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3 Evaluation of a phase change solvent for CO₂ capture:
4 Absorption and desorption tests

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8 **Abstract**

9 A blend of a tertiary amine (DEEA) and a diamine (MAPA) was studied in
10 a screening apparatus for preliminary absorption tests. Two immiscible liquid
11 phases were formed upon CO₂ loading and the system was shown to have large
12 capacity for CO₂. The two phases were analyzed individually for both amines
13 and CO₂. MAPA and water were found concentrated in the heavy CO₂ rich
14 phase whereas the CO₂ lean phase contained mainly of DEEA. Volumetric phase
15 ratio was measured as function of CO₂ loading and together with the individual
16 phase compositions this forms part of a basis for an equilibrium model. The
17 CO₂ rich phase was heated to desorption temperatures and shown to regenerate
18 CO₂ at higher pressures than normally used for 30 wt.% MEA. The data enabled
19 understanding the system behavior as MAPA is first loaded in the heavy phase
20 and subsequently DEEA reacts with CO₂ and dissolves. The data show that
21 the system has potential for significant reduction in regeneration heat through
22 high cyclic capacity, very high CO₂ stripping pressures, and for operating where
23 the heat of reaction lies between primary and tertiary amines. The tests also
24 give good estimates for the absorption rate at higher loadings.

25 *Keywords:* Screening, phase change solvent, DEEA, MAPA, CO₂ Capture

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26 1. Introduction

27 Reduction of carbon dioxide emissions is still a very important topic and the
28 research on reducing cost and energy demand for its capture has increased over
29 the past years. Using energy efficiently, reducing energy waste, changing fuel
30 sources to alternative sources poorer in carbon content and carbon capture and
31 storage (CCS) are some ways of reducing the CO₂ emissions. Among the CCS
32 technologies, chemical absorption using aqueous amine solutions with thermal
33 regeneration of the solvent is the most developed and applied technology for CO₂
34 capture (Svendsen et al., 2011). The 30 mass % monoethanolamine (MEA)
35 system is the benchmark solvent for this technology (Aroonwilas & Veawab,
36 2009; Rey et al., 2013) and research on this solvent is still progressing (see, for
37 example, Razi et al., 2013; Vevelstad et al., 2013).

38 Most of the energy required for CO₂ capture in amine scrubbing systems is
39 used for regenerating the solvent (Aroonwilas & Veawab, 2007). Process modifi-
40 cations are used in an attempt to reduce this demand. Several process modifica-
41 tions were studied and proposed in the literature (Cousins et al., 2011a,b; Karimi
42 et al., 2011; Oyenekan & Rochelle, 2007; Rochelle et al., 2011). Nevertheless,
43 the development of new solvents or solvent blends is an important way of reduc-
44 ing the energy demand in amine scrubbing plants. Apart from MEA, piperazine
45 (PZ) and piperazine blends, AMP(2-amino-2-methyl-1-propanol) based systems,
46 for instance with PZ, and amino acid based systems have been intensively stud-
47 ied (Jockenhvel & Schneider, 2011; Kuettel et al., 2013). Dugas & Rochelle
48 (2009), for instance, showed that the 8 m piperazine solution has 75% higher
49 capacity than a 7 m MEA solution. Also, CO₂ reaction rates are 2-3 times faster
50 on PZ solutions. Other examples are large scale tests with commercial solvents
51 which are claimed to be better than aqueous solutions of MEA, as the Cansolv
52 solvent and KS-1 from MHI, (see Endo et al., 2011; Just, 2013; Kamijo et al.,
53 2013; Shaw, 2009).

54 A new breed of systems, the phase change solvents, has received much at-
55 tention during the last 4-5 years. Precipitating systems with amino acid salts

56 (Ma'mun & Kim, 2013; Sanchez-Fernandez et al., 2013) and carbonated solu-
57 tions (Moene et al., 2013) are claimed to be promising systems for CO₂ capture.
58 IFPEN recently proposed the DMXTM process in which the solvent forms two
59 immiscible liquid phases upon CO₂ loading (Aleixo et al., 2011; Raynal et al.,
60 2011a,b). This process is able to operate with energy demands as low as 2.1
61 GJ/ton of CO₂. Other amine based solvents that form two phases before or
62 after CO₂ loading were recently patented (Svendsen & Trollebø, 2013).

63 The development of a new solvent is not simple. A large set of experiments
64 must be performed in order to characterize the system. Screening experiments
65 is a fast way to identify potential solvents for CO₂ capture, e.g. Ma'mun et al.
66 (2007) used a screening apparatus to evaluate the absorption rate of different
67 amine based solvents for CO₂ capture. Aronu et al. (2009) modified the same
68 apparatus to allow for desorption operation testing. The authors presented
69 a comparison of absorption and stripping performance for some amine/amine
70 blend solutions. Aronu et al. (2010) and Aronu et al. (2011) used the screen-
71 ing apparatus to evaluate amino acid salts and amine/amine blend solvents,
72 respectively.

73 In this work, a blend of 5M DEEA (Diethylethanolamine) and 2M MAPA
74 (N-Methyl-1,3-diaminopropane) was tested in the screening apparatus. This
75 mixture, as in the DMXTM solvent, forms two liquid phases upon CO₂ loading.
76 By separating out the CO₂ rich phase, it is possible to send a smaller volume
77 of solvent for regeneration, thereby, potentially reducing the process energy
78 demand. Also, the CO₂-rich phase can have a potential for easy stripping,
79 thereby also reducing the stripping steam demand and possibly increasing the
80 regeneration pressure. The amines here studied are potential solvents for CO₂
81 capture, and were also studied by other authors (Hartono et al., 2013; Monteiro
82 et al., 2013b,a; Pinto et al., 2014; Voice et al., 2013). The screening apparatus
83 used in Aronu et al. (2010) was used for absorption tests at different CO₂
84 partial pressures and temperatures. The system was characterized by individual
85 analyses of the two liquid phases at equilibrium, and the phase ratio recorded.
86 The CO₂-rich phase formed upon CO₂ loading was separated after absorption

87 and used for desorption tests in a separate apparatus which is also shown in this
88 work.

