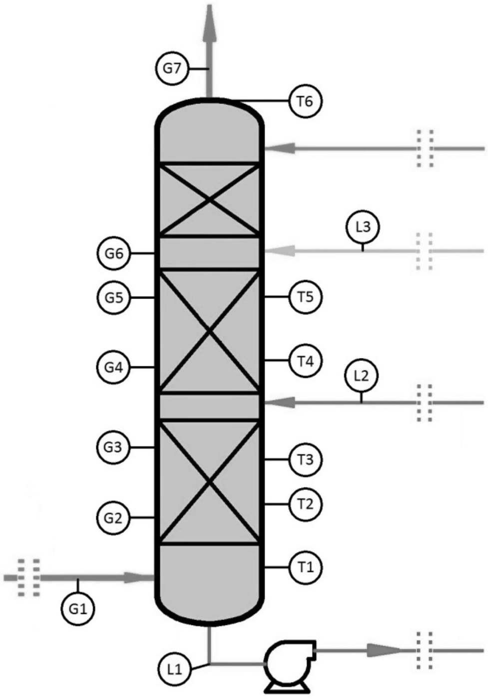

Fig. 2. Arrangement of measurement points

The tests were conducted in a steady state for several hours. Liquid and gaseous samples were collected from individual analysis points. The MEA tests were performed on a higher reboiler power in order to attain the efficiency of ca. 90%. The AMP/PZ tests were carried out under identical conditions and on a lower reboiler power, so that the efficiency of ca. 90% could be attained. The parameters describing the conducted tests have been collated in Table 2. Gas flow rates, values of pressure in the apparatus as well as temperatures of the solvent delivered to the absorber were constant for all the tests.

Table 2. Process conditions for trials

Test	Solvent	Gas flow rate [Nm^3/h]	CO_2 [% vol.]	Lean amine flow rate [m^3/h]	Semi-lean amine flow rate [m^3/h]	Reboiler power [kW]
1	MEA	99.8	12.27	0.255	0.260	33
2	MEA	99.4	12.27	0.306	0.310	33
3	AMP/PZ	99.7	12.38	0.306	0.310	33
4	AMP/PZ	98.9	12.33	0.254	0.260	33
5	AMP/PZ	99.9	12.32	0.254	0.260	24.75
6	AMP/PZ	100.5	12.30	0.304	0.315	24.75

4. RESULTS

The tests have made it possible to compare values of efficiency and reboiler heat duty in the regeneration process for different solvents. For the power of 33 W, the efficiency values recorded for AMP/PZ significantly exceed the level of 90%. When MEA is used, the efficiency values are about 90%. For both solvents, slightly higher process efficiency is attained at an increased solvent flow rate. Due to the decreased desorption time while raising the solvent flow rate, the quality of solvent regeneration drops. Tests conducted at the solvent flow rate of 0.615 m^3/h are characterised by higher loading of rich and lean amine, which is typical of both solvent types. The loading for AMP/PZ is lower compared to MEA. Since the heat absorption of AMP/PZ is lower than of MEA, AMP/PZ regenerates better when the same energy is delivered during regeneration. Moreover, the CO_2 absorption rate decreases as the loading rises. This fact triggers the increased rate of the CO_2 absorption process with AMP/PZ compared to MEA. Table 4 provides a summary of partial CO_2 pressure data and temperatures inside the absorber and the stripper.

