

Solvent selection for CO₂ capture from gases with high carbon dioxide concentration

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Abstract—Amine absorption processes are widely used to purify both refinery and process gases and natural gas. Recently, amine absorption has also been considered for application to CO₂ removal from flue gases. It has a number of advantages, but there is one major disadvantage—high energy consumption. This can be solved by using an appropriate solvent. From a group of several dozen solutions, seven amine solvents based on primary amine, tertiary amine and sterically hindered amine were selected. For the selected solutions research was conducted on CO₂ absorption capacity, an absorption rate and finally a solvent vapor pressure. Furthermore, tests on an absorber-desorber system were also performed. In this study the most appropriate solvent for capturing CO₂ from flue gases with higher carbon dioxide concentrations was selected.

Keywords: CO₂, Absorption, MEA, AMP, Piperazine, CCS

INTRODUCTION

The carbon dioxide concentration in the Earth's atmosphere has oscillated approximately 280 ppm within more than 10 thousand years. However, since the second half of the 19-th century, a significant rise in the carbon dioxide concentration in the atmosphere at the rate of 1.2 ppm per year has been observed. Today, the average global atmospheric concentration of carbon dioxide equals approximately of 380 ppm [1]. The analyses conducted on this topic by the Intergovernmental Panel on Climate Change prove, among others, that human activity contributes significantly to the increase in the CO₂ concentration in the atmosphere. As a consequence, this anthropogenic carbon dioxide leads to climate change [2-4]. Remaining at the current level of fossil fuels utilization will cause an increase of climate change [5]. 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. Main source of carbon dioxide emission is the power industry. How-

ever, there are additional large sources of greenhouse gases emissions and they are industry branches such as cement or iron and steel industry where they emit a significantly higher amount of CO₂ in flue gases [6]. These industries include also quicklime and glass productions. The typical carbon dioxide concentration in the different flue gases is listed in Table 1.

One of the solutions to reduce the large carbon dioxide emissions, that are occurring for example during fossil fuel combustion, may be an implementation of post-combustion technologies. A huge number of post-combustion technologies for carbon dioxide removal exist in the industry, and they include the following processes: physical and chemical absorption, adsorption [14], membrane processes [15], cryogenic and electrochemical methods [2]. The chemical absorption is both the most developed and commonly used method for CO₂ removal from large gaseous streams. Furthermore, the most suitable chemical absorption process for carbon dioxide removal from large gas streams that is close to atmospheric pressure is an amine absorption [1]. The process has a number of advantages and only one major drawback: high energy consumption. However, it can be minimized, either by optimizing the plant operation parameters, where up to 10% energy consumption reduction [16] can be achieved or by using an appropriate solvent leading to up to 30% savings [17,18]. The selection of the process solution is determined by both the pressure and temperature conditions at which the flue gas is available. Furthermore, it depends on gas composition with respect to the major and minor components, and also on the purity requirements for the treated gas [19].

Carbon dioxide capture is often carried by application of various amines types, including, the most widely used monoethanolamine (MEA). MEA is widely described in literature [19-22] and applied in many processes, for example in Econamine FG, or Kerr-McGee/ABB Lummus Crest process [23]. It is characterized by a high absorption rate and a relatively low price. However, MEA has some disadvantages, such as a high heat of desorption, a high sus-

Table 1. Typical carbon dioxide concentration in different industrial flue gases

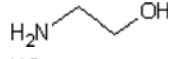
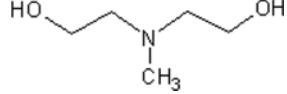
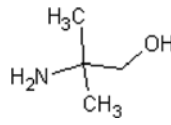
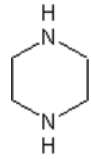
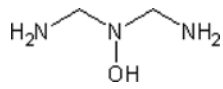
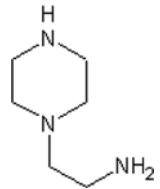
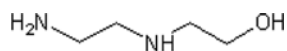
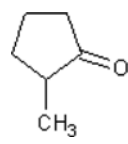
Type of industry	Typical CO ₂ concentration in flue gases [%]	Ref.
Power generation	9-13	[7]
Iron, steel	20-40	[8]
Glass	35-45	[9]
Cement	14-33	[10]
Quicklime	20-28	[11-13]

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Table 2. Amines and physical solvents used in the screening test