89 2. The phase change solvent

90 As in the DMXTM process (Raynal et al., 2011a), the system studied here
91 forms two phases upon CO₂ loading. The chemicals used on the DMXTM process
92 are not given anywhere. In this work, an aqueous solution of a tertiary alka-
93 nolamine (DEEA) and a diamine (MAPA) with a primary and a secondary
94 amine group was studied. In Fig. 1 the chemical structure of the amines used
95 in the solution is shown.

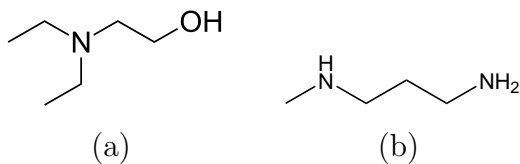


Figure 1: Chemical structure: (a) DEEA; (a) MAPA.

96 The system has an advantage of combining high absorption rate, provided
97 by the diamine, and high capacity from the tertiary amine. Puxty et al. (2009)
98 showed that DEEA has a significant absorption capacity while MAPA has a high
99 initial absorption rate. Moreover, the energy required for regenerating a tertiary
100 amine is lower than for primary and secondary amines (Kim & Svendsen, 2011).
101 It is therefore a potential for lowering the total energy consumption of the overall
102 process significantly.

103 A solution of 5M DEEA and 2M MAPA is a single phase solution. When the
104 solution starts to absorb CO₂, at some point, it becomes turbid which indicates
105 a phase changing behaviour. After leaving the loaded solution to rest two clear
106 liquid phases can be observed. This behaviour is shown in Fig. 2.

107 2.1. The phase change solvent capture process

108 The phase change solvent uses the advantage of the phase split to reduce
109 the energy demand of the CO₂ capture process. The changes in the process

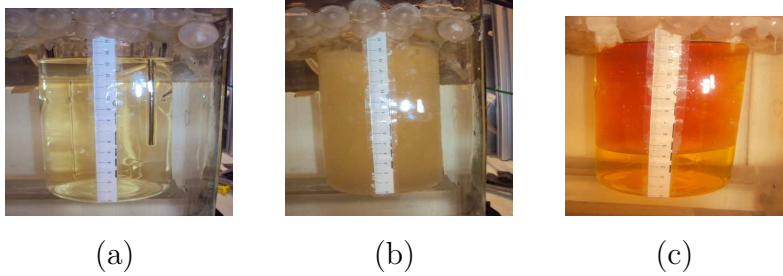


Figure 2: A 5M DEEA/2M MAPA solution: (a) Before, (b) During and (c) after CO₂ loading.

110 configuration, as shown in Fig. 3, are made not only to reduce the energy
 111 demand, but also as a result of the characteristics of the system. The process
 112 flow diagram presented in this work is very similar to the one presented in
 113 Raynal et al. (2011b). However, the phase separator is placed before the cross
 114 heat exchanger.

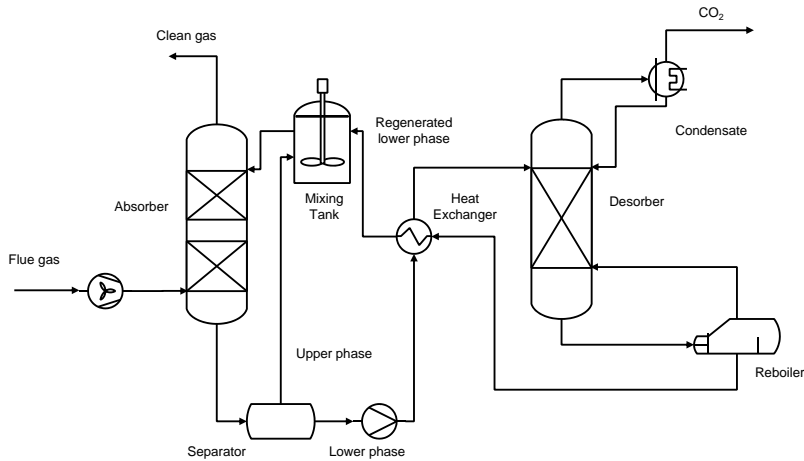


Figure 3: Capture process layout for the phase change solvent

115 3. Experiments

116 The apparatus' descriptions and their operational details are given in this
 117 section.

118 *3.1. Screening apparatus*

119 The screening apparatus (Fig. 4) is designed to operate up to 80°C and at
120 atmospheric conditions for rapid evaluation of absorption and stripping perfor-
121 mance of solvents. As discussed in Aronu et al. (2009) and Ma'mun et al. (2007),
122 the results from the screening apparatus are semi quantitative and should be
123 understood only as an indication of a solvent's performance. The apparatus
124 used in this work is the same as the one used in Aronu et al. (2010) with minor
125 modifications. The reactor was changed to a glass volume graded vessel where
126 it was possible to see the phase change formation and the CO₂ analyzer was
127 also replaced by a Rosemount BINOS 100 IR CO₂-analyzer.

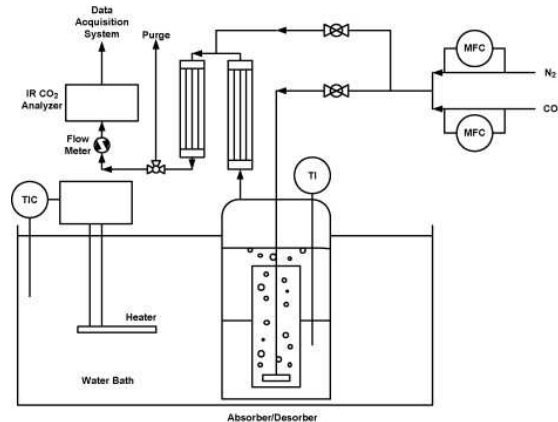


Figure 4: Simplified diagram of the screening apparatus (Aronu et al., 2010).

