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Promoted CO₂ absorption in aqueous ammonia

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Abstract: Aqueous ammonia is a promising solvent for post combustion CO₂ capture but the CO₂ absorption rate in aqueous ammonia alone is low. This short communication investigates the effect of seven additives as potential promoters of CO₂ absorption in aqueous ammonia, and aims to inspire more research to develop effective promoters to increase CO₂ absorption in aqueous ammonia and further advance the aqueous ammonia-based capture process. The additives were monoethanolamine, piperazine, 1-methyl piperazine, 4-amino piperidine and the sodium salts of three amino acids: sarcosine, taurine and glycine. Experiments were performed on a wetted wall column at an absorption temperature of 15 °C using 3 M (mol/L) NH₃ in the presence and absence of 0.3 M of each additive. CO₂ mass transfer coefficients increased to different extents in the presence of different additives. Piperazine, 1-methyl piperazine and sarcosine sodium salt promoted CO₂ absorption in aqueous NH₃ to a greater extent than the other additives.

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Keywords: post-combustion CO₂ capture; aqueous ammonia; NH₃; promoter; amino acid

Introduction

Excess emissions of CO₂ from coal-fired power stations and many other industrial sources are believed to contribute significantly to global warming. Post-combustion capture using chemical solvents is one of the leading technologies for the significant reduction of CO₂ emissions from coal-fired power stations.

Aqueous ammonia is receiving increasing attention as a promising solvent for CO₂ capture.^{1–12} Compared with traditional amines, ammonia has several advantages; it is of low-cost and does not degrade in the presence of O₂ and SO₂ in flue gas. It has a high CO₂

removal capacity and low absorption heat, and hence potentially requires low regeneration energy. It can also capture multiple flue gas components (NO_x, SO_x and CO₂) and be used to produce value-added chemicals, such as ammonium sulfate and ammonium nitrate, which are commonly used as fertilizers.

Most commercially available post-combustion CO₂ capture (PCC) technologies are based on amine solutions, especially monoethanolamine (MEA). However, amine-based processes are reported to reduce power plant efficiency by as much as 30% and involve significant capital investment costs. In addition, the standard aqueous amine solvent has poor SO_x tolerance. This requires a large cut in SO₂ content

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of flue gas prior to CO₂ capture to levels below 10 ppm, and the cost of building a desulfurization unit is substantial. Recently, serious concerns have been raised about the formation of carcinogenic nitrosamines from the use of amines in post-combustion capture and their possible spread into the environment. This is one of the major reasons why the Norwegian government postponed the investment decision on the commercial-scale demonstration of post-combustion capture at Mongstad.¹³

Despite ammonia's advantages over commercially available amine-based solvents, it has a slower rate of CO₂ absorption. Due to its high volatility, aqueous ammonia-based CO₂ absorption must generally take place at temperatures below 20 °C to minimize ammonia loss. This further reduces its CO₂ absorption rate and results in the use of a much larger absorber and thus higher capital costs. Our recent pilot plant trials confirmed the slow absorption rate of aqueous ammonia.¹⁴ To make the ammonia-based process economically feasible, its CO₂ absorption rate must be significantly increased. Limited studies have been published in this area;¹⁵ Alstom has patented a piperazine-promoted chilled ammonia process,¹⁶ but discloses little information.

In this short communication, we report the effect of adding potential promoters of CO₂ absorption to aqueous ammonia solutions. Seven additives were tested: MEA, three amino acid sodium salts (sarcosine, taurine and glycine), and three diamines – piperazine (PZ), 1-methyl piperazine (1-PZ) and 4-amino-piperidine (4-PD).

Amino acid salts are low-cost, environmentally friendly chemicals.¹⁷ They have an ionic structure and are less sensitive to oxygen than amines. These solvents exhibit low thermal sensitivity, meaning that refill requirements are expected to be very low. This has a direct impact on the operating costs of CO₂ capture plants. The solvents' thermal stability also gives more flexibility to the process design; i.e. absorption and desorption can be run at a wide range of temperatures and pressures. Of the diamines, piperazine was chosen because it is commonly used as a promoter in amine and carbonate solutions. Its derivative, 1-methyl piperazine, and another diamine, 4-amino-piperidine, were also tested due to their high CO₂ absorption rates and capacities.^{18,19} Our objective was to show that the addition of small amounts of promoters can significantly increase CO₂ absorption. We hope this short communication inspires more research to develop

effective promoters to increase CO₂ absorption in aqueous ammonia solutions and further advance the aqueous ammonia-based capture process.