No.	Name	CAS number	Structural formula
1	Ethanolamine (MEA)	141-43-5	
2	N-methyldiethanolamine (MDEA)	105-59-9	
3	2-Amino-2-methyl-1-propanol (AMP)	124-68-5	
4	Piperazine (PZ)	110-85-0	
5	1,3-Diamino-2-propanol (DAP)	616-29-5	
6	1-(2-Aminoethylo)piperazine (AEP)	140-31-8	
7	N-(2-hydroxyethyl)ethylenediamine (AEEA)	11-41-1	
8	N-methylpyrrolidone (NMP)	872-50-4	

ceptibility to degradation etc. [24-27]. For this reason, other more complex amines are recently often used in the absorption process, for example, tertiary amines such as MDEA used in the BASF aMDEA process [28,29], or amines with steric hindrance for the KM-CDR Process [30-32], or polyamines etc. There are processes using solutions based on blends of physical and chemical solvents for carbon dioxide removal (e.g., Sulfinol, Amisol) [19]. However, there is little original research on mixed sorbents [33,34]. In recent years a number of studies have been conducted, including mainly synthetic sterically hindered amines [35,36], tertiary amines [37,38], and polyamines [37,39]. However, some basic research is mainly conducted and usually there is no verification of absorbent effectiveness in larger scale in the real process.

As a part of the conducted studies, a number of solutions were tested, including a range from 30% reference solution of MEA up to the systems based on amines with steric hindrance and mixed solvents. Among others, the tests included the determination of following impacts:

- solvent vapor pressure,
- absorption rate and absorption capacity,
- CO₂ removal efficiency and reboiler heat duty.

The study included both the basic absorption parameters determination and verification of solvents absorption efficiency in real

absorption-desorption systems.

MATERIALS AND METHODS

Screening tests were conducted on several solutions based on primary, secondary, tertiary and sterically hindered amines. Physical carbon dioxide absorbents as additive to amine solution in multicomponent solvent were also used. All solvents components are listed in Table 2. All concentrated amines were obtained from *Sigma-Aldrich*. Measurement setup was filled with utilization of carbon dioxide (with purity 4.5 *Linde AG*).

For process studies the gas mixture was prepared from ambient air and carbon dioxide (purity 4.0, *Linde AG*). Carbon dioxide in gas composition oscillated at 30 vol% level. To simplify the experiment, the ambient air instead of nitrogen was used. This simplify brought the experiment to the more real conditions. Due to the relatively short time of the tests, the solvent oxidative degradation was negligible.

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

zine. Both reagents - 2-amino-2-methyl-1-propanol and piperazine were obtained from *Sigma-Aldrich*.

- Multicomponent Solution 2 - developed at the Institute for Chemical Processing of Coal, containing 15 wt% of primary amine, 20 wt% of AMP, 2 wt% of activator, 63 wt% of organic solution and water [40].

- 30 wt% N-methyldiethanolamine with 10 wt% piperazine. MDEA was obtained from *Brenntag* while PZ was obtained from *Sigma-Aldrich*.

- 30 wt% N-(2-hydroxyethyl)ethylenediamine. Concentrated AEEA was obtained from *Sigma-Aldrich*.

Distilled water was used during solvent preparation.

EXPERIMENTAL

1. Laboratory Setup

Basic properties (absorption capacity, absorption rate, vapor pressure) of potential solvents were determined in a laboratory stand. The stands were designed for studying the equilibrium and kinetics of CO₂ absorption in mixtures of amines. They comprise a thermally isolated glass reactor, of a thermostat for maintaining a constant temperature, and of a hybrid vacuum pump for removal of gases from the system. The setup also includes a system for precise metering of liquid samples, and for measuring and recording the pressure in the system. The stand was used for studying the kinetics of absorption, determining absorption isotherms and additionally for determining vapor pressures of amine mixtures. A scheme of the stand together with its picture is shown in Fig. 1.

Prior to investigation of CO₂ absorption kinetics, the system was

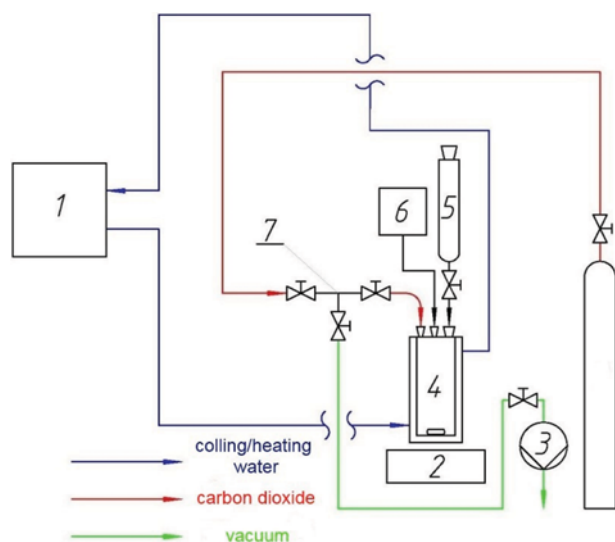


Fig. 1. Schematic diagram of the laboratory setup for studying absorption equilibria and kinetics of CO₂ absorption in mixtures of amines.

