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Heat of absorption of carbon dioxide (CO_2) into aqueous N-methyldiethanolamine (MDEA) and N,N-dimethylmonoethanolamine (DMMEA)

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Abstract

In this work the heat of absorption of CO_2 into aqueous solutions of N-methyldiethanolamine (MDEA) and N,N-dimethylmonoethanolamine (DMMEA) has been investigated in a reaction calorimeter. The experiments were conducted at the concentrations 2M and 4.2M, and the temperatures 313 K, 353 K and 393 K. The resulting differential heats of absorption were plotted as a function of liquid CO_2 loading. An effect of temperature on the heat of absorption was observed for MDEA. This effect was larger at low loadings and the lower amine concentration. This was also observed for DMMEA but here the effect of temperature was larger. The heat of absorption of CO_2 into aqueous DMMEA is slightly larger than for MDEA.

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Keywords: Heat of absorption; Enthalpy of absorption; MDEA; DMMEA; Methyldiethanolamine; Dimethylmonoethanolamine; Calorimeter

1. Introduction

The capture of carbon dioxide (CO_2) from gas streams is important from both an economical and environmental perspective. In the oil and gas industry the capture of CO_2 from a natural gas streams, a process known as natural gas sweetening, is important in order to reach given pipe line specifications and sale gas specifications.

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Because CO_2 is a greenhouse gas there is a need to reduce the emissions of CO_2 into the atmosphere as a way of curbing the effects of climate change.

There are several different ideas and technologies on how to perform CO_2 capture, but the most mature and proven technology is absorption of CO_2 into aqueous alkanolamines. However, even though the technology is mature there is always a demand for a more energy efficient process in order to reduce the operational costs. About half of the energy requirement when capturing CO_2 from exhaust gas from a coal-fired power plant is from the regeneration of the solvent in the stripper reboiler, while the other half is from compression of CO_2 for transportation and sequestration [1]. It is believed that innovations in the field of process design will cut some of the operational costs along with the development of new solvents.

The use of alkanolamines in CO_2 capture processes has received attention due to environmental issues related to emissions and limited biodegradability [2, 3]. According to the Petroleum safety Authority of Norway chemicals used in off-shore applications are separated into four categories; black, red, yellow, and green based on their biodegradability, bioaccumulation and eco-toxicity. Chemicals that falls within the black or red categories should "only be selected if they are necessary for technical or safety reasons"[4]. Chemicals such as N-methyldiethanolamine (MDEA) and piperazine, which are popular solvents in CO_2 capture processes, are categorized as red due to low biodegradability [2]. In a study by Eide Haugmo et al. [2] the alkanolamine N,N-dimethylmonoethanolamine (DMMEA) was the only tertiary amine identified as having acceptable environmental properties (categorized as a "yellow" chemical), and was therefore chosen as a candidate for further investigation in this work.

Having reliable knowledge of the heat of absorption of acid gases into aqueous alkanolamines is an important factor when designing units for acid gas removal. The steam required in the regeneration of the solvent can be directly related to the heat of absorption. A low heat of absorption may be beneficial, however, a low heat of absorption may also mean that the solvent will not receive the full impact of if the increased temperature in the regeneration step [1].

1.1. Heat of absorption

Both MDEA and DMMEA are tertiary amines and therefore do not form stable carbamates. Donaldson and Nguyen [5] proposed a base-catalyzed hydration mechanism for the reaction between carbon dioxide and tertiary amines. The following key reactions take place in aqueous solutions of a tertiary amine and carbon dioxide

Ionization of water

$$2H_2O = H_3O + OH^- \tag{1}$$

Dissociation of carbon dioxide

$$2H_2O + CO_2 = HCO_3^- + H_3O^+ \tag{2}$$

Dissociation of bicarbonate ion

$$H_2O + HCO_3^- = H_3O^+ + CO_3^{2-} \tag{3}$$

Dissociation of protonated alkanolamine

$$RR'R''NH^+ + H_2O = RR'R''N + H_3O^+ \tag{4}$$

The heat of absorption of CO_2 into an aqueous solution of an alkanolamine will then be equal to the heat of reaction from the four key reactions above, combined with the heat of physical dissolution of CO_2 into the solvent.