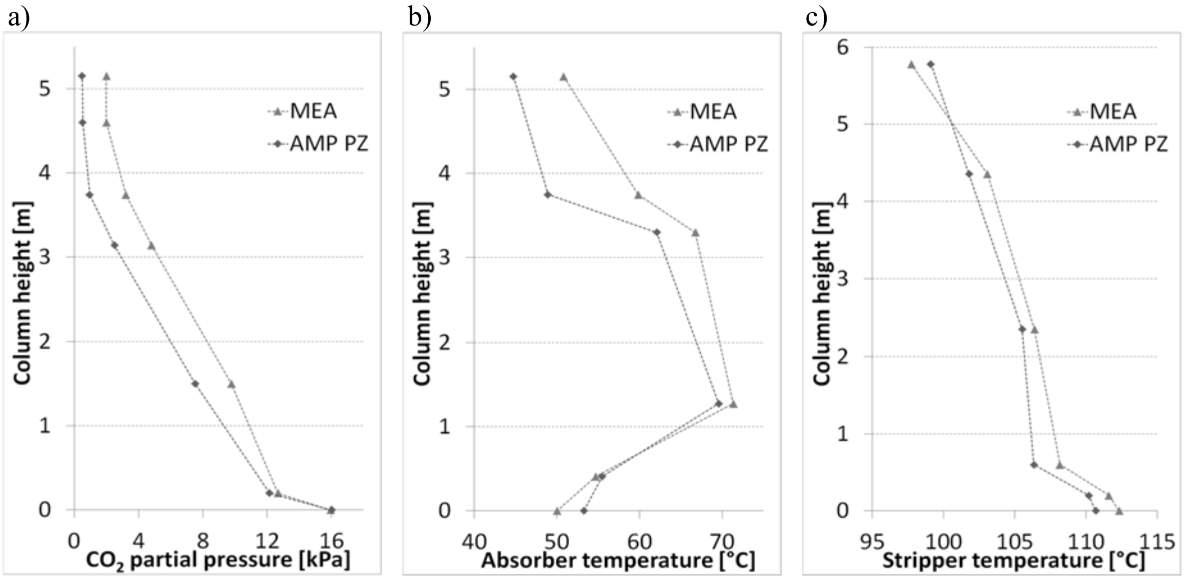

Fig. 3. Distribution of CO_2 partial pressure (a), absorber temperature (b) and stripper temperature (c) for MEA and AMP/PZ at reboiler power 33kW

Having measured the partial CO_2 pressure profiles and the temperatures inside the absorber and the stripper (Figs. 3 a, b, c and Tables 3, 4, 5), one can observe that the partial CO_2 pressure is lower at each point for AMP/PZ. The stripper temperature is also slightly lower, whereas the most significant differences can be observed in the absorber. On the account of lower heat of absorption, a smaller amount of heat is released for AMP/PZ. For both MEA and AMP/PZ, the highest temperature occurs at the lower absorber section. It may imply that it is the point at which the absorption process rate is the highest, since the relatively high partial CO_2 pressure affects the absorption process driving factor.

Table 3. Measured values of CO_2 partial pressure for all the tests

	CO_2 partial pressure [kPa]					
Column height [m]	1	2	3	4	5	6
0.00	15.95	15.96	16.09	16.02	16.01	16.00
0.20	12.68	13.55	10.67	12.14	13.69	11.79
1.50	9.76	9.19	6.21	7.51	10.49	10.70
3.14	4.81	4.38	2.04	2.51	5.02	5.90
3.74	3.21	2.91	0.53	0.96	2.99	2.85
4.60	2.00	1.87	0.22	0.53	2.02	2.07
5.15	1.99	1.83	0.19	0.49	1.97	1.97

Table 4. Measured values of absorber temperatures for all the tests

		Absorber temperature [°C]					
Column height [m]	Measured phase	1	2	3	4	5	6
0.00	liquid phase	50.0	52.1	54.2	53.3	52.0	50.1
0.41	liquid phase	54.7	56.7	56.5	55.5	53.3	52.8
1.28	liquid phase	71.4	59.4	52.6	69.6	62.1	54.6
3.30	liquid phase	66.8	53.9	44.5	62.1	64.5	58.5
3.75	liquid phase	59.9	45.2	42.1	48.9	55.3	46.7
5.15	vapour phase	50.8	39.7	37.2	44.8	49.8	46.1

A similar correlation between the maximum temperature and maximum rate of absorption process has been discussed in the literature (Asendrych et al., 2013; Niegodajew et al., 2013). Reboiler heat duty in the solvent regeneration process is higher when MEA is used. On account of the identical power supplied to the reboiler, it only depends on the amount of CO_2 removed. Reboiler heat duty is a correlation between the energy supplied to the process and the mass of CO_2 removed. Reboiler heat duty values provided entail the losses of heat released to the environment.