- | | |
|---|-----------------------------|
| 1. Thermostat | 6. Vacuum meter |
| 2. Magnetic stirrer | 7. Valves station |
| 3. Vacuum pump | 8. Data acquisition systems |
| 4. Thermostated reactor | |
| 5. Precise cylindrical separatory funnel with scale | |

alternately evacuated and several times filled with carbon dioxide to completely remove air. Afterwards, the reactor was filled with carbon dioxide to a given pressure. After the pressure and temperature in the system stabilization was reached, a single solvent sample was instilled. Upon introducing the solvent, the pressure changes versus time were measured. The adopted time interval enabled attaining a nearly equilibrium state of solution saturation, thereby permitting determination both the basic data, such as the number of moles of CO₂ absorbed in a unit of time, but also the data showing the relation between an absorption rate and a saturation ratio of the carbon dioxide solution.

For studying CO₂ absorption equilibria, several samples of liquid solution were injected into the reactor at certain time intervals. After each sample injection, and after some time required for establishing an equilibrium in the system, the pressure value was recorded and another sample was injected. The obtained data was used to determine the number of CO₂ moles absorbed in a unit of solution volume.

For continuous CO₂ removal from flue gas, a special laboratory unit was constructed. The basic elements of the setup were: absorber, stripper, and main heat exchanger. The columns were made from metal and glass, and were filled with glass Raschig rings of 6×6×0.5 mm (Raschig GmbH) with an option to replace it with another packing type [41]. Glass walls of columns enabled the observation of distributor liquid sprinkling and liquid flow distribution over the applied packing. The main heat exchanger was necessary to be used for exchange of heat between the hot lean solution and the cooler rich solution. The main heat exchanger was also used to reduce the overall stripper size. When necessary, the rich solution entering the absorber could be cooled in the final water cooler. The rich solution was additionally filtered through an activated carbon bed to remove solid contaminants and some amine degradation products. The heat needed for the CO₂ desorption from the solution was delivered to the system by using an adjustable electric heater. This solution is convenient under laboratory conditions, and it simplifies carrying out 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 incorrect, but still sufficient to allow the comparison of the different solvents.

The described laboratory setup was equipped with approximately 30 measuring points and controlled by a supervisory control and data acquisition system (SCADA). The setup had the option of controlling parameters of the gas and solvent solution by using *Intergaz* gas meters and *Tecluid M21* flowmeters. The temperature was controlled by *Limatherm PT-100* sensors and the pressure by pressure transducers from *Wika*. The amount of electricity to the stripper heater was regulated by *Aplisens PMS-200* electric current. The CO₂ volume fraction both at the inlet and outlet was measured with *Siemens Ultramat 23* gas analyzers. A schematic diagram and a picture of the laboratory setup to investigate the CO₂ removal process is presented in Fig. 2. According to the schematic diagram, air was compressed in an air blower to the discharge pressure of 130÷150 kPa_{abs}. After drying, air was directed to the gas mixer where the gas mixture was combined with car-