Because tertiary amines do not form stable carbamates the absorbed CO_2 will primarily be in the form of bicarbonate ion. This gives a lower heat of absorption than what is seen for primary or secondary amines where the formation of a carbamate complex leads to a higher heat of absorption.

In this work the enthalpy change was measured by a direct calorimetric method. Another possibility would be to estimate the enthalpy change from vapor-liquid equilibrium (VLE) data by using equation (5), a form of the Gibbs-Helmholtz equation:

$$\left[\frac{\partial \ln P_{CO_2}}{\partial (1/T)}\right]_{P,x} = \frac{\Delta H_{abs}}{R} \tag{5}$$

As discussed by Kim and Svendsen [6] and Svendsen et al. [7], using this equation has some disadvantages. The uncertainty in the enthalpy of absorption resulting from this equation is increased by a factor of ten compared to the uncertainty in the VLE data. In addition, equation (5) does not allow for investigation of the effect of temperature [6].

The heat of absorption of CO_2 by aqueous MDEA has been measured by several authors. Table 1 gives an overview of previous work and at which conditions these experiments were carried out.

To our knowledge no data on the molar heat of absorption of CO_2 into aqueous DMMEA solutions have been published in the literature.

2. Materials and methods

2.1. Materials

In this work the following chemicals were used; carbon dioxide (CO_2), N-methyldiethanolamine (MDEA, CAS nr. 105-59-9) and N-N-dimethylmonoethanolamine (DMMEA, CAS nr. 108-01-0). The amines were acquired from Sigma Aldrich with a purity of 99%, and were used as received. All solutions were made with de-ionized water.

2.2. Method

The experiments were conducted using a Model CPA 122 reaction calorimeter (Chemisens AB, Sweden). This is the same type of equipment used in the work of Kim and Svendsen [6, 8], Qin et al. [9] and Liu et al. [10] and has been described in detail by Kim and Svendsen [6]. Therefor only a brief description of the equipment and the method employed will be given here. Figure 1 show a schematic of the experimental setup.

This particular calorimeter is a mechanically agitated stainless steel reactor with a volume of 2000 cm^3. The reaction vessel is designed to operate at pressures ranging from 0 to 100 bar and at temperatures between -20 °C to 200 °C. All important parameters, such as heat production, reactor temperature, stirring speed, system pressure and CO_2 flow into the reactor vessel were continuously measured and recorded as a function of time. The experiment consists of measuring the heat generated as a function of time during the isothermal absorption of CO_2. The CO_2 was added from storage cylinders (item 8a and 8b in figure 1) where the temperature and pressure were monitored.

Between 1.2 L – 1.5 L of solution was charged into the preliminary evacuated reaction vessel using the feed bottle (item 7 in figure 1). The feed bottle was weighed before and after the charging procedure. The mechanical stirring was activated and the solution was heated to the experimental temperature.

When reaction temperature and pressure were constant, within ± 0.01 °C and ± 0.01 bar respectively, combined with no heat transfer to or from the reaction system from the circulating heating medium, i.e.

$$\Delta T = \left(T_{in}^{jacket} - T_{out}^{jacket}\right) = \text{constant}$$

the system was considered to be at equilibrium. The system temperature and pressure was noted before CO_2 was fed from storage tanks 8a and 8b (figure 1) to the solution through the bottom valve of the reaction vessel. The gas flow was controlled by a mass flow controller and the flow rate was limited to a maximum of 1 L per minute. The amount of CO_2 added to the solution was determined from a calibrated gas flow meter. The system was then allowed to reach equilibrium before the next loading sequence occurred. The experiment continued until no more CO_2 could enter the reaction vessel and the measured heat flow was close to the baseline. The pressure and temperature in the reactor and the amount of heat transferred between the thermostatting media and the system were recorded. The heat flow was calculated by integrating the heat flow peaks.