Table 5. Measured values of stripper temperatures for all the tests

		Stripper temperature [°C]					
Column height [m]	Measured phase	1	2	3	4	5	6
0.00	liquid phase	112.4	112.1	110.4	110.7	110.0	107.3
0.20	liquid phase	111.6	111.3	110.1	110.2	108.1	107.2
0.60	liquid phase	108.2	107.2	106.3	106.3	99.2	97.1
2.35	liquid phase	106.4	105.7	103.8	105.5	95.8	92.8
4.35	liquid phase	103.1	102.6	100.7	101.8	92.4	90.8
5.78	vapour phase	97.7	99.0	97.6	99.1	84.2	82.2

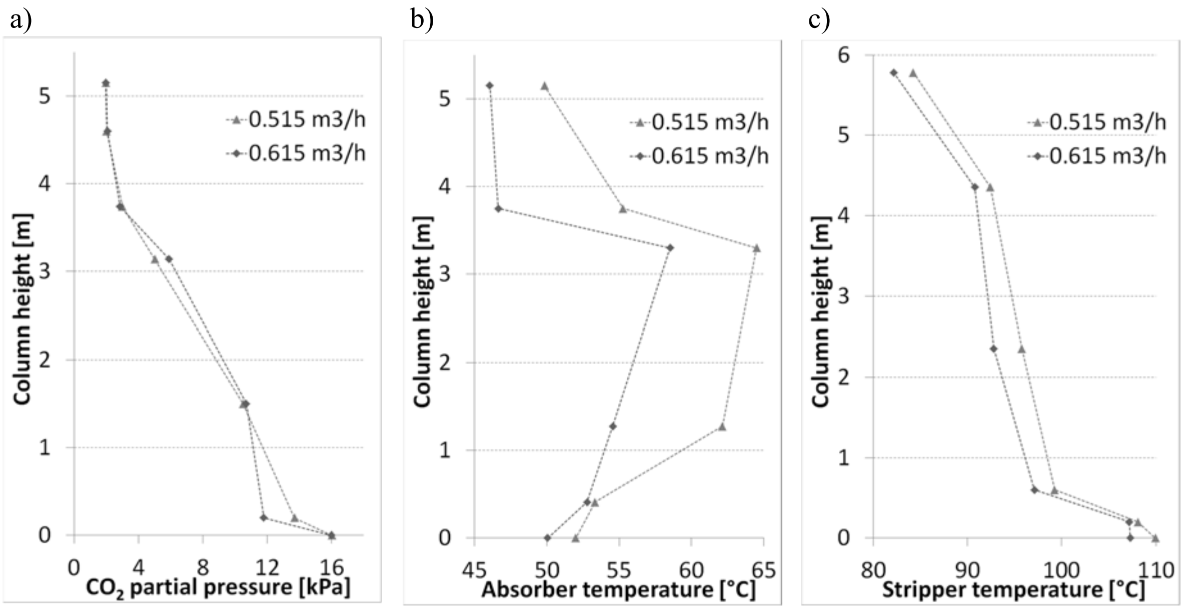

Fig. 4. Distribution of CO_2 partial pressure (a), absorber temperature (b) and stripper temperature (c) for AMP/PZ at different solvent flow rate

Further tests using AMP/PZ were aimed at comparing the results obtained for solvents at a similar value of CO_2 removal process efficiency. The power delivered to the regenerator was reduced to a level enabling process efficiency of ca. 90% to be obtained. Based on the data thus acquired, one could observe an increase loading amine for the tests run on reduced power. The process rate attained when using a regenerated solution of higher loading is lower, and hence the decrease in the process efficiency. Insofar as the loading of the lean amine did not increase much, the semi-lean amine loading was considerable, approaching the value typical for rich amine. The temperature profile (Fig. 4) implies that the maximum absorber temperature point shifted up the column for both solvent flow rates which was probably due to moving the area of the highest process rate. And as in the previous case, the major impact is exerted by partial CO_2 pressure, being higher along the entire height of the column for lower power capacities.

