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A novel process concept for the capture of CO₂ and SO₂ using a single solvent and column

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Abstract

The presence of SO₂ in flue gas has a negative impact on typical CO₂ capture processes utilising aqueous amines. For countries like Australia, that do not have flue gas desulfurisation, retrofitting such equipment to existing coal fired power stations is of the order of 100’s of millions of dollars. In this work a new process configuration is described in which SO₂ is absorbed into a fraction of the absorbent used for CO₂ capture. SO₂ absorption is carried out in the bottom of the absorber column into a bleed stream from the bulk solvent, and a recycle flow allows the absorbent to be near saturation in SO₂. This high concentration allows a number of low cost options for sulfur removal from the absorbent such as chemical treatment and precipitation. The principles underlying the concept, have been studied in the laboratory, and a rate based model of an absorber column has been used to model the SO₂ removal column section. The laboratory and modelling results support both the feasibility and utility of the process concept.

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Keywords: carbon dioxide capture; sulfur dioxide; aqueous amine; modelling; chemistry

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1. Introduction

The removal of CO₂ from SO₂ containing gas streams by chemical absorption is an ongoing challenge due to the acidic nature of these two gases. CO₂ absorption is typically done using amines or other bases as solvents. However, SO₂ is the stronger acid and it is absorbed preferentially resulting in an irreversible reduction in CO₂ absorption capacity and rate due to solvent neutralisation and the formation of heat stable salts. As a consequence CO₂ capture processes based on MEA typically require SO₂ concentrations in the gas stream to be below 100 ppm, and for recently developed solvents less than 10 ppm is required. In countries such as Australia where flue gas desulfurisation (FGD) is not used this is a significant problem, and even when FGD is in place it typically only reduces SO₂ concentrations to around 70 ppm. Thus, when no FGD is used a conventional capture process would require its installation, or the installation of some other SO₂ control at significant cost. If FGD is in place, the addition of deep-FGD would be required to achieve the sub-10 ppm concentrations needed for modern CO₂ capture processes.

We have developed a novel process concept that allows CO₂ and SO₂ removal using a single absorber column and a single solvent. The novel process is based on the premise that even when saturated with CO₂ an amine based solvent will preferentially absorb SO₂ at a maximal rate even when it is present at ppm levels. CO₂ is absorbed using the bulk of the solvent in the upper section of the column while SO₂ is absorbed in a fraction of the solvent in the lower section. Absorbed SO₂ is then concentrated in a small fraction of the total solvent which can be recycled to further concentrate the absorbed SO₂. The concentrated SO₂ stream can then be sent for disposal or regenerated. The bulk of the solvent remains SO₂ free. The basis for the process concept relies on an understanding of aqueous SO₂ chemistry and the chemical selectivity of aqueous amines solvents for SO₂ over CO₂. This has been both modelled and validated experimentally in this work.

2. Experimental

Analytical grade sodium sulfite, monoethanolamine (MEA), and hydrochloric acid were used as supplied without further purification (Sigma). The gases used were liquid nitrogen boil off (>99.99% purity), 99.5% carbon dioxide, and 2000 ppm SO₂ in N₂ all supplied by BOC Gases Australia. All solutions were prepared using ultra-pure Milli-Q water that had been boiled for one hour to remove dissolved CO₂ and then stored under N₂.

2.1. S(IV) speciation in aqueous solution

UV spectra were recorded on an Applied Photophysics Chirascan spectrometer in the range of 230-380 nm using a quartz cell of 1.000 cm path length. The spectrophotometric titrations were conducted automatically in the stirred absorption cell using a computer-controlled Metromh 665 Dosimat burette. The titrations were carried out by successive additions of standardised 10 or 1 M hydrochloric acid to known volumes of 0.03, 0.06, 0.10, 0.30, and 0.60 M S(IV) solutions, prepared from a sodium sulfite stock solution. The titrations covered a large range of pH values (from 9.6 to 0.5) and dilution was taken into account. The quartz cell was thermostatted during the titrations at 25.0 ± 0.1°C.