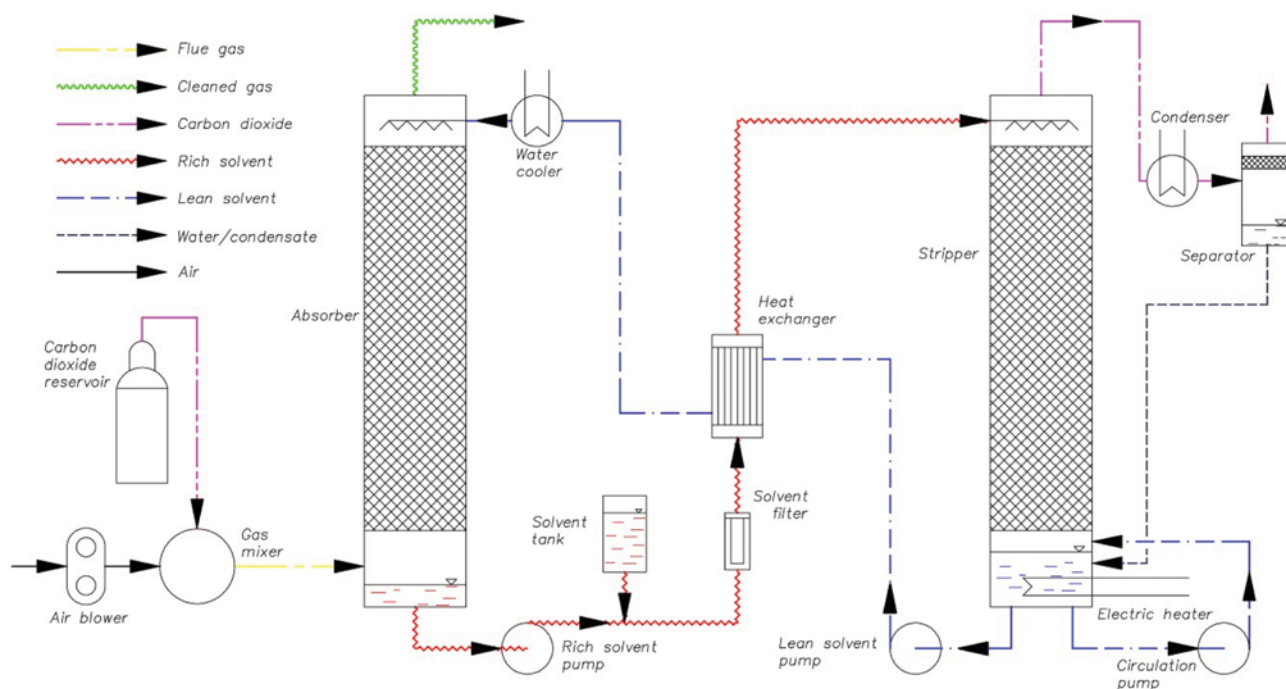


Fig. 2. Schematic diagram of the laboratory unit to investigate the CO₂ capture process [42].

bon dioxide. Next, the composed gas mixture was directed to the absorber where the CO₂ absorption process took place. Cleaned from CO₂ the gas was evacuated from the column through the gas meter and removed outside.

In the absorption column, the gas mixture came into contact with the lean amine solution from the stripper in a counter-current manner. The rich solution with the absorbed carbon dioxide was 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 took place. In next step, the lean solution was pumped back to the absorber top from the bottom of the stripper. Losses of water/sorbent were periodically supplemented from the solvent tank.

RESULTS AND DISCUSSION

1. Solvent Screening

In the screening test several dozen of solvents based on different amine types were tested. For comparison purposes, seven promising solvents based on primary amine (MEA, as a reference solvent), polyamines (AEEA, AEP, DAP) and amine blends (activated MDEA, activated AMP, multicomponent solution) were chosen. The compositions of the used solutions were:

1. 30 wt% monoethanolamine,
2. 30 wt% N-methyldiethanolamine and 10 wt% piperazine,
3. 30 wt% 2-amino-2-methyl-1-propanol and 10 wt% piperazine,
4. 30 wt% N-(2-hydroxyethyl)ethylenediamine,
5. 30 wt% 1-(2-aminoethoxy)piperazine,
6. 30 wt% 1,3-diamino-2-propanol,
7. 37 wt% amine blend and 63 wt% organic liquid with water.

The conducted research included determining the vapor pres-

sure, carbon dioxide absorption equilibria, and carbon dioxide absorption rate tests.

1-1. Vapor Pressure

Basic parameter, important for further calculations, was the vapor pressure. It was also essential due to the solvent losses in absorption-desorption process. Amine volatility losses are usually not significant. Main alkanolamines have very low vapor pressures at the typical amine absorber and regenerator operating conditions. However in some cases, a water wash section is provided for low pressure absorbers [43]. Low solvent vapor pressure allows to gain pure carbon dioxide stream and tail gas with only amine traces. That means lower cost of gas cleaning before it further use.

Vapor pressures of examined absorbents were determined and compared for two absorption temperatures: 30 and 50 °C. The results of the conducted tests are shown in Fig. 3.

The lowest vapor pressures were obtained in the case of the multicomponent solvent and in activated 2-amino-2-methyl-1-propanol. The possible reason for the observed pressure of multicomponent solvent was partial replacement of water in a solution by an organic liquid characterized by low volatility. At 20 °C temperature both solvents had vapor pressure lower than that for the other tested solutions for approximately 25-35%. With an increasing temperature the difference between vapor pressure of multicomponent solvent and activated AMP increased. At 50 °C, activated AMP vapor pressure was lower for approximately 10-15% compared to other solvents, while for multicomponent solvent it was lower for approximately 25-30%.