Theory

Overall rate constant (k_{ov}) for CO₂ absorption

The reaction of CO₂ with primary and secondary amines (RNH₂), including ammonia, can be described by a Zwitterion mechanism as shown in Reactions 1 and 2.^{20–22}



B represents any base present in the solution and can be molecule of amine, water or a hydroxide ion. Apart from the above reactions, the reaction of CO₂ with a hydroxyl ion can also contribute to CO₂ absorption.



At quasi steady state, the overall chemical rate of absorption of carbon dioxide by amine (r_{CO_2}) is given by:

$$r_{\text{CO}_2} = \frac{C_{\text{CO}_2} C_{\text{RNH}_2}}{1/k_1 + (k_{-1}/k_1)(1/\sum k_B C_B)} + k_{\text{OH}^-} C_{\text{CO}_2} C_{\text{OH}^-} = k_{\text{ov}} C_{\text{CO}_2} \quad (1)$$

where the overall rate constant,

$$k_{\text{ov}} = \frac{C_{\text{RNH}_2}}{1/k_1 + (k_{-1}/k_1)(1/\sum k_B C_B)} + k_{\text{OH}^-} C_{\text{OH}^-} \quad (2)$$

k_1 , k_{-1} , k_B , k_{-B} and k_{OH^-} are rate constants for forward or backward reactions in R1–R3.

Mass transfer coefficients

The CO₂ absorption rate in a chemical solvent can be calculated by:

$$r_{\text{CO}_2} = K_G A (P_{\text{CO}_2} - P_{\text{CO}_2}^*) \quad (3)$$

where r_{CO_2} is the CO₂ absorption rate, mol/s; K_G is the overall gas phase mass transfer coefficient, mol/(s m² kPa); P_{CO_2} is the partial pressure of CO₂ in

the flue gas, kPa; $P_{\text{CO}_2}^*$ is the CO₂ equilibrium partial pressure in the solvent, kPa; A is the effective interfacial surface area, m².

K_G is related to the gas phase mass transfer coefficient, K_G , and liquid phase mass transfer coefficient, k'_G , by:

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{1}{k'_G} \quad (4)$$

As mentioned previously, the gas phase resistance is small and can be negligible under conditions studied, Hence, Eqn (3) can be simplified as $\frac{1}{K_G} \approx \frac{1}{k'_G}$

If the pseudo first-order regime applies to the reaction of CO₂ with amine or ammonia, k'_G can be calculated by:

$$k'_G = \frac{\sqrt{k_{\text{ov}} D_{\text{CO}_2}}}{H_{\text{CO}_2}} \quad (5)$$

where D_{CO_2} and H_{CO_2} are CO₂ diffusivity coefficients and Henry's Law constants in aqueous ammonia or amine solutions.

To evaluate the effect of additives on CO₂ mass transfer coefficients, a 'promotion factor' is introduced, which is defined as follows:

$$\beta = \frac{k'_G - k'_{G,\text{mix}}}{k'_{G,\text{mix}}} \% \quad (6)$$

where $k'_{G,\text{mix}}$ is the expected liquid phase mass transfer coefficient, assuming that there is no interaction between ammonia and additive. k'_G is defined previously and refers to the liquid phase mass transfer coefficient in the mixture, mmol/(s m² kPa).

The expected liquid phase mass transfer coefficient can be calculated as follows:

$$k'_{G,\text{mix}} = \frac{\sqrt{k_{\text{ov,m}} D_{\text{CO}_2,\text{m}}}}{H_{\text{CO}_2,\text{m}}} \approx \frac{\sqrt{(k_{\text{ov,NH}_3} + k_{\text{ov,add}}) D_{\text{CO}_2}}}{H_{\text{CO}_2}} \quad (7)$$

$$k'_{G,\text{mix}}{}^2 = \frac{(k_{\text{ov,NH}_3} + k_{\text{ov,add}}) D_{\text{CO}_2}}{H_{\text{CO}_2}^2} = \frac{k_{\text{ov,NH}_3} D_{\text{CO}_2}}{H_{\text{CO}_2}^2} + \frac{k_{\text{ov,add}} D_{\text{CO}_2}}{H_{\text{CO}_2}^2} = k'_{G,\text{NH}_3}{}^2 + k'_{G,\text{add}}{}^2 \quad (8)$$

$$k'_{G,\text{mix}} = (k'_{G,\text{NH}_3}{}^2 + k'_{G,\text{add}}{}^2)^{0.5} \quad (9)$$

