



GHGT-12

Modelling and experimental study of SO₂ removal and NH₃ recycling in an ammonia based CO₂ capture process

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Abstract

The high NH₃ loss to the gas stream (NH₃ slip) is one of the major issues in the ammonia based CO₂ capture technology. Meanwhile, the removal of sulphur dioxide pollutant (SO₂) from flue gas is a prerequisite for many CO₂ capture processes. Flue gas desulphurization (FGD) is not installed in Australian power plants and significant capital/investment costs are required to install FGD. In this study, we proposed an advanced process configuration to combine SO₂ removal and NH₃ recycling in one process development and solve these two problems together. A rate-based model for the system of NH₃-CO₂-SO₂-H₂O was established and employed to simulate the process flow sheet. The temperature, pH and N/S ratio profile, SO₂ removal and NH₃ recycling efficiency along the column were analysed. Experimental work using a bubble column was carried out to provide an initial test on the technical feasibility of the SO₂ and NH₃ absorption process. Both the modelling and experimental results suggest that the proposed process results in excellent removal and recovery of SO₂ and NH₃.

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1. Introduction

Carbon dioxide (CO₂) and sulphur dioxide (SO₂) are two of the main harmful emissions from coal-fired power stations, causing serious environment problems such as climate change and acid rain. Adequate measures should be taken against the emissions of these two gases. The chemical absorption is considered as the most cost effective and feasible option for post combustion capture of CO₂ and SO₂ [1]. Aqueous ammonia (NH₃) as a promising solvent, has gained growing attention in the past decades, since it provides several advantages over the conventional amine-based solvents, including a high CO₂ loading capacity, low corrosiveness and no absorbent degradation, a low regeneration energy and potential for simultaneous capture of flue gas acidic gases (CO₂, SO₂, NO₂, HCl, and HF)

[2]. The technical feasibility of the NH_3 based CO_2 capture technology has been successfully proven in the pilot and demonstration plants conducted by Alstom power company [3, 4, 5] and Commonwealth Scientific and Industrial Research Organization (CSIRO) [6,7]. Specifically, for the representative Alstom's chilled ammonia process (CAP), it is reported that the CO_2 capture efficiency can be maintained at 90% with a CO_2 product purity of over 99.5 % using 28 wt% chilled NH_3 solution and the net efficiency loss to the power station is calculated to be potentially 8.5% net efficiency loss if the CAP capture process is added onto the power station. CSIRO in collaboration with Delta Electricity conducted the pilot plant trials using a slipstream of the flue gas from Munmorah Power Station. The results show that using a 5 wt% NH_3 solutions, an 80-90% CO_2 removal efficiency and more than 99% CO_2 product purity were obtained

However, the aqueous NH_3 based capture technology is still hindered by the technical problem of NH_3 slip due to the high NH_3 volatility and the corresponding economic problem of NH_3 recovery during the CO_2 capture process. The suppression of NH_3 volatility therefore is of primary importance for the further development of this technology for commercial application. The available approaches to resolving the issue of NH_3 slip include: (1) suppression by introducing NH_3 suppressants [8, 9]; (2) recovery by water scrubbing and regeneration [10], (3) utilization by producing fertilizers such as sulfate and nitrate [11]. In the Alstom's chilled ammonia process (CAP), the NH_3 abatement system consists of an NH_3 water wash and an NH_3 stripper to capture and recycle the vaporized NH_3 , but this process requires refrigeration equipment for solvent and gas cooling and a large energy consumption for NH_3 regeneration and the production of cooling water, leading to a significant increase in capital and operation costs [10]. Powerspan company has developed a patented ECO multi-pollutant control system integrated with the ECO_2 process, which reuses the vaporized NH_3 to remove SO_2 pollutant and produce the value added sulphur fertilizers [12]. However, some unresolved issues are still associated with this ECO process. For example, the amount of vaporised NH_3 is substantially greater than the small amount of SO_2 in the flue gas and it is not clear what to do with the excessive NH_3 in the process. So far a detailed study of SO_2 recovery using slipped NH_3 has not been reported.