128 A known volume and mass (around 750 ml) of the solvent was weighed into
129 the reactor and a synthetic mixture of CO₂ and N₂, with a total flow of 5
130 Nl/min, was bubbled into the solvent. The CO₂ concentration (flow) in the
131 inlet gas was adjusted to the desirable value by adjusting the mass flow of N₂
132 and CO₂ while keeping the total flow at 5 Nl/min. After bubbling through the
133 solution, the gas stream was cooled on-line through two condensers placed on
134 top of each other and the condensate was directly returned to the reactor. The
135 dried gas was sent to the IR analyzer for CO₂ analysis.

136 The absorption tests were performed at 40, 60 and 80°C and from 1 to

137 20 kPa of CO₂ partial pressure in the inlet gas. An experiment was stopped
138 when 95% of the concentration of the inlet gas was achieved in the outlet of the
139 reactor. The mixture was then left to rest so that the phases could separate
140 at the experimental temperature. The volumes of the lower and upper phases
141 were recorded and sampled individually at the experimental temperature, after
142 which they were separated and stored in different bottles for further analysis.

143 *3.2. High pressure desorption apparatus*

144 Stripping experiments were done on the lower phase (CO₂ rich phase) gener-
145 ated in the screening apparatus. A different apparatus was used for this purpose.
146 The apparatus consisted of a 150 ml stainless steel vessel immersed in an oil
147 bath where the temperature was held constant by a Julabo 6 heating system. A
148 thermocouple was placed inside the vessel and in contact with the liquid (Fig.
149 5). The temperature of the liquid as well as the pressure of the system were
150 read and recorded through a program coded in LabView software. The cylinder
151 was evacuated to about 20 mbar and around 80 ml of a lower phase sample was
152 sucked in. The oil bath was set to a desired temperature, and once the tempera-
153 ture and pressure were stable, a point was recorded. Temperature and pressure
154 were considered stable if in a 10 minutes window no variations occurred above a
155 given limit ($\pm 0.1^\circ\text{C}$ and ± 1 mbar). The temperature was then increased and
156 the procedure was repeated until the pressure was close to 6 bar. This was due
157 to the pressure transducer having an upper limit of 6 bar.

158 A total of eight samples from the screening tests were tested for high pressure
159 desorption. Table 1 shows the samples selected for the high pressure desorption
160 tests.

161 **4. Results**

162 *4.1. Screening calculations*

163 A Labview data acquisition software was used to record the measured vari-
164 ables (temperatures, flows and CO₂ content in the outlet gas) every minute from

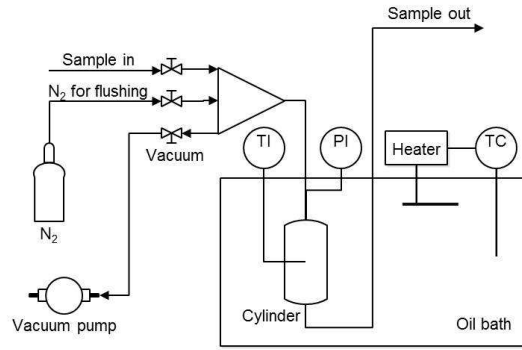


Figure 5: Simplified diagram of the high pressure desorption apparatus.

Table 1: Screening samples taken for high pressure desorption tests

Sample	Absorption Temperature [$^{\circ}C$]	P_{CO_2}
1	40	6
2	40	8
3	40	10
4	40	13
5	60	8
6	60	10
7	60	13
8	80	8

165 the start of the experiment. The flows of dry N₂ and CO₂ in the inlet gas were
 166 adjusted to the desirable concentration and were kept constant throughout the
 167 experiment. The dry N₂ gas flow was considered to be the same in the inlet and
 168 outlet since N₂ is an inert gas. The total molar flow could then be calculated
 169 from the N₂ inlet flow and the CO₂ content given by the IR analyzer, as shown
 170 by Eq. 1. The CO₂ mole fraction in the outlet gas stream was calculated by
 171 Eq. 2. Finally, the amount of CO₂ absorbed is given by the difference between
 172 amounts of CO₂ in the inlet and outlet, and can be calculated from the measured
 173 variables according to Eq. 3.

$$Q_{total}^{out} [mol/min] = \frac{n_{N_2}^{in}}{x_{N_2}^{out}} = \frac{n_{N_2}^{in}}{(1 - x_{CO_2}^{out})} \quad (1)$$

$$x_{CO_2}^{out} = \frac{(CO_2 \text{ vol}\%)}{100} \quad (2)$$

$$Q_{CO_2} [mol/min] = n_{CO_2}^{in} - n_{CO_2}^{out} = n_{CO_2}^{in} - x_{CO_2}^{out} Q_{total}^{out} \quad (3)$$

174 The CO₂ absorption rate was calculated according to Eq. 4. Since the mass
 175 (M^{sol}) and the volume (V) of the solvent in the reactor and the accumulated
 176 amount of CO₂ ($Q_{CO_2}^{Acc.}$) are known, the loading in moles of CO₂ per kilogram
 177 of solvent could be calculated according to Eq. 5. It is important to point it
 178 out that Eq. 5 computes the loading with respect to the total solvent mass. In
 179 other words, this accounts for the weight of both the lower and upper phases.

$$r_{CO_2} \left[\frac{mol}{kg.min} \right] = \frac{Q_{CO_2}}{M^{sol}} \quad (4)$$

$$\alpha \left[\frac{mol \ CO_2}{kg \ solution} \right] = \frac{Q_{CO_2}^{Acc.}}{M^{sol}} \quad (5)$$

180 4.2. Screening results

181 The absorption rates of CO₂ at 40, 60 and 80°C are shown in Fig. 6, 7 and
 182 8, respectively. As reference a 5M MEA solution (30 % wt.) was tested at 40
 183 °C and 10 kPa of CO₂ partial pressure.