1-2. Absorption Capacity

The main parameter of amine solvents is absorption capacity, as it shows how much of carbon dioxide can be absorbed by the solvent per unit of volume. The higher the absorption capacity of

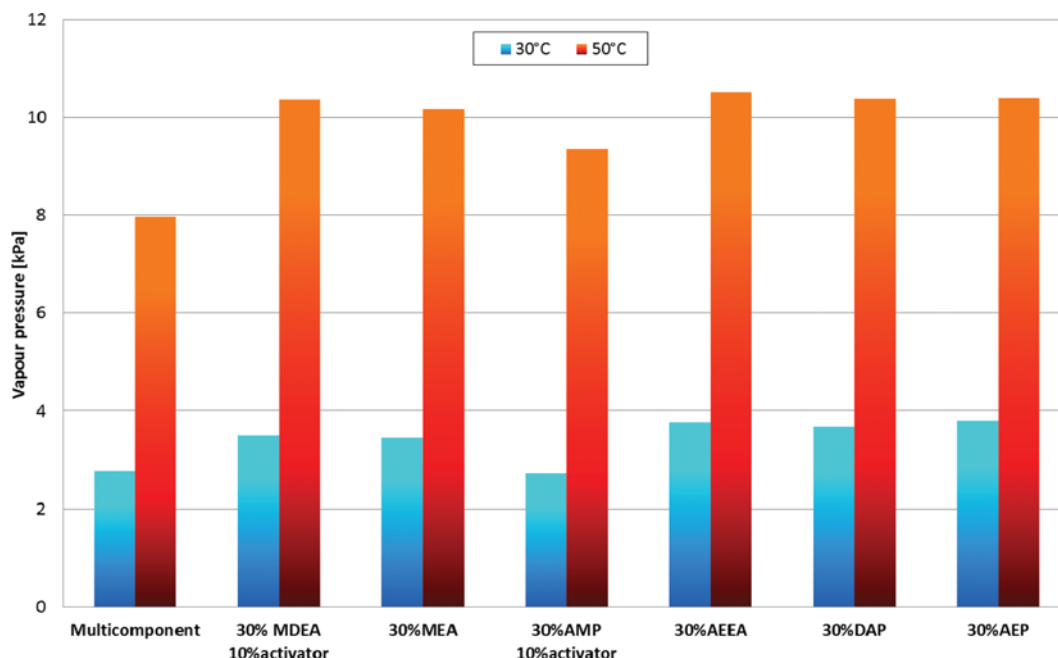


Fig. 3. Comparison of different amine solvents vapor pressure at 30 and 50 °C.

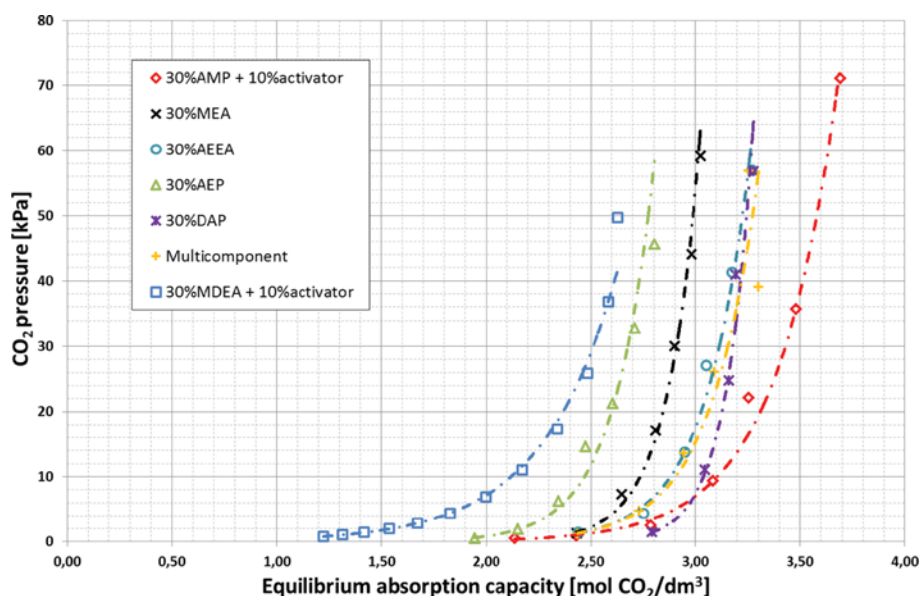


Fig. 4. Comparison of amine solvents equilibrium absorption capacity at 30 °C.