Furthermore, a solution of a relatively high loading is supplied to the lower column section, causing the process driving force to be low in this part of the column. The CO_2 removal efficiencies are similar for both solvent flow rates. When comparing Figs. 4b and 4c one can notice that the temperatures of the absorber and of the stripper decrease at the increasing solvent flow rate.

Comparing partial pressure and temperature distributions (Fig. 5) for tests with similar efficiency for MEA and AMP/PZ, one may observe significant differences in temperature profiles. Temperatures in columns are far lower when AMP/PZ is used. The maximum temperature difference between both solvents comes to ca. 10°C. A similar temperature difference occurs at the stripper top. The higher the mixture temperature leaving the stripper, the larger the quantity of cooling water needed to effectively cool it. Moreover, the partial pressure of steam increases as temperature rises, which triggers an increase of the quantity of water condensing in the separator. Having analysed the loading values, one can certainly notice that loading values of rich and of the semi-lean one are rather similar for both solvents. However, the loading of the lean amine is lower for AMP/PZ.

Lean amine loading increases insignificantly as the solvent amine flow rate increase.

The reboiler heat duty for tests involving smaller power is much lower in comparison with both AMP/PZ tests for larger powers as well as for MEA. Compared to MEA, the reboiler heat duty was dropped by more than 25% while similar efficiency was maintained.

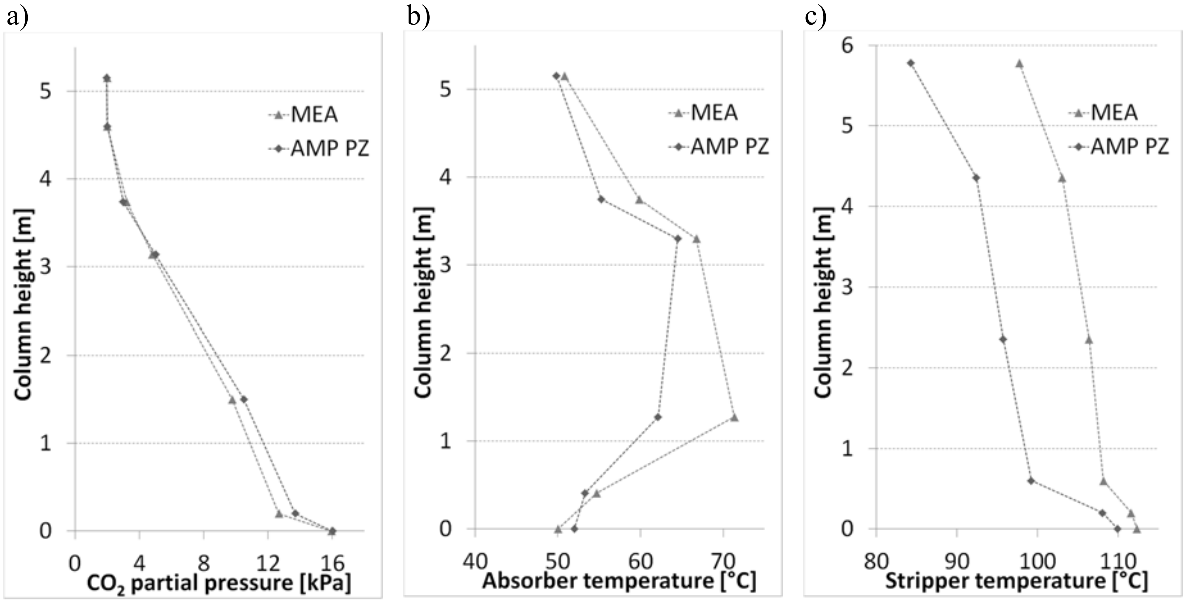

Fig. 5. Distribution of CO_2 partial pressure (a), absorber temperature (b) and stripper temperature (c) for MEA and AMP/PZ at similar process efficiency