2.2. NMR equilibrium investigations

Equilibrium investigations of MEA and sulfite were conducted at 40 and 100°C. Several sample solutions with a ratio of 2:1 Na₂SO₃:MEA were prepared. Different volumes of HCl were then added to the mixture of the solutions to cover the pH range from 10 to 3. Samples were thermostatted in NMR test tubes in water and oil baths at 40 and 100°C respectively (pressure resistant tubes were used at 100°C). Samples at 40°C were equilibrated for a period of two weeks. Samples at 100°C were equilibrated for over one year with spectra regularly collected during that time to investigate any kinetically slow long term reactions. All ¹H-NMR spectra were recorded using Bruker AVANCE III 600MHz at 25°C.
2.3. Wetted-wall

A gas flow was mixed using three Bronkhorst mass flow controllers. The amount of CO₂, SO₂ and N₂ in the gas flow was varied to achieve CO₂ partial pressures in the range 0 to 20 kPa and SO₂ gas concentrations in the range 0 to 800 ppmv. The gas was passed through a 1/8” stainless steel coil and saturator containing 0.1 M sulphuric acid (to avoid SO₂ absorption), which were both immersed in a thermostated water bath (Techne). From the coil and saturator the gas then flowed past the outside of the wetted-wall column, entering at the bottom and being exhausted at the top. The exhaust gas was directed through a membrane dryer and gas conditioner to remove moisture from the gas stream prior to entering a Horiba VA3000 CO₂ and SO₂ analyser. A membrane dryer was used in lieu of a condenser to avoid SO₂ removal by the condensate. The system was maintained at atmospheric pressure and determination of CO₂ and SO₂ partial pressure in the column took into account the water content of the gas phase. The temperature of the column was maintained by circulating liquid from the water bath through a jacketed cover.

600 mL of the test solution was placed in a Pyrex bottle in the water bath. A large solution volume relative to the gas-liquid contact area was chosen to ensure that during experiments the total CO₂ and SO₂ loading of the solution did not change significantly. The solution was pumped in a loop through the wetted-wall column and back to the bottle using a gear pump (Micropump GA-V21) at a rate of 220 mL·min⁻¹. This liquid flow rate was chosen to ensure continuous and ripple free film formation. A needle valve and a syringe filled with extra solution were used to provide liquid level control in the system. A full schematic diagram of the complete apparatus is given in Figure 1.

3. Results and discussion

3.1. Reactions in aqueous solution

In order to determine the speciation of S(IV) in aqueous solution, a comprehensive study of the protonation of sulfite was carried out by UV spectrophotometric titrations covering a wide range of pH and different initial concentrations of S(IV). Titrations were performed by stepwise addition of a known concentration of hydrochloric acid to solutions of sodium sulfite followed by recording the spectrum of the solution after thorough mixing. From these measurements the relevant protonation equilibrium constants were determined [1].

The similar chemical formulas suggest that the chemistry of SO₂ should have similarities with that of CO₂, but there are also significant differences. In terms of similarities, both react with water to form the corresponding sulfurous and carbonic acids, H₂SO₃ and H₂CO₃ respectively. The equilibrium constants for the formation of these
acids from the dissolved gases are low. Both H$_2$SO$_3$ and H$_2$CO$_3$ are diprotic acids and deprotonate to form the bisulfite and bicarbonate ions, HSO$_3^-$ and HCO$_3^-$, and sulfite and carbonate ions, SO$_3^{2-}$ and CO$_3^{2-}$, respectively. At the relatively high pH conditions of absorbents used in CO$_2$ capture, both dioxides also react directly with hydroxide to form HSO$_3^-$ and HCO$_3^-$.

In terms of differences, sulfurous acid is a much stronger acid than carbonic acid, and bisulfite is a much stronger acid than bicarbonate. As a result at a pH of 8-10 that is found in a typical CO$_2$ absorbent the speciation is different with the major species being HCO$_3^-$ and SO$_3^{2-}$ respectively. Another major difference is the kinetics of reaction with H$_2$O and OH$. Both reactions are orders of magnitude faster for SO$_2$ than for the CO$_2$. Further, in the desorber the deprotonation of H$_2$CO$_3$ is partially reversed and CO$_2$ is formed and released. However there is no formation of SO$_2$ under these conditions and thus SO$_2$ accumulates in the capture process. The net reaction of SO$_2$ in aqueous amine solutions is to form sulfite with the release of two protons. When exposed to oxygen sulfite will oxidise to sulfate (SO$_4^{2-}$) which is a stronger acid and less soluble than its S(IV) precursor, but otherwise has no impact [2].