184 The comparison between the DEEA/MAPA system and MEA is not straight
185 forward. Several properties, for example, the viscosity, which is much higher in
186 the DEEA/MAPA system, are different making the comparison difficult. Al-
187 though the superficial gas velocity was the same for all experiments, the gas-
188 liquid interfacial area could not be guaranteed to be the same, as explained
189 before in Ma'mun et al. (2007). In Fig. 6, up to the region of 1.2 mol CO₂/kg
190 solution loading, it is possible to conclude that the DEEA/MAPA system shows
191 a slightly higher absorption rate than the 5M MEA (indicating that it absorbs
192 CO₂ faster than the 5M MEA) and it retains the absorption rate more constant
193 than 5M MEA. However, when approaching the equilibrium (high loading re-
194 gion) the MEA solution has a sharper fall towards zero absorption rate, while
195 the DEEA/MAPA system presents a more drawn out tail type of ending. This
196 is due to the saturation of the primary and secondary amine groups in MAPA
197 while the tertiary amine (DEEA) is still absorbing but at lower absorption rates.

198 Increasing the temperature led to a small increase in the CO₂ absorption
199 rate. Increasing the CO₂ partial pressure in the gas, also, increases the reaction
200 rate, as shown in Fig. 9. This is already expected since the driving force is
201 increase when the amount of CO₂ is increased in the gas. It should be noted,
202 however, that the initial flat part of all the screening curves is not representative
203 of the real absorption rates as in this region, all, or close to all, CO₂ in the gas
204 is removed. However, in the tail end of the curves the measurements give good
205 indication of the rate of absorption in the DEEA/MAPA system.

206 After an experiment was terminated (achieving 95% of the CO₂ concentra-
207 tion in the inlet gas stream in the outlet gas stream), the solvent was left to
208 separate at the experiments temperature, so the volume ratios could be regis-
209 tered. The phase separation time was also recorded and it varied from 25-30
210 minutes at 40°C to 3-5 minutes at 80°C.

211 Even though the rate measurements are only semi-quantitative, the screening
212 experiments contribute with many other measurements which can be used in
213 understanding and modeling of the system. The volume ratio (ϕ), defined as
214 the ratio between the volumes of the lower and the upper phase, and the final

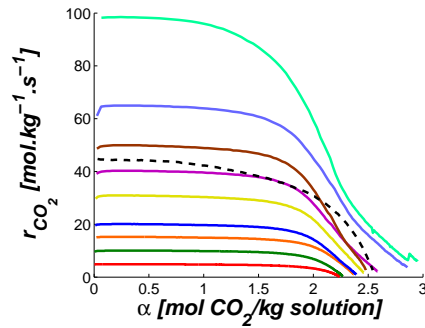


Figure 6: Screening tests performed at 40°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa P_{CO_2} . Dashed curve: 30 mass% MEA at 10 kPa P_{CO_2} .

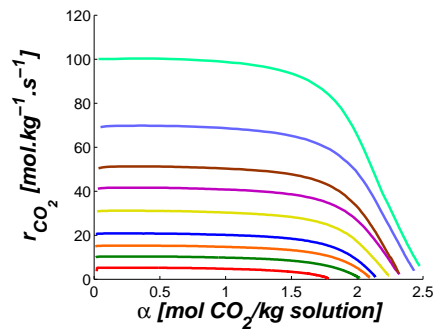


Figure 7: Screening tests performed at 60°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa P_{CO_2} .

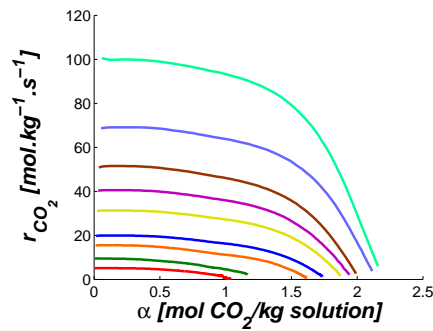


Figure 8: Screening tests performed at 80°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa P_{CO_2} .

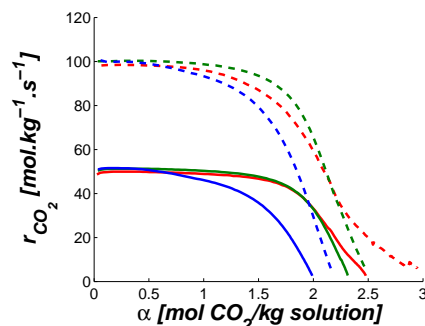


Figure 9: Screening tests performed at 10 kPa and: (—) 40°C, (—) 60°C and (—) 80°C. Screening tests performed at 20 kPa and: (- -) 40°C, (- -) 60°C and (- -) 80°C

215 loading (α_{final}), in mol CO₂/ kg of solution, are given in Table A1 in the
 216 appendix.

217 Both the upper and lower phases were analyzed for CO₂ and amine content.
 218 LCMS analyses were used to quantify the ratio between the concentrations
 219 of DEEA and MAPA while total alkalinity and CO₂ content analyses were
 220 performed by the procedure described in Monteiro et al. (2013a). With the
 221 concentration ratio, the total alkalinity and the CO₂ content, the individual
 222 species concentrations were calculated for all samples and are shown in Table
 223 A2 in the appendix.

224 From the analyses it was possible to identify a CO₂ rich phase (the lower
 225 phase) and a CO₂ lean phase (the upper phase). The CO₂ rich phase was rich in
 226 MAPA and H₂O whereas the CO₂ lean phase was composed mainly of DEEA.
 227 The upper phase would work as a buffer of DEEA, which would move to the
 228 lower phase as more CO₂ is capture by the solvent.