$k_{\text{ov,mix}}$, $k_{\text{ov,NH}_3}$ and $k_{\text{ov,add}}$ are the overall rate constants for the mixture of ammonia with additive, aqueous

ammonia and the aqueous additive solutions, respectively. $H_{\text{CO}_2,\text{m}}$ and $D_{\text{CO}_2,\text{m}}$ are CO₂ Henry's constants and diffusivity coefficients in the mixture of ammonia and additive, respectively. In Eqns (7)–(9) it is assumed that there is no interaction between ammonia and additives, and that the physical and chemical properties, including CO₂ diffusivities (D_{CO_2}) and Henry's constants (H_{CO_2}), are the same in aqueous ammonia, the solution containing additive alone and the mixtures of ammonia and additives. An approach and conclusion used by Darde *et al.*²³ was used to estimate the diffusivity and the Henry's Law constant for CO₂ in aqueous ammonia solution. Considering that the concentration of additives is small and ammonia content in the solution is low, CO₂ diffusivities and Henry's constants in all solvents are expected to be very similar.

Materials and methods

Chemicals

Analytic grade aqueous ammonia with a weight concentration range of 25–30% was purchased from Scharlau (Barcelona, Spain), and the exact ammonia concentration was determined by titration using standard HCl (0.1 M solution, Fluka (Sigma Aldrich, St. Louis, MO, USA)) with a titrator (Mettler Toledo T50). Other chemicals used were: MEA (99%), PZ (99%), 1-PZ (99%), 4-PD (purity is not given and assumed to 100%), ammonium bicarbonate (>99%), glycine (98%), taurine (99%), sarcosine (98%), sodium hydroxide (99%) and 85 wt% orthophosphoric acid (analytic grade). Chemicals were purchased from Sigma Aldrich, except the glycine (from Scharlau) and the orthophosphoric acid (Chemsupply, Gillman, SA, Australia).

Experimental

The CO₂ absorption experiments were performed on a newly built wetted wall column. The column is identical to the one reported previously²⁴ except that

an ammonia scrubber containing concentrated phosphoric acid solution was installed immediately after the column outlet. Ammonia is volatile and will

Table 1. Summary of aqueous ammonia solvents tested.

Solvent	Condition
Fresh NH ₃ solution	NH ₃ concentration = 1, 2, 3, 4 M
Loaded NH ₃ solution	NH ₃ concentration = 3 M, CO ₂ loading = 0, 0.1, 0.3, 0.5
Fresh NH ₃ solution with additive	3 M NH ₃ + 0.3 M additive
Loaded NH ₃ solution with additive	3 M NH ₃ + 0.3 M additive; loading = 0.27, 0.45

inevitably slip to the gas during the wetted wall column experiments. An ammonia scrubber was used to remove ammonia in the gas. This prevented ammonia from dissolving in the water which condensed in the cooler before the gas entered the Horiba gas analyzer and avoided the absorption of CO₂ in the ammonia solution in the cooler. Otherwise a superficially high CO₂ absorption rate will be obtained.

Absorption temperatures for aqueous ammonia in the presence and absence of additives were fixed at 15 °C. A gas flow rate of 5 L/min at 15 °C was used to minimize the gas phase resistance, and solvent flow rate was kept at 100–120 ml/min to produce a laminar and ripple-free flow. The approach adopted by Darde *et al.* was used to evaluate the gas phase resistance, in which 30 wt% fresh MEA was used at 10–30 °C.²³ The results suggested that under the conditions applied, the gas phase resistance is small and can be negligible.