The relevant reactions described so far are presented in the equations (1) to (5). Note that X is either S or C in these equations.

\[
XO_2(aq) + H_2O \xrightleftharpoons[k_{1, k_1, K_1}]{k_{1, k_1, K_1}} H_2XO_3
\]  
(1)

\[
XO_2(aq) + OH^- \xrightleftharpoons[k_{2, k_2, K_2}]{K_2} HXO_3
\]  
(2)

\[
XO_3^{2-} + H^+ \xrightleftharpoons[K_3]{K_3} HXO_3
\]  
(3)

\[
HXO_3^- + H^+ \xrightleftharpoons[K_4]{K_4} H_2XO_3
\]  
(4)

\[
XO_2(aq) \xrightleftharpoons[K_H]{K_H} XO_2(g)
\]  
(5)

In addition to the above reactions there is an additional reaction that is specific to aqueous solutions of bisulfite. It is known that dimerisation of bisulfite (6) occurs at pH values of between 6 and 2 when bisulfite is the major species in the solution. This is relevant to this work as much lower pH values may be obtained in the fraction of solvent used for SO$_2$ absorption. Its equilibrium constant was also determined from the UV spectrophotometric titrations [1].

\[
2HSO_3^- \xrightleftharpoons[k_{5r, k_5r, K_5}]{K_5} S_2O_3^{2-} + H_2O
\]  
(6)

The rate constants and Henry constant for X = S were taken from literature [3-5]. The rate and equilibrium constants and Henry constant for X = C were also taken from literature [6].

3.2. Reactions with amine

The most common CO$_2$ capture absorbents contain amines in aqueous solution. CO$_2$ reacts directly with primary and/or secondary unhindered amines to initially form carbamic acid, which immediately deprotonates to carbamate. The reaction of amine with CO$_2$ is fast and thus leads to rapid absorption of CO$_2$, but it is also detrimental as the released proton will be absorbed by a second amine with a net ratio of 2:1 amines per absorbed CO$_2$. The reactions for MEA, the most commonly used amine, are given in equations (7) to (9). The rate and equilibrium constants were taken from literature [7].

\[
CO_2 + MEA \xrightleftharpoons[k_{6, k_6}]{k_{6, k_6}} MEACOOH
\]  
(7)

\[
H^+ + MEACOO^- \xrightleftharpoons[K_7]{K_7} MEACOOH
\]  
(8)
As part of this work it has been investigated if SO$_2$ undergoes the equivalent reactions to (7) and (8) under CO$_2$ capture relevant conditions. SO$_2$ in gas or liquid form reacts with neat amines to form thionylamines (R-N=S=O) [8], however little detail is available about what happens in aqueous solution. To investigate this MEA was equilibrated with sulfite solutions at different pH, adjusted by addition of different amounts of hydrochloric acid. No sign of formation of sulfurous acid amide was observed when held at 40°C for a period of two weeks. However, it is possible that these types of reactions are very slow at low temperature and occur with faster kinetics at higher temperature desorber conditions. In order to investigate the occurrence of these reactions at high temperatures similar experiments were conducted by equilibrating samples in pressure resistant NMR tubes at 100°C in an oil bath for more than one year, and recording their $^1$H-NMR spectra regularly. NMR spectroscopy results at high temperature did not show any interaction between sulfur species and amine.