229 The concentration ratios between DEEA and MAPA (mole/mole) in the
 230 lower phase given by the LCMS are shown in Fig. 10. Apart from some points
 231 at low CO₂ concentration at 80°C, the concentration of DEEA in the lower phase
 232 increases more or less linearly with the increase of CO₂ in the system and the
 233 slope of this linear tendency decreases with the increase of temperature. The 2-3
 234 points at low CO₂ loading at 80°C are believed not to be outliers, but a result

235 of the increased miscibility between the two phases at higher temperatures.
 236 There was, however, no second experiment performed at 80°C for checking
 237 reproducibility. As temperature increases, the solubility of DEEA in the lower
 238 phase goes up and this is reflected in the increased DEEA concentration seen in
 239 these points. This is also shown in Fig. 11 where the volume ratio (ϕ), between
 240 the lower and upper phase is given as function of CO₂ partial pressure and
 241 temperature. At 40°C the lower/upper ratio increases with loading in an almost
 242 linear fashion. At 60°C the ratio increases rapidly at low partial pressures, and
 243 loadings, whereas it levels off at higher loadings. At 80°C this tendency is even
 244 clearer as the lower/upper ratio is very low at low loading, indicating better
 245 miscibility. This supports the previously discussed results shown in Fig. 10 at
 246 80°C. Although the measurements show clear trends for the volume ratios of the
 247 phases, the uncertainty in the measurements is relatively high and estimated to
 248 ± 0.03 - 0.05 in volume ratio.

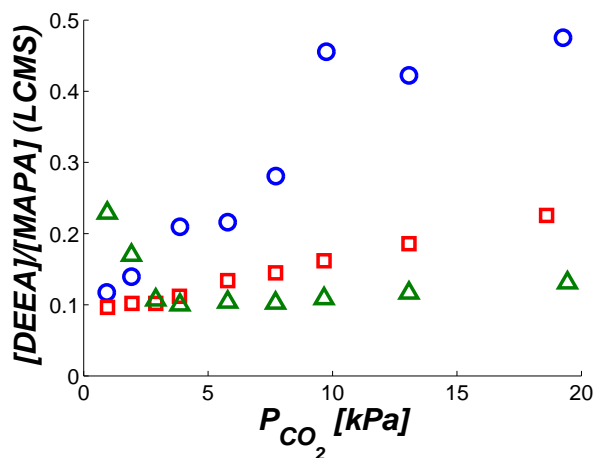


Figure 10: Concentration ratio (mole DEEA/ mole MAPA) from LCMS in the lower phase.
 Experiments performed at: (○) 40, (□) 60 and (△) 80°C.

249 The mole fractions of the species are shown in Fig. 12, 13 and 14 for the ex-
 250 periments performed at 40, 60 and 80°C respectively. As previously mentioned,
 251 it is possible to identify a CO₂ rich (lower) and lean (upper) phase. The DEEA

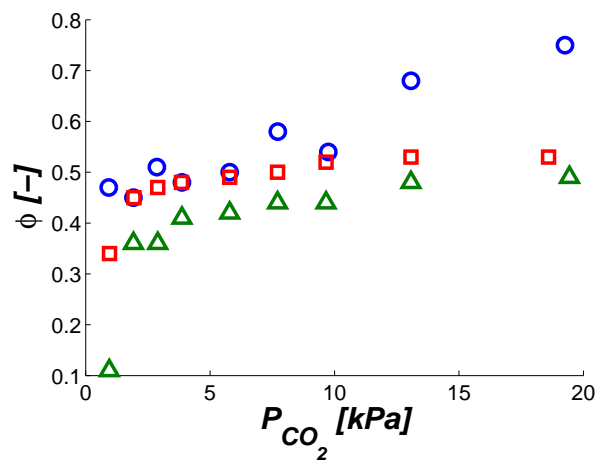


Figure 11: Volume distribution. Experiments performed at: (○) 40, (□) 60 and (△) 80°C.

252 is mainly concentrated in the upper phase. However, as more CO₂ is added to
 253 the system, the DEEA tends to migrate to the lower phase and, therefore, its
 254 concentration is reduced in the upper phase.

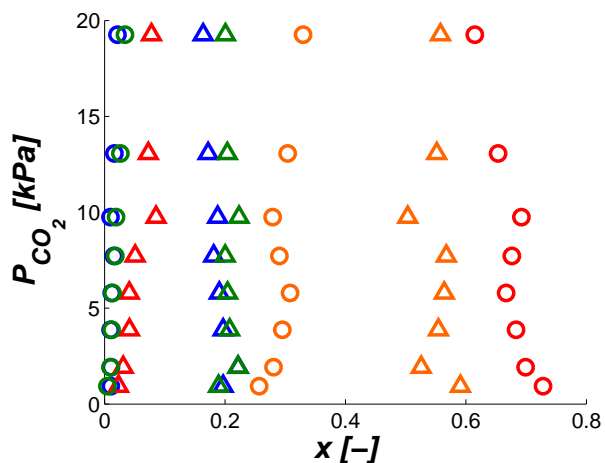


Figure 12: : Mole fraction distribution for experiments taken at 40°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO₂ and (○) H₂O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO₂ and (△) H₂O.

255 Fig. 15 shows the CO₂ content (from titration) per kg solution for the lower

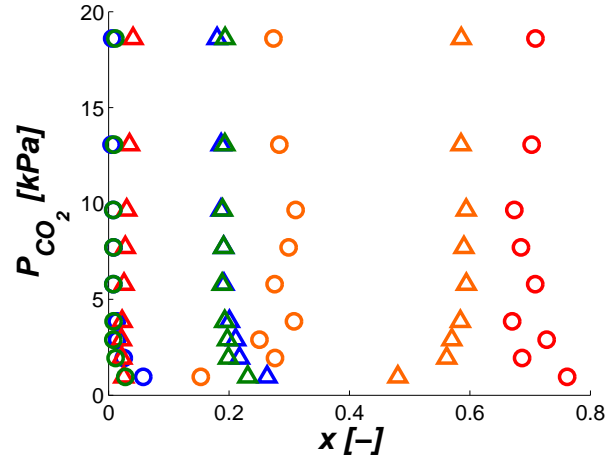


Figure 13: Mole fraction distribution for experiments taken at 60°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO₂ and (○) H₂O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO₂ and (△) H₂O.