In the present study, we introduced an advanced SO_2 removal and NH_3 recycle system to the aqueous NH_3 based CO_2 capture process. A simple but effective process configuration was proposed to realize the combined SO_2 removal and NH_3 recovery. This process is of particular interest to Australian power stations as desulphurization is not implemented in Australia. In the previous work, we have investigated the NH_3 abatement and recycling process using the same process configuration [13]. This study was based on the previous research findings and extended to the combined SO_2 removal and NH_3 recycling. The combination of modeling work using commercial software Aspen Plus[®] and experimental work using bubble column was carried out to test the technical feasibility of the proposed SO_2 removal and NH_3 recycle process.

2. Methodology

2.1. Process modeling

The combined SO_2 removal and NH_3 recycling process is depicted in Figure 1, which consists of an NH_3 wash column, a pretreatment column and a CO_2 absorber. This process utilizes the heat contained in high temperature flue gas and takes advantage of the high SO_2 and NH_3 solubility in water. The process is described briefly as follows: (1) the NH_3 absorber is installed at the exit of CO_2 absorber to capture volatilized NH_3 (12 000 ppm) using washing solution; (2) the NH_3 -rich wash water is then pumped to the pretreatment column where the liquid is heated by the high temperature flue gas (120 °C) to release the captured NH_3 to the CO_2 absorber (NH_3 recycle), while the SO_2 pollutant in the flue gas (200 ppm SO_2) is quickly absorbed in wash water to form ammonium sulphite/bisulphite ($\text{NH}_4\text{HSO}_3/(\text{NH}_4)_2\text{SO}_3$) (SO_2 capture); (3) the lean- NH_3 washing water is circulated back to the NH_3 absorber starting another cycle of NH_3 recycle and SO_2 removal until the $\text{NH}_4\text{HSO}_3/(\text{NH}_4)_2\text{SO}_3$ concentration reaches the saturated state. The saturated solution will be removed from the system for producing sulphur fertilizers. The make-up water is introduced to the top of wash column to maintain the water balance in the system. As the washing solution absorbs the basic NH_3 , the pH of circulated solution will be over 7. The process modelling of SO_2 and NH_3 absorption was carried out under the optimized operating conditions determined in our previous study [13]: 350 kg/hr solvent circulation rate, 10 °C wash solution, wash column size $\varnothing 0.5\text{m}\times 3.0\text{m}$, pretreatment column size $\varnothing 0.5\text{m}\times 3.0\text{m}$, 5 °C temperature approach of the heat exchanger between hot inlet and cold outlet streams.

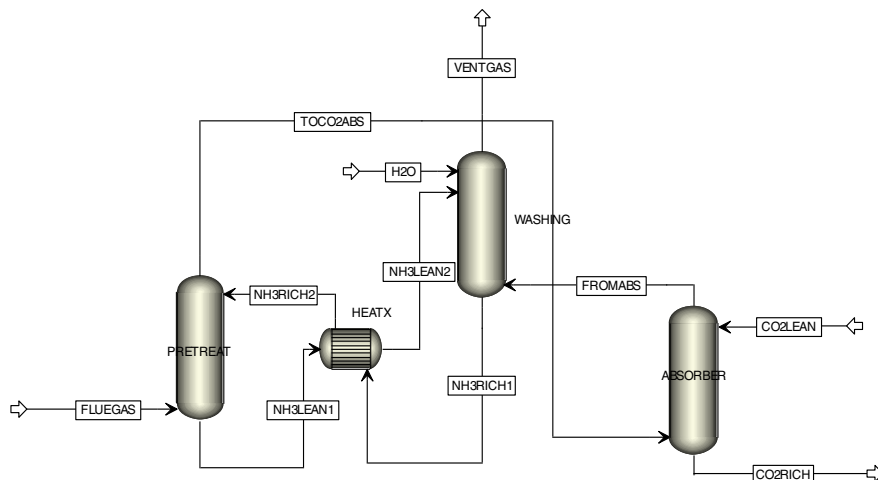


Figure 1 Process flow-sheet diagram of the SO₂ removal and NH₃ recycling process

The commercial simulator Aspen Plus[®] was used to simulate the process and evaluate its technical feasibility. The PITZER model was used to calculate the fugacity coefficient, Gibbs energy, Enthalpy, speciation in the liquid phase and vapour-liquid equilibrium of the NH₃-SO₂-CO₂-H₂O system. NH₃, CO₂, SO₂, N₂, O₂ were defined as Henry components and the corresponding Henry constants were retrieved from the Electrolytes Expert System. The binary interaction parameters for electrolyte pairs were automatically retrieved from the Aspen Plus databank.