3.3. Mass transfer of SO$_2$ and CO$_2$

As a consequence of the $^1$H-NMR study it was found that no direct reaction occurs between SO$_2$ and MEA at either absorber or desorber conditions. This can be assumed to be the case for other amines as well as they share similar chemistries'. Thus only equations (1) to (6) are relevant for SO$_2$. SO$_2$ reacting with H$_2$O and OH$^-$ ((1) and (2) respectively) and HSO$_3^-$ dimerisation (6) are defined by forward and reverse reaction rate constants. The protonation reactions can be considered instantaneous on timescales relevant for this work, and are defined by equilibrium constants. (5) is the solubility of gaseous SO$_2$ dissolved in water defined according to Henry’s law, and this is also defined as an equilibrium. The interplay between all of these processes, and the physical properties of the absorbent, govern the rate of SO$_2$ mass transfer. The same physical properties and equivalent processes, with the addition of a carbamate formation reaction and exclusion of dimerisation, govern the rate of CO$_2$ mass transfer.

To assess the rates of mass transfer of SO$_2$ in the presence of CO$_2$ a mass transfer model based on the numerical solution of partial differential equations describing diffusion and chemical reaction of all species was used. The model has been described in detail previously for modelling CO$_2$ mass transfer into a falling thin film [9]. The same model as described for 4.9 mol·dm$^{-3}$ (30 wt%) MEA has been used here with the addition of equations (1) to (6) for SO$_2$. The diffusion coefficient of SO$_2$ at 40°C in 4.9 mol·dm$^{-3}$ MEA was determined using the Wilkie-Chang correlation ($1.67\times10^{-9}$ m$^2$·s$^{-1}$) [10]. Based on the larger reaction rate constants and greater solubility for SO$_2$ it would be expected that SO$_2$ mass transfer is favoured over CO$_2$. This is borne out in the modelling results shown in Figure 2. The solid lines are model calculations of the CO$_2$ and SO$_2$ absorption flux as a function of CO$_2$ loading (mol CO$_2$ / mol MEA). As the CO$_2$ loading increases the flux of CO$_2$ absorption falls. SO$_2$ absorption is predicted to be unaffected by the presence of CO$_2$ in the liquid even at a near saturated CO$_2$ loading of 0.5.

Wetted-wall measurements were also made at the same conditions as the model predictions using the wetted-wall setup and procedure described in the experimental section. These are given as markers in Figure 2 and agree well with the modelling results. The mass transfer of SO$_2$ is unaffected by the presence of absorbed CO$_2$, indicating that absorption is highly selective for SO$_2$. 

\[
\text{H}^+ + \text{MEA} \rightleftharpoons \text{MEAH}^+ \quad (9)
\]
Figure 2 Model calculated (lines) and measured (points) values of the CO₂ and SO₂ absorption flux into a falling thin film of 4.9 mol·dm⁻³ MEA at 40°C, 100 ppmv SO₂(g) and 10 kPa CO₂(g) as a function of CO₂ loading.

Figure 3 shows the SO₂ flux as a function of SO₂ gas phase concentration for a number of different conditions. The striking result is that even at the highest CO₂ loading of 0.5 and highest CO₂ gas partial pressure of 20 kPa the SO₂ flux is unaffected. Also shown in this figure is the CO₂ flux as a function of SO₂(g) concentration for a CO₂ loading of 0.1 and 20 kPa CO₂. The CO₂ flux falls as the SO₂(g) concentration increases. This further highlights the selectivity towards SO₂ absorption even in the presence of a large (~2000×) excess of CO₂(g).
4. Process concept and modeling