Table 6. Efficiency, reboiler heat duty and CO_2 loading for all the tests

Test	Efficiency [%]	Reboiler heat duty [MJ/kg CO_2]	L/G ratio [kg/kg]	Rich amine loading [mol/mol]	Lean amine loading [mol/mol]	Semi-lean amine loading [mol/mol]
1	88.6	5.21	4.33	0.43	0.21	0.30
2	89.5	5.12	5.12	0.40	0.22	0.31
3	98.9	4.80	5.08	0.27	0.05	0.16
4	96.9	4.88	4.25	0.21	0.04	0.10
5	90.6	3.69	4.17	0.38	0.09	0.29
6	88.8	3.81	5.15	0.38	0.12	0.33

Table 7 contains a comparison of the results with literature data for AMP/PZ aqueous solvents. It can be seen that the reboiler heat duty obtained during the tests is comparable to that for CESAR1 solvent (Mangalapally and Hasse, 2011). This similarity can be partly explained by a comparable concentration of the CESAR1 solvent, which is 45 wt% (28 wt% AMP and 17 wt% PZ). The concentration of the second solvent used in this comparison is 30 wt% (25 wt% AMP and 5 wt% PZ) (Artanto et al., 2014; and Table 7).

Table 7. Comparison of the efficiencies and reboiler heat duties with literature data

Source	Gas flow rate [Nm^3/h]	L/G ratio	Efficiency [%]	Reboiler heat duty [MJ/kg CO_2]
Mangalapally and Hasse (2011)	≈ 66	1.4 [kg/kg]	90	3.3
Artanto et al. (2014)	100	5.6 [$m^3/1000m^3$]	85	4.9
This paper	100	4.17 [kg/kg]	90.6	3.69

From our research it can be seen that an increase of the scale of the plant causes a decrease in the heat duty of the reboiler (Table 8). The lowest reboiler heat duty can be observed for pilot plant tests where similar removal efficiency was achieved for all units. This fact is related to the increase of efficiency of heat exchangers while increasing the scale and the decrease of overall heat losses to ambient. The laboratory unit works in standard process flow sheet, while other plants use modified, advanced process flow sheets allowing the reduction of energy consumption.

Table 8. Scaling up effects of the efficiencies and reboiler heat duties

Unit type	Gas flow rate [Nm^3/h]	L/G ratio [kg/kg]	Efficiency [%]	Reboiler heat duty [MJ/kg CO_2]
Laboratory unit	5	8.15	89.7	4.84
PDU	100	4.17	90.6	3.69
Pilot plant	200	3.92	89.9	3.13

5. CONCLUSION

A comparison of test results obtained for both solvents studied implies a considerable drop in the reboiler heat duty on similar process efficiency attained when using AMP/PZ. Furthermore, the temperatures recorded for AMP/PZ both in the absorber and the regenerator were lower than those when MEA was applied, even for identical energy supplied to the reboiler. Increasing the L/G ratio causes a slight efficiency increase for both solvents in the case when power of 33 kW is applied. However, as regards AMP/PZ, the efficiency slightly declines for decreased power. Increasing the L/G ratio also triggers an increase in the solvent loading value and a drop of temperatures in both columns.

The results presented in this paper were obtained during research co-financed by the National Centre of Research and Development in the framework of Contract SP/E/1/67484/10 – Strategic Research Programme – Advanced technologies for energy generation: Development of a technology for highly efficient zero-emission coal-fired power units integrated with CO_2 capture.

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Received 16 September 2014
Received in revised form 19 January 2053
Accepted 28 January 2015