Table 1 lists the ammonia solvents tested in this investigation. Various concentrations of aqueous ammonia solvents were prepared by mixing concentrated ammonia solution with distilled water. Ammonia solvents in the presence of additives at various CO₂ loadings were prepared by mixing the concentrated ammonia solution with ammonium bicarbonate and additives. The amino acids were neutralized with an equimolar amount of NaOH before use. The concentrations of NH₃, promoters and CO₂ are low and the densities of aqueous ammonia solutions studied in this paper are close to that of water at the same temperature. For each measurement, 500 or 1000 ml of solvent was used. Four to six inlet CO₂ partial pressures were used, varying from 0 to 10 kPa. The choice of solvent volume and CO₂ partial pressure was made to ensure that the CO₂

loading change in each measurement was less than 0.01. Plotting r_{CO_2} vs $A(P_{\text{CO}_2} - P_{\text{CO}_2}^*)$ gave a linear curve, and its slope was K_G (Eqn (3)). The log mean of inlet and outlet values of $(P_{\text{CO}_2} - P_{\text{CO}_2}^*)$ was used. For fresh solutions, $P_{\text{CO}_2}^* = 0$, while for CO₂ loaded ammonia solutions with additives, the equilibrium pressure was assumed to equal that for ammonia at the same concentration and CO₂ loading in the absence of additives. It has been found that for the conditions studied, the values of the equilibrium pressures for the solvents investigated affected the y-intercept of the r_{CO_2} vs $A(P_{\text{CO}_2} - P_{\text{CO}_2}^*)$ plot, but had little effect on the value of slope.

In addition, 30 wt% MEA solutions at various CO₂ loadings were tested at 15 °C and 40 °C respectively, to enable comparison with aqueous ammonia and with published results obtained under the same conditions. MEA with different CO₂ loadings was achieved by bubbling CO₂ gas in the MEA solution until the targeted amount of CO₂ is absorbed in the solution.

Results and discussion

The overall gas phase mass transfer coefficients for CO₂ in ammonia and MEA obtained from the wetted wall columns are shown in Fig. 1, along with published coefficients for ammonia obtained from pilot plants for comparison. The experimental conditions of the pilot plant trials can be found in our recent publication.¹⁴ Figure 1 shows that the mass transfer coefficients for MEA obtained by different authors under the same conditions on an identical wetted wall column agree very well. However, this is not the case for ammonia; the mass transfer coefficients obtained in this study are lower than the previous work by Puxty *et al.*²³ at low CO₂ loading. As mentioned in the Experimental section, ammonia slipping to the gas is likely to react with CO₂, causing superficially high mass transfer coefficients. An ammonia scrubber must be used to remove ammonia slipping to the gas, thereby minimizing its influence on CO₂ measurements.

Figure 1 shows good agreement between results obtained from the pilot plant and wetted wall column for the ammonia solvent. This suggests that the mass transfer coefficients obtained from the wetted wall column can be used to estimate those in large-scale packed columns, at least under the conditions

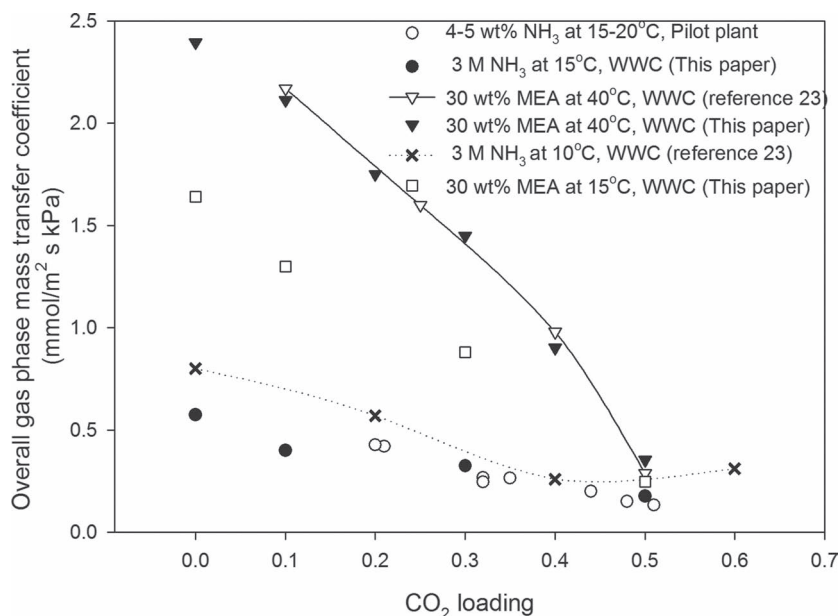


Figure 1. Overall gas phase mass transfer coefficient for ammonia (NH₃) and monoethanolamine (MEA) at a range of CO₂ loadings.