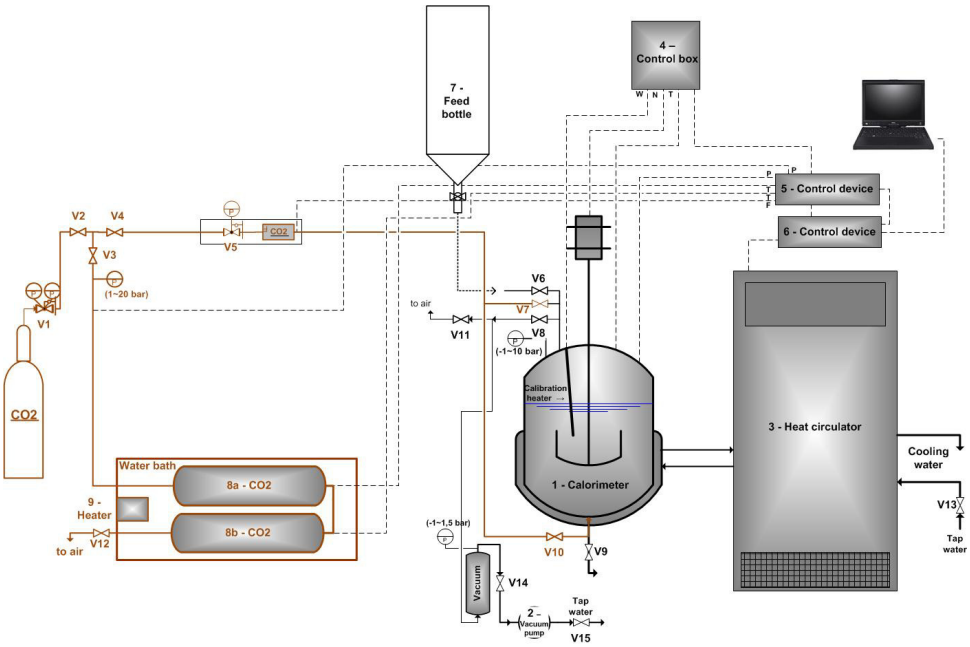

Figure 1. Experimental setup: 1 – Calorimeter; 8a and 8b - CO_2 storage tanks; CO_2 flow controller located after valve 5 (V5)

Table 1. Earlier work on the heat of absorption of CO_2 into aqueous solutions of MDEA.

Reference	Method used	Concentration	T/K
Rayer and Henni (2014) [11]	Setaram C-80 flow calorimeter	5 and 30 wt %	298, 313 and 343
Svensson et al. (2013) [12]	CPA - 202	27 wt %	308, 318, 325 and 333
Arcis et al. (2008) [13]	Setaram C-80 flow calorimeter	15 and 30 wt %	318.5
Kim and Svendsen (2011) [8]	CPA – 122	30 wt %	313
Carson et al. (2000)	In-house displacement calorimeter	10, 20 and 30 wt %	298
Kierzkowska-Pawlak (2007) [14]	CPA – 202	10, 20, 30 and 40 wt %	293, 313 and 333
Oscarson et al. (2000) [15]	In-house isothermal flow calorimeter	20, 35 and 50 wt %	300, 350 and 400
Mathonat et al. (1997) [16]	Setaram C-80 flow calorimeter	30 wt %	313, 353 and 393
Merkley et al (1987) [17]	In-house isothermal flow calorimeter	20, 40 and 60 wt %	288.71, 333.15, 388.71 and 422.04