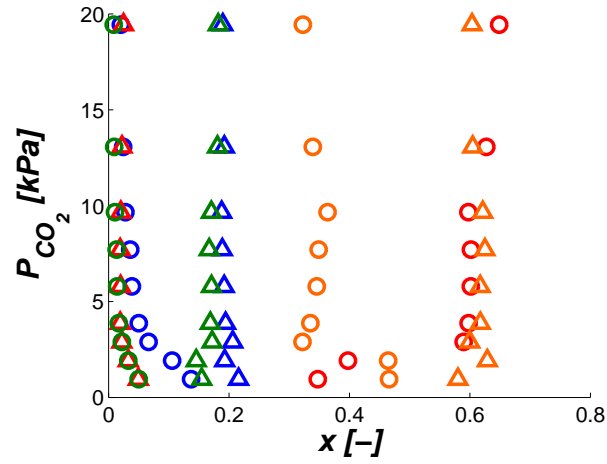


Figure 14: Mole fraction distribution for experiments taken at 80°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO₂ and (○) H₂O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO₂ and (△) H₂O.

256 and upper phases as function of the absorption CO_2 partial pressure. The CO_2
 257 content is nearly constant as function of the absorption CO_2 partial pressure,
 258 except for the 2-3 first points. As more CO_2 is added to the system, more DEEA
 259 migrates to the lower phase, thus, keeping the ratio mole CO_2/kg solution close
 260 to constant.

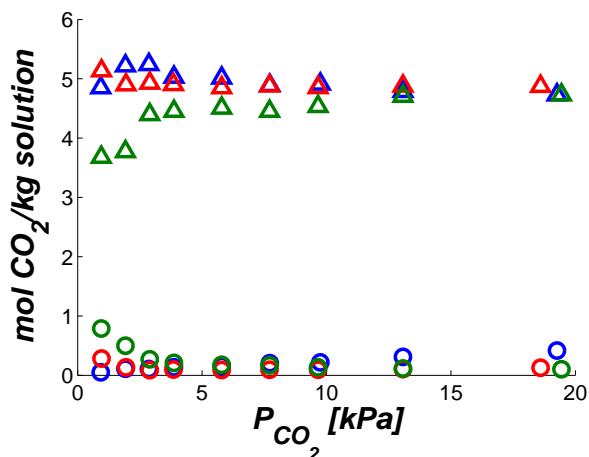


Figure 15: CO_2 content in the lower and upper phases as function of absorption CO_2 partial pressure. Upper phase: (\circ) 40, (\circ) 60 and (\circ) 80°C. Lower phase: (Δ) 40, (Δ) 60 and (Δ) 80°C.

261 After separating the phases, the density of each phase was measured at 25°C
 262 for all experiments. Apart from the measurements at 80°C and low CO_2 partial
 263 pressures, it seems that there is no significant variation in the sample densities.
 264 Fig. 16 shows the densities for the upper and lower phases as function of the
 265 experimental CO_2 partial pressure and temperature. The density data for low
 266 loadings at 80°C again show the increased miscibility at higher temperatures.
 267 The data are given in Table A4 in the appendix.

268 4.3. High pressure desorption tests

269 The lower phase solutions from the various screening tests were tested for
 270 stripping performance as described in section 3.2. The lower phase of the DEEA

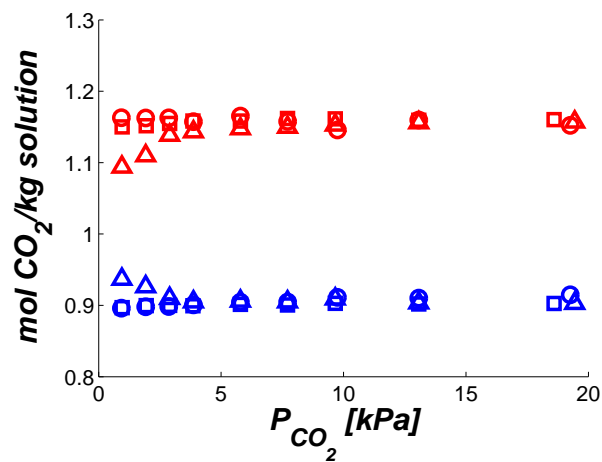


Figure 16: Densities of the phases at 25°C. Upper phase from experiments performed at: (○) 40, (□) 60 and (△) 80°C. Lower phase from experiments performed at: (○) 40, (□) 60 and (△) 80°C.

271 /MAPA system showed a high potential for generating CO₂ at elevated pres-
 272 sures.

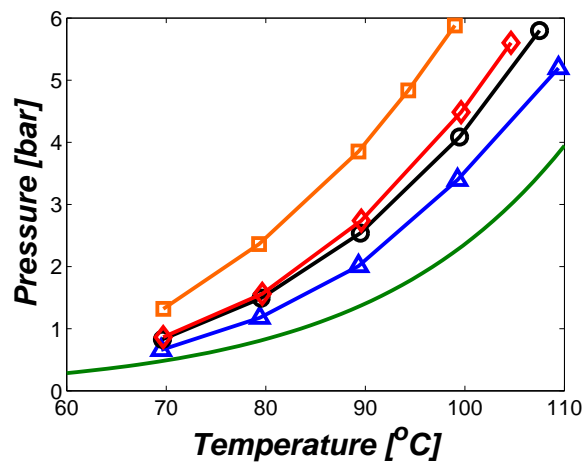


Figure 17: Total pressure from lower phase samples with absorption taken at 40°C from the screening apparatus. P_{CO_2} : (△) 6 kPa, (○) 8 kPa, (◇) 10 kPa and (□) 13 kPa. (—) MEA at loading 0.5 mol CO₂/mol MEA (model from Hessen et al. (2010)).