The model for the combined SO₂ removal and NH₃ recycle process was based on the following hypotheses: (1) There is no reaction between SO₂ and CO₂ in the liquid phase and gas phase, so the model of NH₃-CO₂-SO₂-H₂O system could be adequately described by combining the characteristics of system NH₃-CO₂-H₂O and system NH₃-SO₂-H₂O; (2) The SO₂ absorption process by NH₃ is thermodynamically controlled; (3) The SO₂ in the flue gas and HSO₃²⁻/SO₃²⁻ in aqueous solution were not oxidised by the O₂ during the absorption process.

Table 1 lists the possible reactions of system NH₃-CO₂-SO₂-H₂O. The corresponding equilibrium constants of reactions (1)-(6) were regressed from experimental data [14] and the constants of reactions (7) and (8) were computed from Gibbs Energies built in Aspen databank, respectively [15]. The kinetic parameters were obtained from the work of Pinsent et al [16].

Table 1 Equilibrium and kinetic reactions in the system of NH₃-CO₂-SO₂-H₂O

No.	Reaction type	Reaction
1	Equilibrium	$\text{NH}_3 + \text{H}_2\text{O} \leftrightarrow \text{NH}_4^+ + \text{OH}^-$
2	Equilibrium	$2\text{H}_2\text{O} \leftrightarrow \text{H}_3\text{O}^+ + \text{OH}^-$
3	Equilibrium	$\text{HCO}_3^- + \text{H}_2\text{O} \leftrightarrow \text{CO}_3^{2-} + \text{H}_3\text{O}^+$
4	Equilibrium	$\text{H}_2\text{O} + \text{HSO}_3^- \leftrightarrow \text{H}_3\text{O}^+ + \text{SO}_3^{2-}$
5	Equilibrium	$2\text{H}_2\text{O} + \text{SO}_2 \leftrightarrow \text{H}_3\text{O}^+ + \text{HSO}_3^-$
6	Equilibrium	$\text{NH}_4\text{HCO}_3(\text{s}) \leftrightarrow \text{NH}_4^+ + \text{HCO}_3^-$
7	Equilibrium	$(\text{NH}_4)_2\text{SO}_3(\text{s}) \leftrightarrow 2 \text{NH}_4^+ + \text{SO}_3^{2-}$
8	Equilibrium	$(\text{NH}_4)_2\text{SO}_3(\text{s})/\text{H}_2\text{O} \leftrightarrow 2 \text{NH}_4^+ + \text{SO}_3^{2-} + \text{H}_2\text{O}$
9	Kinetics	$\text{CO}_2 + \text{OH}^- \rightarrow \text{HCO}_3^-$
10	Kinetics	$\text{HCO}_3^- \rightarrow \text{CO}_2 + \text{OH}^-$

11	Kinetics	$\text{NH}_3 + \text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{NH}_2\text{COO}^- + \text{H}_3\text{O}^+$
12	Kinetics	$\text{NH}_2\text{COO}^- + \text{H}_3\text{O}^+ \rightarrow \text{NH}_3 + \text{CO}_2 + \text{H}_2\text{O}$