4.1. Process concept

The process concept is one that allows both CO₂ and SO₂ removal from a gas stream using a single absorber tower and single aqueous amine absorbent. The process utilises the differences in physical solubility, absorption rate and acidity of CO₂ and SO₂ already outlined to achieve this. A schematic of the process is shown in Figure 4. Flue gas enters at the bottom of a packed absorber column. An aqueous amine absorbent suitable for CO₂ capture such as aqueous MEA, and lean in CO₂ and SO₂, enters at the top of the absorber column (blue liquid stream) and the flue gas stream at the bottom. As the gas stream moves up the column SO₂ absorption occurs in the bottom section. This section of column is exposed to a small part of the total absorbent stream which entered at the top of the absorber. Even though the absorbent is CO₂ rich at this point effective SO₂ removal still occurs due to the selectivity for SO₂. Some CO₂ desorption may occur in this section increasing the CO₂ content of the gas stream. The SO₂ lean gas stream then moves into the mid and upper sections of the column. In this part of the column CO₂ absorption occurs as in a traditional CO₂ capture process. At the interface between the mid-top section of the column and the bottom section, most of the now CO₂ rich but SO₂ lean absorbent is removed for CO₂ stripping and absorbent regeneration (red liquid stream). The remainder passes into the bottom section of the column (bleed stream) where SO₂ removal occurs. The fraction of the remainder of the total process liquid stream needed to provide bulk capture of SO₂ will depend on the ratio of SO₂ / CO₂ content in the flue gases and typically will range between 0.01 % and 3% of the total process stream needed for CO₂ capture. Given this much reduced flow, it is likely that it will be recirculated multiple times in the bottom section of the column to provide adequate contact (recycle stream). Alternatively a
dedicated gas/liquid contactor able to operate at high gas/liquid ratios, such as a membrane contactor, might be used. The SO2 rich absorbent at the bottom of the column (green liquid stream) is then removed for sulfur recovery and absorbent regeneration. The regenerated absorbent streams are then mixed and returned to the top of the absorber.

CO2 stripping and absorbent regeneration for the CO2 rich and SO2 lean absorbent stream (red liquid stream) would be carried out using a standard CO2 desorption process. Sulfur recovery and absorbent regeneration of the SO2 rich stream (green liquid stream) would be carried out using a sulfur recovery process suitable for use with aqueous amines and a concentrated SO2 stream. Due to oxidation of sulfite species as a result of the presence of oxygen in the flue gas both sulfites and sulfates may be present in this stream. The traditional way to recover the sulfur and regenerate the absorbent is using a thermal reclaim. By concentrating the sulfur into a small solvent volume the efficiency of reclamation can be improved by minimising the water and amine content relative to the sulfur. Further the energy lost to reclamation can be minimised by introducing the steam and amine vapour into the CO2 stripper to act as stripping gas. Alternatively, other lower cost options can also be considered such as precipitation with limestone, membranes, ion-exchange and biological sulfate removal.

![Figure 4 Process concept in which SO2 absorption occurs in the bottom section of the absorber column into a fraction of the total solvent inventory.](image)

### 4.2. Model predictions

A previously developed rate-based process model implemented in Matlab® [11] has been used to predict the behaviour in the bottom section of the column where SO2 absorption is to occur. The model was based on 30 wt% aqueous MEA as an absorbent. The absorbent chemistry was extended to include the reactions of equations (1) to
(6) for SO$_2$ as equilibria. The gas phase was extended to include SO$_2$ using the same binary gas model with mass transfer assumed to be entirely gas-side controlled (as was demonstrated in the wetted-wall measurements).

The model was run using the dimensions of the absorber column and typical operating parameters for CSIRO’s CO$_2$ capture pilot plant at Loy Yang [12], as given in Table 1. However a packed height of only 0.308 m was used to represent the bottom section of the column. The initial CO$_2$ loading in the liquid stream entering from the upper section of the absorber was set to 0.51 and it was set to 0.40 in the recycle stream. The solvent in the recycle stream was also assumed to contain 3.4 wt% SO$_2$. The bleed stream liquid flow rate from the upper section of the column where CO$_2$ absorption occurs was set at 0.64% of the total flow rate (total flow = 0.117 kg·s$^{-1}$) and the recycle stream adjusted to maintain the same total flow rate as the upper section. These values have been chosen to represent an example set of operating conditions, but do not constitute optimal values. Calculated profiles of the SO$_2$ and CO$_2$ content in the gas and liquid phases are shown in Figure 5. What is most apparent is that little CO$_2$ mass transfer occurs while essentially all of the SO$_2$ is absorbed.

In this scenario essentially 100% of the SO$_2$ in the gas stream is removed, which is comparable to the 98% that is removed at CSIRO’s Loy Yang pilot plant using a separate pre-treatment column containing 32 wt% sodium hydroxide [12]. The bleed stream represents the rate of accumulation of solvent concentrated in SO$_2$. In this case that is $7.5 \times 10^{-4}$ kg·s$^{-1}$ which corresponds to 65 kg / day, while the plant captures approximately 250 kg CO$_2$ / day.