273 In Fig. 17 are shown the vapor pressures of the lower phase, from absorption
274 tests at 40°C and different CO₂ partial pressures, as a function of temperature.
275 As expected, the total pressure increases with the sample CO₂ loading, reflected
276 in the partial pressure at which it has been generated. It should be kept in
277 mind that the solutions were generated at 95% of the given pressure. As can
278 be seen the pressures that can be generated are significantly higher than for a
279 representative 30 mass% MEA solution with loading 0.5 mole CO₂/mole MEA.
280 The high pressures that can be obtained can be exploited in two ways. One
281 way is to use a normal regeneration temperature of about 120°C and produce
282 CO₂ at elevated pressure, possibly at 6-8 bar. This method will save electrical
283 energy for the recompression of CO₂ to transportation pressure (around 110
284 Bar). In a coal based power station typically recompression energy will be 25-
285 30% of the total energy demand and thus significant savings can be achieved. A
286 second option is to operate the regenerator at lower temperatures than normal,
287 e.g. below 100°C. By doing this the stripping pressure will not be increased
288 compared to the normal situation but the quality of the heat to be supplied will
289 be reduced. In some cases this may be a better option. A lower regeneration
290 temperature will also positively affect the solvent degradation processes as these
291 will be slowed down. The reason for the ease of stripping from this system is a
292 result of the behavior of the blended system. As observed, the diamine MAPA
293 is very rapidly loaded. This is seen from the flat part of the screening curves
294 in Fig. 6-8. When MAPA is almost fully loaded, DEEA starts loading up
295 and is transferred to the heavy bottom phase. When stripping it is basically
296 DEEA that strips and the loading of MAPA remains nearly unchanged. This
297 mechanism has another advantage. As shown by Arshad et al. (2013), the heat
298 of absorption goes significantly down when MAPA is almost fully saturated
299 and DEEA is being loaded. The heat of absorption drops from about 85-90
300 kJ/mole CO₂ in the range where MAPA loads to about 60 kJ/mole CO₂ when
301 DEEA predominantly loads. The operational cycle in this system will be in
302 this intermediate range where the heat of absorption and desorption will be
303 determined mainly by DEEA, but also to some extent by MAPA. All the high

304 pressure desorption test results are displayed in Table A3.

305 **5. Conclusions**

306 Preliminary absorption tests made on a phase change solvent, composed
307 of an aqueous mixture of 5M DEEA and 2M MAPA, were performed. The
308 solvent showed a great absorption capacity. Upon CO₂ loading, the solvent
309 splits in two immiscible phases. From the analyses it was possible to see that
310 the CO₂ rich phase was rich in MAPA and H₂O whereas the CO₂ poor phase
311 was mainly composed of DEEA. As more CO₂ was added to the system, more
312 DEEA is transferred to the lower phase. The volume of the lower phase was
313 also increased by adding more CO₂ to the system. The phase separation was
314 accelerated by increasing the temperature.

315 Desorption tests made on the CO₂ rich phase from the screening tests showed
316 that the DEEA/MAPA system can produce CO₂ at elevated pressures with the
317 solvent regeneration performed at lower temperatures compared to the tradi-
318 tional 30 mass% MEA process.

319 The new, biphasic system will thus have three advantages. The cyclic capac-
320 ity is high thereby reducing the sensible heat demand; the lower phase shows a
321 significant potential for increased CO₂ pressure during stripping thus enabling
322 regeneration at elevated pressure or lower temperature, and finally the operation
323 will take place in a domain where mainly loading and stripping of the tertiary
324 amine takes place, thereby lowering the heat of absorption.

325 The data generated are of great value for further modeling purposes. Nonethe-
326 less, more experiments need to be carried out to confirm the systems potential
327 as a solvent for CO₂ capture.

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332 **Appendix A: Experimental data**

Table A1: DEEA/MAPA system volume ratio and final loading at different temperatures and P_{CO_2}

40 °C			60 °C			80 °C		
P_{CO_2} [kPa]	ϕ	α_{final} [mol/kg]	P_{CO_2} [kPa]	ϕ	α_{final} [mol/kg]	P_{CO_2} [kPa]	ϕ	α_{final} [mol/kg]
0.93	0.47	2.2347	0.96	0.34	1.7786	0.95	0.11	1.0368
1.92	0.45	2.2636	1.94	0.45	2.0180	1.92	0.36	1.1682
2.86	0.51	2.3675	2.90	0.47	2.0958	2.90	0.36	1.6157
3.87	0.48	2.3906	3.85	0.48	2.1380	3.87	0.41	1.7372
5.79	0.50	2.4595	5.79	0.49	2.2407	5.79	0.42	1.8720
7.72	0.58	2.5849	7.71	0.50	2.3207	7.71	0.44	1.9409
9.75	0.54	2.4792	9.66	0.52	2.3141	9.66	0.44	1.9901
13.07	0.68	2.8579	13.07	0.53	2.4329	13.07	0.48	2.1142
19.26	0.75	2.9505	18.6	0.53	2.4753	19.44	0.49	2.1587

Table A2: Individual species concentration (mol/l) for the upper and lower phases after phase separation.