reported here. The mass transfer coefficients for CO₂ in 3 M aqueous ammonia are significantly lower than those for CO₂ in MEA at a typical operating temperature of 40 °C and concentration of 30 wt%. A greater ammonia concentration can be used to increase the mass transfer coefficients, but this approach has limitations. The mass transfer coefficients are roughly

proportional to square root of ammonia concentration, as suggested in Eqn (5), while ammonia loss would increase to a much greater extent with an increase in ammonia concentration, as showed in our pilot plant studies.⁹

Figure 2 shows the mass transfer coefficients for CO₂ in NH₃ (3 M), additive only (0.3 M), and 0.3 M

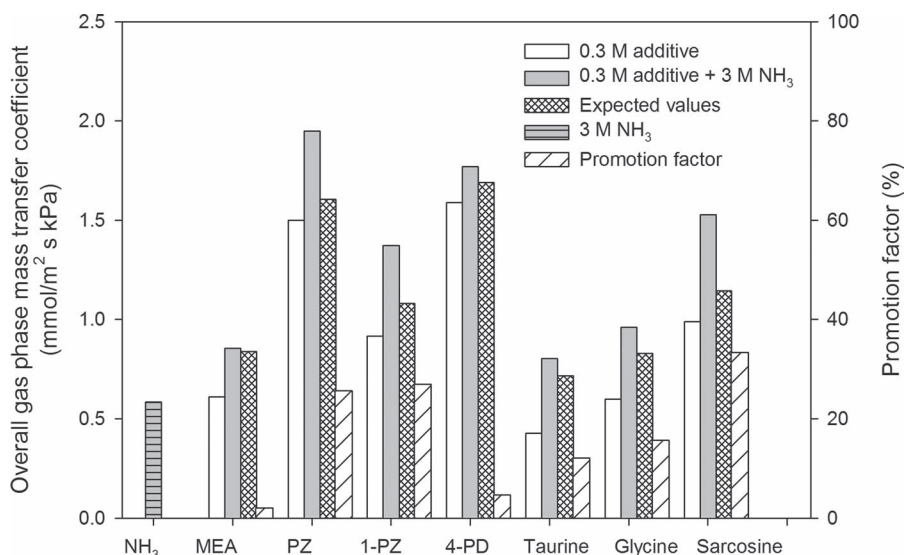


Figure 2. Mass transfer coefficients for CO₂ and promotion factors at an absorption temperature of 15 °C in NH₃ (3 M), additive only (0.3 M), and 0.3 M additive + 3 M NH₃