The SO$_2$ content in this accumulated solvent is 0.55 mol·dm$^{-3}$ or 35 g·dm$^{-3}$. A number of low cost options exist that can reduce the sulfate concentration to mM levels that could be used in isolation or in combination. Mineral precipitation involves the addition of lime and limestone and precipitation of calcium sulfate which can reduce the sulfate concentration to 1-2 g·dm$^{-3}$. More advanced precipitation processes can achieve < 0.1 g dm$^{-3}$ residual sulfate. Membrane techniques, ion-exchange resins and biological methods are also options [13]. These methods vary in capital costs between USD 210-670 / m$^3$ / day and operating costs between USD 0.1-0.88 / m$^3$ / day [13]. Assuming a linear scale-up, a full scale plant treating 1 million tonnes of CO$_2$ per year would have approximately 712 m$^3$ / day of solvent concentrated in sulfur to treat. Taking median values this would be a capital and operating cost of USD 313,280 and USD 349 / day respectively. Although the suitability of all of these methods for use with aqueous amine solutions has not been assessed, crystallisation as potassium sulfate has been demonstrated to be effective for sulfate removal from aqueous MEA solution [14]. Although the absorber would also require some modifications, the total cost will still be much less than the hundreds of millions required for installation and operation of separate flue gas desulfurisation.

Table 1 Parameters used to define the model of the bottom column section where SO$_2$ absorption occurs.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Height</td>
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</tr>
<tr>
<td>Diameter</td>
<td>0.211 m</td>
</tr>
<tr>
<td>Packing</td>
<td>16 mm Pall rings</td>
</tr>
<tr>
<td>Bleed Stream Liquid Flow Rate</td>
<td>$7.5 \times 10^{-4}$ kg·s$^{-1}$</td>
</tr>
<tr>
<td>Recycle Stream Liquid Flow Rate</td>
<td>0.116 kg·s$^{-1}$</td>
</tr>
<tr>
<td>Liquid Inlet Temperature</td>
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</tr>
<tr>
<td>Gas Flow Rate</td>
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</tr>
<tr>
<td>Gas Inlet Conditions</td>
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</tr>
<tr>
<td>Total Pressure</td>
<td>103 kPa</td>
</tr>
</tbody>
</table>
Figure 5 Bottom of column where SO$_2$ absorption occurs for the conditions described in Table 1 with a bleed flow of 0.64% total flow and recycle flow of 0.07% total flow.
5. Conclusions

In this work an SO₂ tolerant CO₂ capture process has been proposed that provides for simultaneous removal of CO₂ and SO₂ from flue gas using a single absorber column and aqueous amine absorbent. The process concept involves using a modified absorber column in which SO₂ is absorbed in a bleed stream from the main absorbent flow in the bottom of the column. There is also a recycle flow allowing a small volume of the absorbent to be concentrated in SO₂, which both increases the options for and reduces the cost of SO₂ removal. For a full scale plant median capital and operating costs are estimated at USD 313,280 and USD 349 / day, with some small additional expenses required for absorber modification. This is compared to 100’s of millions for the installation of separate flue gas desulfurisation.

The process concept has been investigated through laboratory studies of the interaction between SO₂ and MEA, mass transfer measurements and modelling of the SO₂ removal section of an absorber column. Long term ¹H-NMR experiments demonstrated no detectable reaction between MEA and SO₂. Thus, the existing known aqueous chemistry of SO₂ reflects the chemistry of absorption into aqueous amine solutions. Mass transfer experiments were completed using a wetted-wall column that demonstrate the kinetic selectivity of SO₂ over CO₂, even when the absorbent is saturated in CO₂ and with CO₂ present in the gas phase some 2000× in excess. A rate based model incorporating all relevant MEA, CO₂ and SO₂ chemistry was then used to predict SO₂ removal using the bottom fraction of CSIRO’s Loy Yang pilot plant absorber column. The modelling results support the viability of the concept.

6. Acknowledgements

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7. References