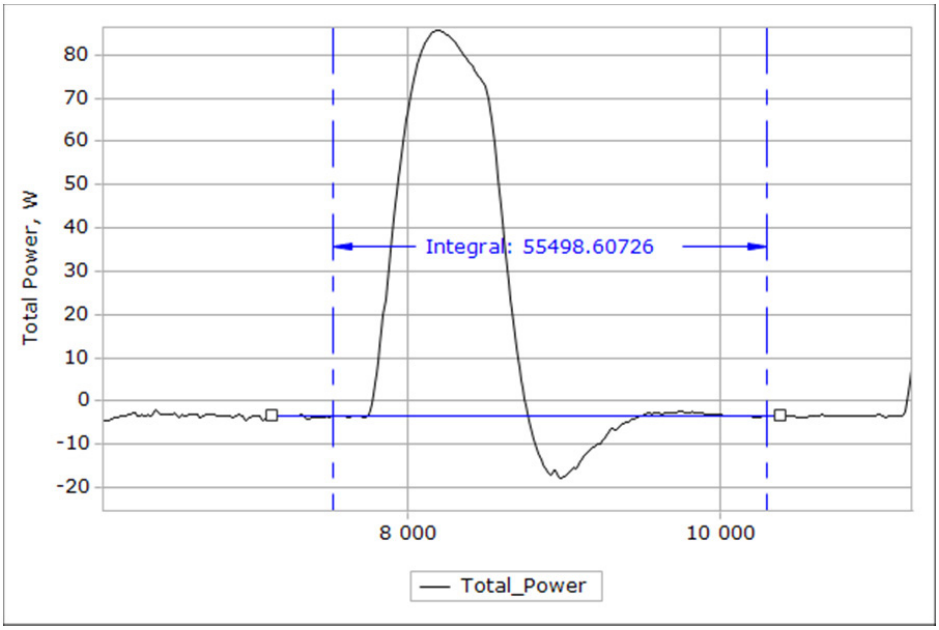

Figure 2. Integration of the heat flux peak in a single loading interval.

3. Results

The heat of absorption of CO_2 into single amine solutions of MDEA and DMMEA was measured in this work. Table 2 lists the different temperatures and concentrations that were investigated.

Table 2. List of temperatures and concentrations tested

Amine	Short name	CAS nr.	Composition [mol/L-solution]	Temp. [K]
N-methyldiethanolamine	MDEA	105-59-9	2.0 and 4.2	313, 353 and 393 (only 2.0 mol/L)
N,N-dimethylmonoethanolamine	DMMEA	108-01-0	2.0 and 4.2	313, 353 and 393

Figure 2 shows an example of the integration of the heat flux. The baseline and integration borders are set manually and are therefore the main source of uncertainty in the data along with determining the amount of CO_2 fed to the reaction vessel. The heat flux peaks and the recorded CO_2 flow peaks were integrated by the trapezoidal method and the baselines were assumed to change linearly. The molar enthalpy of absorption was calculated by taking the ratio of the heat flux to the amount of CO_2 absorbed by the solution in a single interval. The concentration of amine in the liquid phase was assumed constant and equal to the initial concentration of the solution charged into the reaction vessel. The vapor pressure of amine and water in the gas phase was also assumed constant and equal to the vapor pressure before the first interval was measured. This means that the amount of CO_2 in the gas phase could be calculated from the pressure difference in the reactor. This assumption constitutes as a negligible error in the calculation of the molar heat of absorption. In this case the Peng-Robinson equation of state was used to calculate the amount of CO_2 in the gas phase. The experiments in figure 5 were carried out with one year in between them, and except for two points in the low loadings, the reproducibility of the experiments are acceptable.

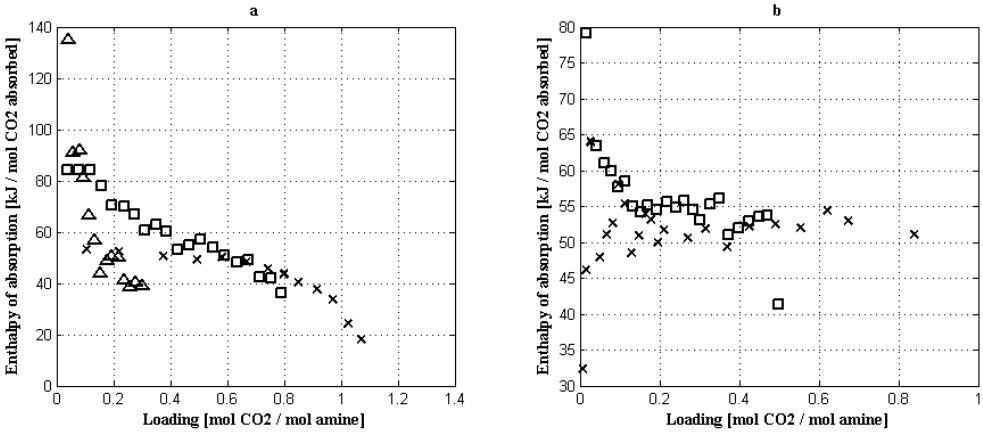

Figure 3. (a) Enthalpy of absorption of CO_2 into a 2M solution of MDEA: (x) 313 K, (□) 353 K and (Δ) 393 K. (b) Enthalpy of absorption on of CO_2 into a 4.2M solution of MDEA: (x) 313 K, and (□) 353 K °C.