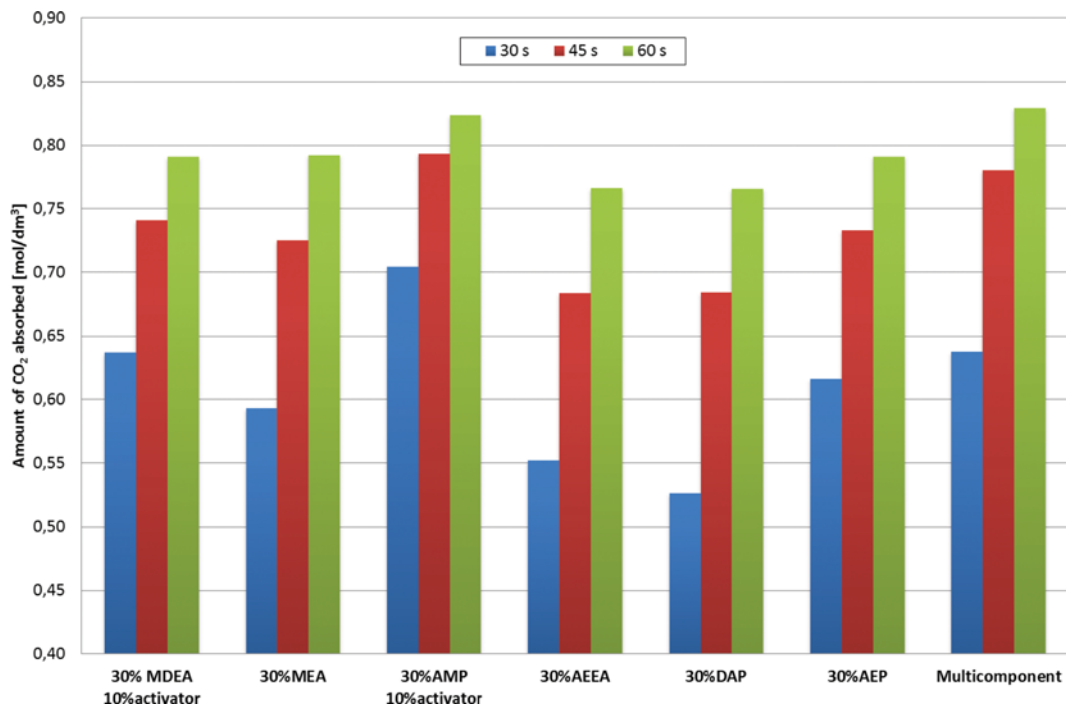
solution, the less volume of solvent has to contact with the flue gas to remove a predetermined amount of CO₂. This parameter also determines the necessary solvent flow rate, which results in absorption column dimensions.

Carbon dioxide absorption equilibrium determination tests were conducted at 30 °C and for CO₂ partial pressure from 0 to 50 kPa. Several equilibrium points were determined for every solvent. On the basis of these points the exponential dependence was determined, so-called absorption isotherms. It is presented with dashed lines in Fig. 4. By comparison of the obtained data, the best capacity parameters had an activated AMP solvent. For carbon dioxide

partial pressure over 10 kPa, it had an absorption capacity significantly higher than 3 mole CO₂ per dm³. Relatively high values of absorption capacity (in terms of the CO₂ partial pressure range 10-20 kPa) also show a solution of a primary diamine, i.e., 30% of DAP. Comparable absorption capacity to DAP solution at higher CO₂ concentrations shows 30% AEEA solution and multicomponent solution. For lower CO₂ concentrations the quantities of carbon dioxide absorbed in AEEA were lower than for DAP and activated AMP solutions. Solvents based on AEP and activated MDEA showed the lowest absorption capacity of all tested solutions. Reference solution that was 30% MEA was characterized by

Table 3. Comparison of absorption capacities for different carbon dioxide partial pressures

No.	Solvent	Absorption capacity [mole/dm ³]		
		p _{CO₂} =10 kPa	p _{CO₂} =20 kPa	p _{CO₂} =30 kPa
1	30% MEA	2.75	2.85	2.90
2	30% MDEA+activator	2.10	2.35	2.50
3	30% AMP+activator	3.10	3.30	3.45
4	30% AEEA	2.85	3.00	3.10
5	30% DAP	3.05	3.15	3.20
6	30% AEP	2.45	2.60	2.70
7	Multicomponent	2.90	3.05	3.15

**Fig. 5. Comparison of the absorbed CO₂ amounts for different gas-liquid contact time condition.**

an intermediate capacity between value for the solutions with high capacity (activated AMP, 30% AEEA, multicomponent solvent and 30% DAP) and value for solutions with a low absorption capacity (activated MDEA and 30% AEP).

Based on absorption isotherms, the equilibrium absorption capacity of the solution at CO₂ removal from gases with a specific concentration of CO₂ can be calculated. The comparison of solvents equilibrium absorption capacities for carbon dioxide capture from the gases (at atmospheric pressure), that contain approximately 10, 20 and 30% of CO₂ is shown in Table 3.

AMP solution showed an absorption capacity value for about 5-40% higher than the value for other solvents for the gases that contained 20% of CO₂. Besides, it was approximately 8-40% higher for a gas with 30% CO₂. In comparison to the reference solution, i.e., 30% MEA, the AMP solvent had a capacity higher for 16 and 19%, respectively.