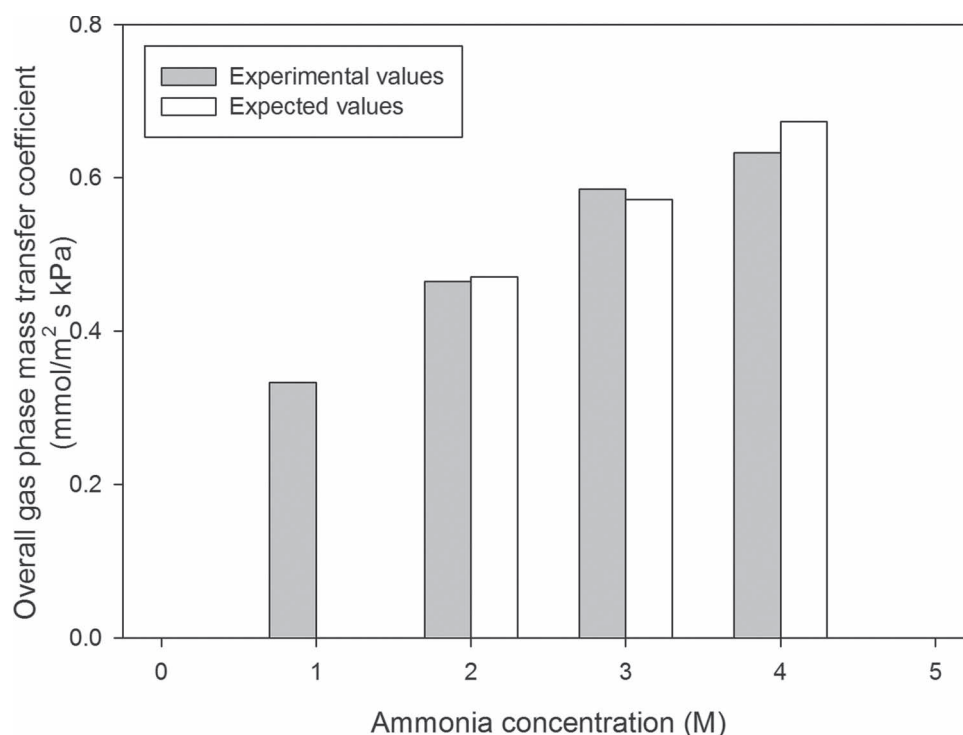


Figure 3. Experimental and expected mass transfer coefficients for CO₂ at an absorption temperature of 15 °C for aqueous ammonia at various concentrations.

additive + 3 M NH₃, with zero CO₂ loading. All additives displayed fast absorption rates. The mass transfer coefficients at a concentration of 0.3 M were higher than for ammonia at 3 M, with the exception of taurine. Introducing a small amount of additives (0.3 M) increased the mass transfer coefficients for the ammonia mixture significantly compared with ammonia itself, and to different extents compared with additive alone. The highest mass transfer coefficients were obtained when PZ, 4-PD and sarcosine were added to the ammonia solution.

Figure 3 illustrates the experimental and expected mass transfer coefficients for aqueous ammonia in the absence of additives. The expected mass transfer coefficients for 2 M are calculated from the experimental values for 1 M solution, 3 M from 1 M and 2 M, and 4 M from 1 M and 3 M, respectively. The close agreement between the experimental and expected mass transfer coefficients and the small β values ($<\pm 6\%$) for the ammonia mixture suggest that the approach is valid for ammonia by applying the 'non-interaction' hypothesis. Figure 2 shows that the mass transfer coefficients for aqueous ammonia in the presence of PZ, 1-PZ and sarcosine are significantly

higher than the expected values, and their β values (promotion factor) are more than 20%. These results suggest that of all the additives tested, PZ, 1-PZ and sarcosine have the strongest interaction with ammonia and thus have the higher promotion factors.

Figure 4 shows the CO₂ mass transfer coefficients in mixtures of ammonia and additives at various CO₂ loadings. As expected, higher CO₂ loadings led to smaller mass transfer coefficients. Generally, mass transfer coefficients for CO₂ in ammonia–additive mixtures were higher under all CO₂ loadings than in aqueous ammonia alone. The coefficients were even larger when additive had higher absorption rates.

PZ-promoted ammonia solution, which has been patented by Alstom,¹⁶ is a highly effective promoter of CO₂ absorption in ammonia. Our research has shown that alternative additives, which are environmentally friendly, can also increase CO₂ absorption in ammonia. It is not clear about the interaction between NH₃ and promoters in the absorption process. Puxty and Rowland used a new software tool to model CO₂ absorption in aqueous PZ, and the mixture of PZ and 2-amino-2-methyl-1-propanol (AMP) in which PZ is regarded as a promoter.²⁵ The tool solves equations