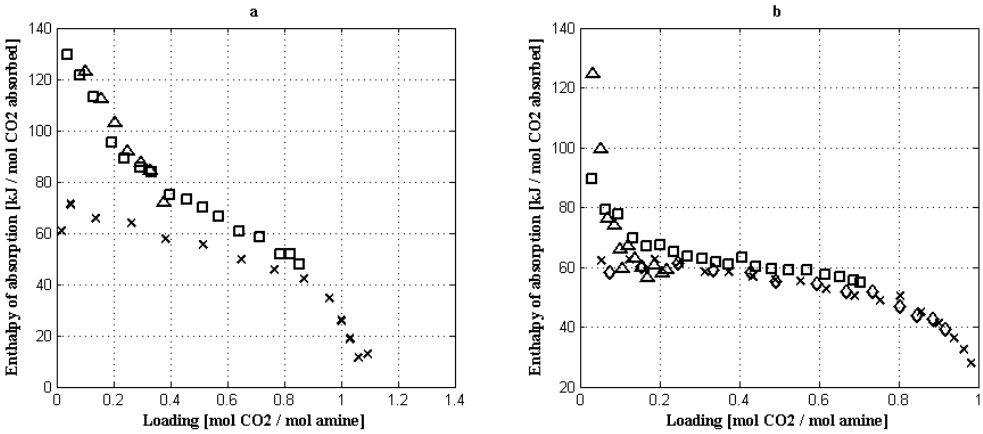

Figure 4. (a) Enthalpy of absorption of CO_2 into a 2M solution of DMMEA: (x) 313 K, (□) 353 K and (Δ) 393 K. (b) Enthalpy of absorption of CO_2 into a 4.2M solution of DMMEA: (x) 313 K, (□) 353 K and (Δ) 393 K.

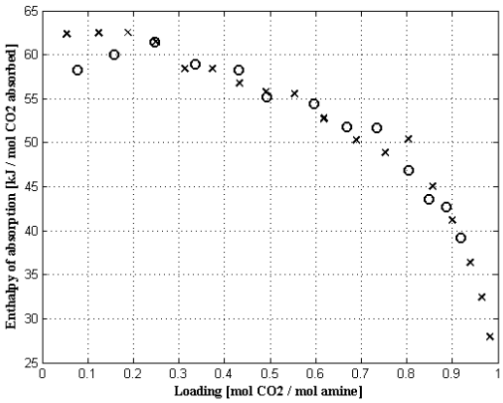

Figure 5. Enthalpy of absorption of CO_2 into a 4.2M solution of DMMEA at 313 K: (x) parallel 1 and (○) parallel 2.

3.1. MDEA

Tabulated data of the enthalpy of absorption of CO_2 into aqueous solutions of MDEA are presented in appendix A. The experimental data are also presented graphically in figure 3. In figure 3(a) the data for the 2M MDEA solution is plotted as a function of the liquid loading of CO_2. At 313 K the differential enthalpy of absorption is close to constant until a loading of approximately 0.7, at which point the $-\Delta H_{diff}$ starts to decrease. The data also show that an increase in temperature increases $-\Delta H_{diff}$ at lower loadings but also that it decreases faster. This means that the increased temperature reduces the capacity of the amine solvent. In figure 3(b) the results for the 4.2M MDEA solutions is shown and at 313 K the enthalpy of absorption is more or less constant except at the lower loadings where there is some scatter in the data. Here also, the effect of temperature is evident, although the effect is small. A weak increase in the heat of absorption with increasing temperature is in line with data published on MDEA from other authors. When comparing the two concentrations there seems to be a larger temperature effect at the lower concentration. The heat of absorptions for MDEA from this work tends to be slightly lower than what other authors have published.

3.2. DMMEA

All the data for the enthalpy of absorption of CO_2 aqueous DMMEA solutions are presented in appendix B. The differential heat of absorption of CO_2 into aqueous solutions of DMMEA is plotted against the CO_2 liquid loading in figure 4. At this moment no literature data has been found to compare with data from this work. However, in figure 6 a comparison between the MDEA data and DMMEA data from this work is shown.