1-3. Absorption Rate

The third important parameter, the absorption rate, determines

the necessary gas-liquid contact time, and thus the height of the absorber. Therefore, research on CO₂ absorption rate in amines solutions was conducted. CO₂ absorption rate was tested under the same conditions such as stirring, pressure, temperature, amount of solution, for each solvent. Results for all solvents, showing the amount of CO₂ absorbed when using different contact times at 30 °C can be seen in Fig. 5. Only AEEA and DAP solutions have absorption rate lower than that observed for 30% MEA solution. Other solvents are characterized by high and similar absorption rates. The highest value of the absorption rate had both the activated AMP and multicomponent solutions. In the first case high absorption rate is a result of piperazine addition, which is characterized by a very high absorption rate constant equal to 59,000 L·mol⁻¹·s⁻¹, while for MEA it is only 6,000 L·mol⁻¹·s⁻¹ [29]. For multicomponent solvent high absorption rate is a result of limited amount of water in the solution, which favors the fast CO₂ reaction with the amine. Since, it leads to indicating carbamate formation [40].

Table 4. Process conditions for laboratory unit tests

Parameter	Value	Unit
Gas flow rate	3.0±0.1	m _n ³ /h
CO ₂ content	30±1	% vol
Absorption temperature	40±1	°C
Absorber pressure	1.2	bar _a
Stripper pressure	1.02	bar _a
Liquid/solvent flow rate	50±1	dm ³ /h
Heater power set	2000	W

2. Absorption-desorption Tests

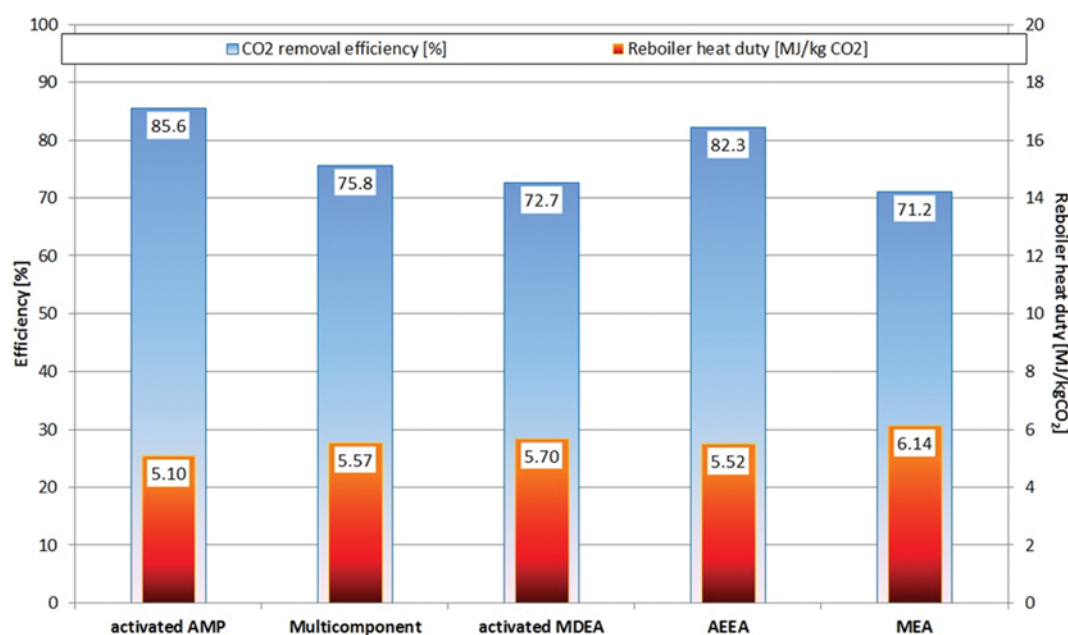
Another crucial thing is to specify other solution parameters: CO₂ absorption heat, specific heat, viscosity, foaming, degradation, corrosivity, toxicity and of course solution price. For an improved solvent verification it is necessary to test the solvents in a real continuous absorption-desorption process. Because of that, in the study five solutions were chosen from previous realized tests: activated AMP, multicomponent, activated MDEA, MEA and AEEA. The DAP solution was excluded from further testing due to the high price, and the AEP solution due to its potential toxicity [44].

Tests on the absorption-desorption system for CO₂ removal from flue gases were conducted for each tested solvent under the same reaction conditions. The main process parameters applied in the test are shown in Table 4.