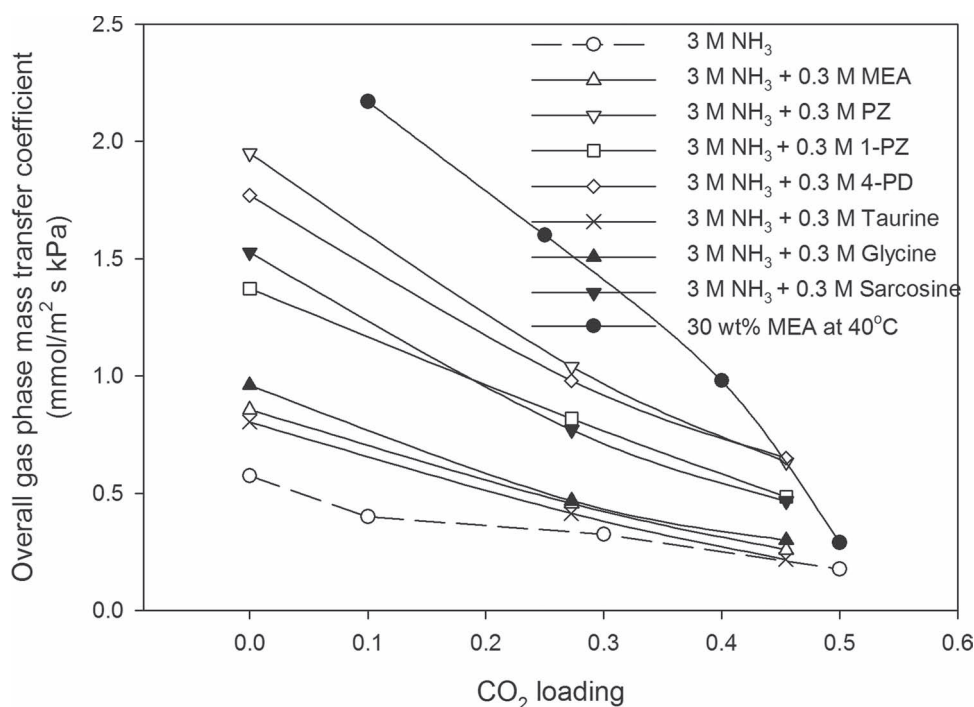


Figure 4. Mass transfer coefficients for CO₂ at various CO₂ loadings obtained from wetted wall column experiments. For 30 wt% MEA, absorption temperature = 40 °C; for other solvents, absorption temperature = 15 °C.

describing diffusion and chemical reactions which combine the chemical reactions and associated parameters determined for single amines. The observed enhanced mass transfer in the mixture can be explained through chemical interactions. As shown in Reaction (2), the zwitterions intermediate exchanges proton with a base which can be PZ or AMP. The role of AMP is that it acts to preferentially accept protons over PZ since AMP is present at high concentrations and accepts a greater proportion of the protons. This allows more free PZ to remain in the solution enhancing mass transfer compared to PZ alone. It could be true for mixture of ammonia and promoters in which NH₃ works as pH buffer. But it does not explain why some promoters perform better than others if the promoter factor is used to assess the enhancing effect (Fig. 2). Research is currently underway to elucidate the role of additives in ammonia-based CO₂ absorption.

Conclusions

Aqueous ammonia absorbs CO₂ at a much slower rate than the standard MEA solution under their respec-

tive operational conditions. This paper investigated seven potential promoters to increase ammonia-based CO₂ absorption. Under all CO₂ loadings studied, aqueous solutions of additives with high CO₂ mass transfer coefficients generally had even higher mass transfer coefficients when mixed with ammonia. The highest mass transfer coefficients were obtained in the presence of PZ and 4-PD. In terms of the promotion effect evaluated by the promotion factor, PZ, 1-PZ and sarcosine sodium salt are three best promoters. This research can potentially facilitate the development of an aqueous ammonia based solvent which can maintain the advantages associated with the aqueous ammonia based post combustion CO₂ capture processes and has CO₂ absorption rates which match MEA-based solvents.

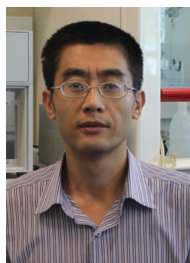
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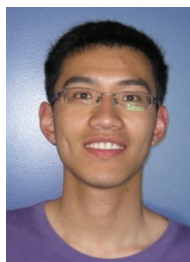
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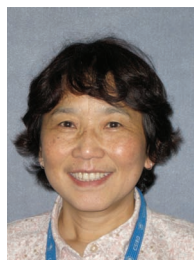
Mengxiang Fang

Prof. Mengxiang Fang got his PhD degree in Engineering Thermal-physics at Zhejiang University in 1991 and since then, he has worked at the Department of Energy Engineering of Zhejiang University. His interest is in CO₂ capture and coal combustion and gasification technology.



Paul Feron

Paul Feron has been leading CSIRO's post combustion capture (PCC) research program, as an OCE Science Leader since June 2007. He is a physicist (Technical University Eindhoven, NL) and obtained a PhD in Water Sciences at Cranfield University (UK).



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