The heat of absorption of CO_2 into 4.2M DMMEA was tested with the same experimental method with one year apart. The results are in good agreement with each other which means that the reproducibility of the experiment is satisfactory.

In figure 4a it can be seen that the heat of absorption for 2M DMMEA increases when the temperature increases from 313K to 353 K. However, the data at 353 K and 393 K are almost identical. The reason for this behavior is unclear. The effect of temperature is also evident in figure 4b for 4.2M DMMEA. At low loadings the effect is quite strong but it diminishes as the loading increases. The effect of temperature seems to be stronger for lower concentrations of amine. This was observed in the experiments with MDEA as well. The heat of absorption for DMMEA is slightly higher than for MDEA at 313 K for both concentrations (figure 6a). However, this difference diminishes as the loading increases indicating that the CO_2 loading has a larger influence on the heat of absorption for DMMEA than for MDEA. When the temperature is increased to 353 K it can be seen (figure 6b) that the difference in heat of absorption between DMMEA and MDEA increases when the concentration of amine decreases. At 393 K (figure 3c) the difference between 2M MDEA and 2M DMMEA increases with increasing loading while the heat of absorption for 4.2M DMMEA is similar to that of 2M MDEA.

4. Conclusion

In this work the heat of absorption of CO_2 into aqueous solutions of MDEA and DMMEA has been investigated in a reaction calorimeter. The experiments were conducted at the concentrations 2M and 4.2M and the temperatures 313 K, 353 K and 393 K. The resulting differential heats of absorption were plotted as a function of liquid CO_2 loading. An effect of temperature on the heat of absorption was observed for MDEA. This effect was larger at low loadings and the lower amine concentration. This was also observed for DMMEA but here the effect of temperature was larger. The heat of absorption for DMMEA was observed to be slightly larger than for MDEA.