Absorption taken at 40°C									
P_{CO_2} [kPa]	0.93	1.92	2.86	3.87	5.79	7.72	9.75	13.07	19.26
Upper phase									
MAPA	0.1036	0.1073	N/A	0.1043	0.126	0.1631	0.1016	0.1747	0.2421
DEEA	7.1625	7.1071	N/A	7.0916	7.0686	7.0616	7.1797	7.0264	6.9115
CO ₂	0.0460	0.1000	N/A	0.1211	0.1412	0.1861	0.1988	0.2826	0.3826
H ₂ O	2.5246	2.8536	N/A	3.0625	3.2645	3.0329	2.895	3.2692	3.7069
Lower phase									
MAPA	5.8666	6.0861	N/A	5.5173	5.4462	5.1229	4.7332	4.6847	4.4476
DEEA	0.6882	0.8489	N/A	1.1560	1.1761	1.4384	2.1558	1.9786	2.1137
CO ₂	5.6418	6.0682	N/A	5.8192	5.8410	5.6590	5.6292	5.5524	5.4492
H ₂ O	17.6096	14.418	N/A	15.5282	16.1239	16.0315	12.6823	15.0301	15.1468
Absorption taken at 60°C									
P_{CO_2} [kPa]	0.96	1.94	2.9	3.85	5.79	7.71	9.66	13.07	18.6
Upper phase	0.5265	0.2572	0.1404	0.1430	0.0810	0.0862	0.0825	0.0508	0.0613
MAPA	6.9469	7.0057	7.1656	7.0374	7.1722	7.1121	7.1069	7.1783	7.1859

DEEA	0.2530	0.1200	0.0786	0.0887	0.0800	0.0833	0.0836	0.0938	0.1156
CO ₂	1.3974	2.8197	2.4677	3.2325	2.7909	3.1043	3.2767	2.8973	2.7762
H ₂ O	0.5265	0.2572	0.1404	0.143	0.0810	0.0862	0.0825	0.0508	0.0613
Lower phase									
MAPA	6.7278	6.1633	6.0820	5.9125	5.7070	5.6784	5.5565	5.4773	5.2666
DEEA	0.6480	0.6275	0.6202	0.6617	0.7641	0.8228	0.8992	1.0175	1.1877
CO ₂	5.9074	5.6423	5.6908	5.6793	5.6169	5.6718	5.6340	5.6551	5.6545
H ₂ O	12.282	15.9186	16.4308	17.1997	17.6921	17.5203	7.6773	17.1371	17.1123
Absorption taken at 80°C									
<i>P</i> _{CO₂} [kPa]	0.95	1.92	2.90	3.87	5.79	7.71	9.66	13.07	19.44
Upper phase									
MAPA	2.0265	1.4879	0.7399	0.5586	0.4338	0.4045	0.3218	0.2660	0.2178
DEEA	5.1342	5.6052	6.5639	6.6581	6.7498	6.7594	6.8310	6.8973	6.9735
CO ₂	0.7380	0.4630	0.2445	0.1890	0.1599	0.1516	0.1229	0.1047	0.0915
H ₂ O	6.8850	6.5461	3.5844	3.7324	3.8779	3.9188	4.1590	3.7296	3.4692
Lower phase									
MAPA	5.6119	5.5516	5.9994	5.8536	5.818	5.7878	5.7497	5.7860	5.6992
DEEA	1.2844	0.9419	0.6417	0.5854	0.6042	0.5925	0.6253	0.6744	0.7453
CO ₂	4.0244	4.1857	5.0056	5.0913	5.1720	5.1214	5.2283	5.4405	5.4730

H2O	15.0900	18.0978	17.4517	18.5929	18.6896	19.1558	19.0301	18.1897	18.1393
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Table A3: High pressure desorption data for the screening lower phase samples.

Screening experiment	Temperature [$^{\circ}C$]	Pressure [mbar]
40 $^{\circ}C$ and 6 kPa	69.50	656.479
	79.41	1174.313
	89.32	2007.091
	99.27	3394.332
	109.41	5193.126
40 $^{\circ}C$ and 8 kPa	69.62	826.588
	79.53	1490.750
	89.51	2537.697
	99.47	4084.989
	107.50	5797.586
40 $^{\circ}C$ and 10 kPa	69.70	863.062
	79.65	1559.486
	89.60	2738.535
	99.62	4484.843
	104.63	5602.400
40 $^{\circ}C$ and 13 kPa	69.71	1318.472
	79.33	2361.130
	89.32	3850.758
	94.32	4834.645
	99.00	5879.321
60 $^{\circ}C$ and 8 kPa	69.63	376.760
	79.57	655.999
	89.51	1119.848
	99.50	1940.140
	109.50	3195.233
	119.60	5105.616

60°C and 10 kPa	69.47	411.752
	79.51	669.675
	89.46	1201.867
	99.46	2056.919
	109.47	3365.524
	119.57	5383.384
60°C and 13 kPa	69.30	437.794
	79.33	776.192
	89.13	1363.450
	99.25	2243.332
	109.01	3648.222
	119.24	5606.304
80°C and 8 kPa	69.64	209.694
	74.60	270.101
	79.44	344.823
	89.40	589.248
	99.37	990.095
	109.42	1641.672
	119.50	2697.620

Table A4: Densities of the loaded and unloaded phases at 25°C.

Temperature [°C]	P_{CO_2}	ρ_{upper}	ρ_{lower}
40	0.93	0.8960	1.1630
	1.92	0.8981	1.1625
	2.86	0.8984	1.1627
	3.87	0.9007	1.1574
	5.79	0.9044	1.1652
	7.72	0.9047	1.1577
	9.75	0.9112	1.1458

	13.07	0.9101	1.1597
	19.26	0.9149	1.1522
	0.96	0.8968	1.1500
	1.94	0.8997	1.1516
	2.90	0.9000	1.1550
	3.85	0.8994	1.1582
60	5.79	0.9014	1.1582
	7.71	0.9006	1.1619
	9.66	0.9028	1.1613
	13.07	0.9020	1.1594
	18.60	0.9026	1.1603
	0.95	0.9367	1.0939
	1.92	0.9262	1.1097
	2.90	0.9097	1.1384
	3.87	0.9050	1.1433
80	5.79	0.9061	1.1476
	7.71	0.9050	1.1498
	9.66	0.9092	1.1527
	13.07	0.9035	1.1559
	19.44	0.9029	1.1571

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