The obtained results showed that the chosen alternative to 30%

MEA solutions exhibited lower reboiler heat duty (per kg of CO₂ removed); that is shown in Fig. 6. For these solutions also higher carbon dioxide removal efficiency was observed. Activated AMP, multicomponent, activated MDEA and AEEA solutions were characterized by 17, 9, 7, and 10%, respectively, lower reboiler heat duty in the CO₂ removal process than that observed for 30% of the MEA. CO₂ removal efficiencies were, respectively, higher by 20, 6, 2 and finally 15% as shown in Fig. 6. The highest improvement in the efficiency and reboiler heat duty was observed for both the polyamine solution (AEEA) and the activated sterically hindered amine (AMP).

The obtained reboiler heat duty values are high due to the small-scale of laboratory stand. The reason for that is a relatively high percentage of heat loss in overall heat duty. This generates a problem when direct use of the obtained results for the industrial scale would be expected. However, it is still sufficient for the comparison of different solvents. To determine close to full scale energy demand, it is important to scale-up the process. Strategy for process scale-up adopted by IChPW research team is based on two major research plants. The process development unit (PDU, with a flue gas capacity up to 100 m³/h (1,013 hPa, 273 K), is installed at the Clean Coal Technology Centre in Zabrze, Poland. The plant enables evaluation of CO₂ capture from flue gas, process gas or mixtures of technical gases using different solvents. It is capable of processing flue gases and flammable gases from other units installed in this facility, being the largest Polish research center dedicated for the development of clean coal technologies [45]. An overview of

**Fig. 6. Carbon dioxide efficiency and reboiler heat duty for different solvents achieved in the same reaction conditions.****Table 5. Scaling up effects on L/G ratio and reboiler heat duty**

Unit type	Gas flow rate [m _n ³ /h]	L/G ratio [kg/kg]	Efficiency [%]	Reboiler heat duty [MJ/kg CO ₂]
Laboratory unit	3	12.7	85.6	5.10
PDU	70	5.2	85.2	3.32

Table 6. Comparison of results gained in screening and process tests

No.	Solvent	Screening tests			Process tests	
		Vapor pressure [kPa]	Absorption capacity [mole/dm ³]	Absorption rate	Efficiency [%]	Reboiler heat duty [MJ/kg CO ₂]
1	30% MEA	3.46	2.90	medium/high	71.2	6.14
2	Activated MDEA	3.51	2.50	medium/high	72.7	5.70
3	Activated AMP	2.73	3.45	high	85.6	5.10
4	30% AEEA	3.77	3.10	medium	82.3	5.52
5	30% DAP	3.69	3.20	medium	-	-
6	30% AEP	3.80	2.70	medium/high	-	-
7	Multicomponent	2.78	3.15	high	75.8	5.57

the PDU provided in the publication [22] presents the results of process scale-up for CO₂ capture from power plants flue gas. Industrial studies of the selected solvent are planned with the use of mobile pilot plant for CO₂ capture [46], located at TAURON facilities in Poland. Comparison of tests carried out both in a laboratory and PDU scales for gases containing 30% CO₂ is shown in Table 5.

CONCLUSIONS

Simple tests based on the determination of absorbent basic parameters allowed us easily to compare a wide range of sorbents. However, because of the multitude of the parameters which may affect the absorption process it is crucial to verify selected sorbents in real absorption-desorption systems.

The results obtained for selected solvents showed that it was to prepare solutions that feature significantly higher CO₂ removal efficiency and lower reboiler heat duty per kg of captured CO₂ than common solution of 30 wt% MEA, which is the most widely used solvent for the CO₂ removal process. The activated AMP solution was the most perspective for investigating the CO₂ absorption process in a larger scale due to the highest carbon dioxide removal rate and the lowest reboiler heat duty, which is shown in Table 6. An overall summary of the results obtained for different solvents is given in Table 6.

The obtained reboiler heat duty values appear to be fairly high compared to the values obtained elsewhere [48,49]. The reason is the substantial heat losses that appeared in the small scale installation. This method works when comparative studies are carried out, but it is challenging to implement directly the gained results into full-scale process. Furthermore, to improve the usefulness of the obtained results, it is necessary to perform additional tests on a larger scale (i.e., pilot or PDU).

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Technologies for Energy Generation: “Developing a technology of oxyfuel combustion for pulverized fuel and fluidized-bed furnaces integrated with CO₂ capture system”.

LIST OF ABBREVIATIONS

MEA	: monoethanolamine
AMP	: 2-amino-2-methyl-1-propanol
MDEA	: N-methyldiethanolamine
AEEA	: N-(2-hydroxyethyl)ethylenediamine
AEP	: 1-(2-aminoethyl)piperazine
DAP	: 1,3-diamino-2-propanol
PZ	: piperazine
n	: subscript indicating standard conditions in volume flow rate unit (1,013 hPa, 273 K)
PDU	: process development unit

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