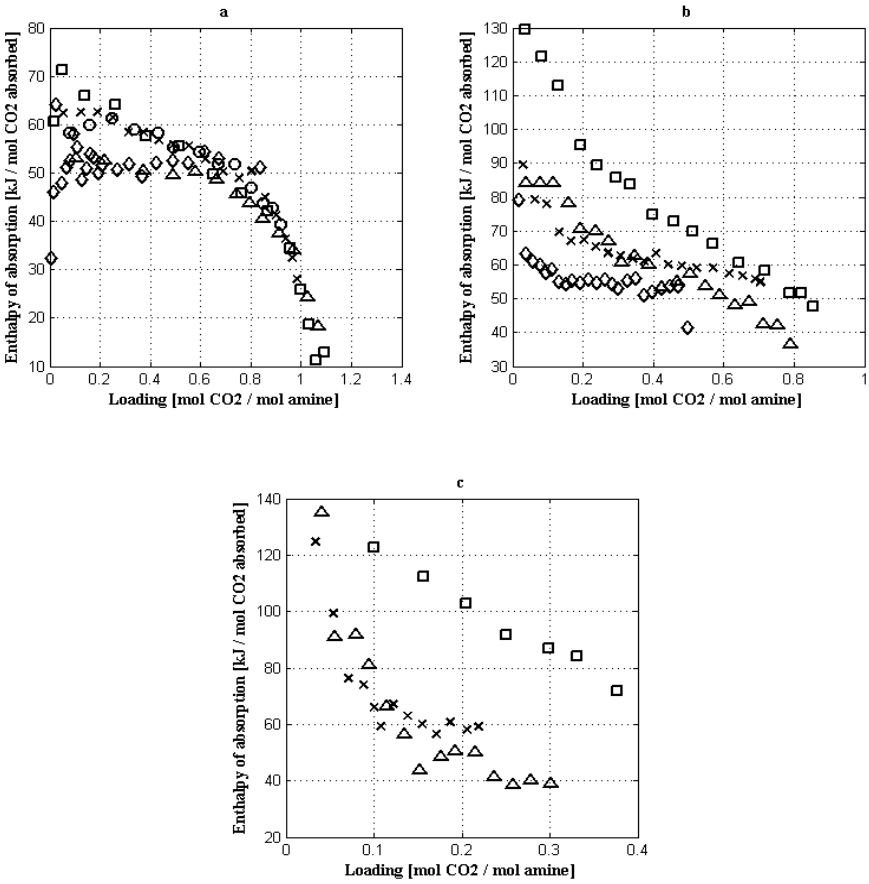

Figure 6. (a) Enthalpy of absorption of CO_2 at 313 K: (□) 2M DMMEA, (x) 4.2M DMMEA(1), (○) 4.2M DMMEA(2), (Δ) 2M MDEA and (◊) 4.2M MDEA. (b) Enthalpy of absorption of CO_2 at 353 K: (□) 2M DMMEA, (x) 4.2M DMMEA, (Δ) 2M MDEA and (◊) 4.2M MDEA. (c) Enthalpy of absorption of CO_2 at 393 K: (□) 2M DMMEA, (x) 4.2M DMMEA and (Δ) 2M MDEA.

Nomenclature

ΔH_{diff}	differential heat of absorption [kJ / mol CO_2]
P_{CO2}	partial pressure of CO_2
R	gas constant
T	temperature

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Appendix A. The heat of absorption of CO_2 into a 2.0M MDEA solution at 313 K, 353 K and 393 K, and a 4.2M MDEA solution at 313 K and 353 K.

2M MDEA			4.2M MDEA		
Amine conc. [wt %]	α [mol CO_2/mol amine]	$-\Delta H_{diff}$ [kJ/mol CO_2]	Amine conc. [wt %]	α [mol CO_2/mol amine]	$-\Delta H_{diff}$ [kJ/mol CO_2]
	313 K			**313 K**	
23.54	0.104	53.053	49.81	0.007	32.401
23.54	0.219	52.435	49.81	0.015	46.111
23.54	0.375	50.610	49.81	0.027	64.015
23.54	0.492	49.460	49.81	0.049	47.914
23.54	0.580	50.281	49.81	0.066	51.157
23.54	0.666	48.509	49.81	0.082	52.623
23.54	0.743	45.640	49.81	0.097	58.089
23.54	0.798	43.711	49.81	0.112	55.379
23.54	0.849	40.532	49.81	0.129	55.379
23.54	0.914	37.552	49.81	0.148	50.972
23.54	0.970	33.895	49.81	0.164	53.990
23.54	1.025	24.169	49.81	0.178	53.132
23.54	1.071	18.165	49.81	0.195	49.962
	353 K		49.81	0.211	51.673
23.56	0.039	84.116	49.81	0.270	50.608
23.56	0.079	84.071	49.81	0.315	51.885
23.56	0.116	84.188	49.81	0.369	49.391
23.56	0.158	78.148	49.81	0.425	52.149
23.56	0.194	70.491	49.81	0.491	52.502
23.56	0.236	69.999	49.81	0.554	51.984
23.56	0.272	66.983	49.81	0.620	54.405
23.56	0.310	60.511	49.81	0.673	53.007
23.56	0.348	62.770	49.81	0.839	51.036
23.56	0.387	60.132		**353 K**	
23.56	0.426	53.293	48.65	0.017	79.204
23.56	0.467	55.066	48.65	0.040	63.340
23.56	0.506	57.166	48.65	0.060	60.988
23.56	0.548	53.818	48.65	0.079	59.898

23.56	0.588	51.037	48.65	0.097	57.722
23.56	0.634	48.187	48.65	0.113	58.520
23.56	0.673	48.933	48.65	0.133	55.030
23.56	0.714	42.371	48.65	0.153	54.235
23.56	0.754	42.082	48.65	0.171	55.210
23.56	0.791	36.321	48.65	0.193	54.598
	393 K		48.65	0.218	55.568
23.56	0.020	306.802	48.65	0.240	54.797
23.56	0.041	134.917	48.65	0.262	55.740
23.56	0.056	90.945	48.65	0.285	54.496
23.56	0.080	91.664	48.65	0.302	53.304
23.56	0.094	80.968	48.65	0.327	55.249
23.56	0.115	66.313	48.65	0.351	56.124
23.56	0.135	56.445	48.65	0.374	51.090
23.56	0.152	43.783	48.65	0.399	51.977
23.56	0.176	48.536	48.65	0.423	52.926
23.56	0.192	50.516	48.65	0.449	53.612
23.56	0.216	50.078	48.65	0.471	53.708
23.56	0.237	41.164	48.65	0.499	41.377
23.56	0.259	38.606			
23.56	0.279	40.029			
23.56	0.301	38.889			

Appendix B. The heat of absorption of CO_2 into a 2.0M DMMEA solution at 313 K, 353 K and 393 K, and a 4.2M DMMEA solution at 313 K, 353 K and 393 K.

2M DMMEA			4.2M DMMEA		
Amine conc. [wt %]	α [mol CO_2/mol amine]	$-\Delta H_{diff}$ [kJ/mol CO_2]	Amine conc. [wt %]	α [mol CO_2/mol amine]	$-\Delta H_{diff}$ [kJ/mol CO_2]
	313 K			**313 K (1)**	
18.07	0.017	60.668	38.14	0.054	62.337
18.07	0.048	71.266	38.14	0.124	62.493
18.07	0.137	65.683	38.14	0.188	62.526
18.07	0.262	64.057	38.14	0.249	61.486
18.07	0.383	57.618	38.14	0.313	58.429
18.07	0.515	55.476	38.14	0.373	58.437
18.07	0.649	49.861	38.14	0.433	56.760
18.07	0.765	45.832	38.14	0.491	55.808
18.07	0.869	42.172	38.14	0.553	55.523
18.07	0.958	34.515	38.14	0.618	52.764

18.07	1.000	25.927	38.14	0.689	50.337
18.07	1.030	18.805	38.14	0.753	48.873
18.07	1.061	11.313	38.14	0.803	50.404
18.07	1.092	12.837	38.14	0.856	45.059
	353 K		38.14	0.900	41.191
18.00	0.036	129.489	38.14	0.939	36.379
18.00	0.082	121.543	38.14	0.964	32.448
18.00	0.130	112.993	38.14	0.982	27.989
18.00	0.193	95.555		**313 K (2)**	
18.00	0.239	89.278	38.60	0.077	58.219
18.00	0.293	85.634	38.60	0.157	60.041
18.00	0.333	83.878	38.60	0.246	61.397
18.00	0.396	74.917	38.60	0.335	58.947
18.00	0.459	72.993	38.60	0.431	58.242
18.00	0.512	69.974	38.60	0.492	55.205
18.00	0.568	66.396	38.60	0.596	54.452
18.00	0.643	60.585	38.60	0.669	51.846
18.00	0.715	58.451	38.60	0.753	51.724
18.00	0.786	51.744	38.60	0.803	46.903
18.00	0.822	51.739	38.60	0.848	43.625
18.00	0.854	47.738	38.60	0.886	42.748
	393 K		38.60	0.919	39.222
18.00	0.045	a		**353 K**	
18.00	0.100	122.830	38.73	0.030	89.475
18.00	0.157	112.349	38.73	0.063	79.200
18.00	0.205	102.730	38.73	0.096	77.853
18.00	0.251	91.810	38.73	0.132	69.801
18.00	0.299	87.112	38.73	0.166	67.166
18.00	0.331	84.083	38.73	0.202	67.283
18.00	0.377	71.935	38.73	0.236	65.270
			38.73	0.271	63.558
			38.73	0.307	62.668
			38.73	0.341	61.726
			38.73	0.373	61.063
			38.73	0.407	63.414
			38.73	0.442	60.106
			38.73	0.481	59.588
			38.73	0.523	59.118
			38.73	0.569	58.996
			38.73	0.615	57.447
			38.73	0.653	56.912
			38.73	0.688	55.666
			38.73	0.704	54.949

	393 K	
38.68	0.017	a
38.68	0.034	124.665
38.68	0.054	99.415
38.68	0.071	76.286
38.68	0.088	73.967
38.68	0.100	65.915
38.68	0.108	59.309
38.68	0.122	67.137
38.68	0.138	62.928
38.68	0.155	59.953
38.68	0.171	56.549
38.68	0.187	60.735
38.68	0.206	57.978
38.68	0.219	59.230

[a]Data point omitted from integration, but loading is still presented in order to show the real loading interval of the next point.

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