2.2. Experimental

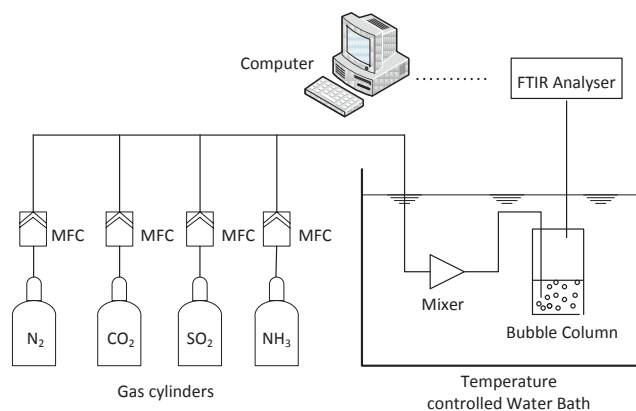


Figure 2 Schematic diagram of experiment apparatus

The SO_2 removal and NH_3 absorption experiments were carried out separately in a bubble column to help validate the simulation results. The schematic flow-sheet diagram of the experiment setups is described in Figure 2. Carbon dioxide, Nitrogen, sulphur dioxide and NH_3 were supplied from gas bottles with a purity of CO_2 (99.8 %), N_2 (99.9 %), SO_2 (1.0 % in N_2) and NH_3 (1.06 % in N_2), respectively. This enabled the supply of different CO_2 , SO_2 and NH_3 concentrations in the feed gas. The flow rates of the gases were controlled by mass flow controllers (Bronkhorst). The total gas flow rate was fixed at 5.0 L/min in the SO_2 removal experiments and 3.0 L/min in the NH_3 absorption experiments with a gas NH_3 concentration of 2000 ppmv. The gases went through a mixer before reaching the bubble column. Both mixer and bubble column were placed in the water bath to ensure that the gas temperature was close to the solvent temperature before the gas mixture entered the bubble column. The simulation results to be presented in the following section showed that the major species in the wash water is ammonium sulphite (NH_4) $_2$ SO_3 . Therefore the ammonium sulphite monohydrate ((NH_4) $_2$ $\text{SO}_3 \cdot \text{H}_2\text{O}$, 92% purity from Sigma Aldrich) was used to prepare (NH_4) $_2$ SO_3 solutions in the experiments to simulate the real process. The gas mixture was dispersed at the bottom of the bubble column and contacted the solvent with a short residence time. The FTIR gas analyzer (GasetmTM Dx-4000) was used to determine the gaseous CO_2 , SO_2 , NH_3 concentration before and after absorption. The SO_2 and NH_3 removal efficiency can be expressed as the following equation.

$$\eta\% = \frac{C_{i,\text{inlet}} - C_{i,\text{outlet}}}{C_{i,\text{inlet}}} \times 100\%$$

where η is the removal efficiency; i represents the component SO_2 or NH_3 ; $C_{i,\text{inlet}}$ is the inlet concentration of component i , ppmv; and $C_{i,\text{outlet}}$ is the outlet concentration of component i , ppmv.

3. Results and discussion

3.1. Modelling of SO_2 removal and NH_3 recycling

Figure 3 (a) shows the NH_3 reuse efficiency and SO_2 removal efficiency as a function of number of cycles. It is evident that a 99.9% NH_3 reuse and a 99.9% SO_2 removal efficiency were achieved. The SO_2 was absorbed by the solution and accumulated in forms of (NH_4) $_2$ SO_3 , and NH_4HSO_3 . As shown in Figure 3(b), the (NH_4) $_2$ SO_3 in the

SO₂-rich solution was always the dominant species and increased gradually with the increasing cycles while the NH₄HSO₃ remained at relatively very low level. This is because the SO₂ absorption process by aqueous NH₃ was conducted at pH over 7 under conditions studied and the alkaline environment facilitated the generation of SO₃²⁻ species. The results confirmed that in the experiment it is reasonable to use the ammonium sulphite solutions to test the SO₂ and NH₃ absorption. The concentrated sulphur-containing solution is then expected to undergo a further treatment, e.g. ammonium sulphate/sulphite fertilizers.

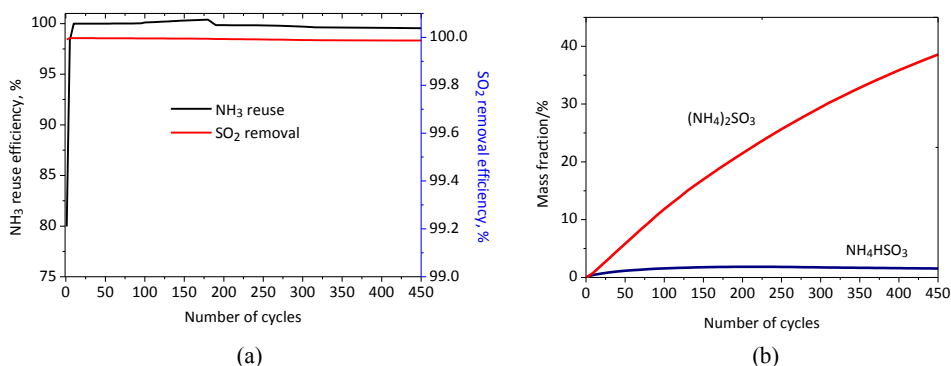
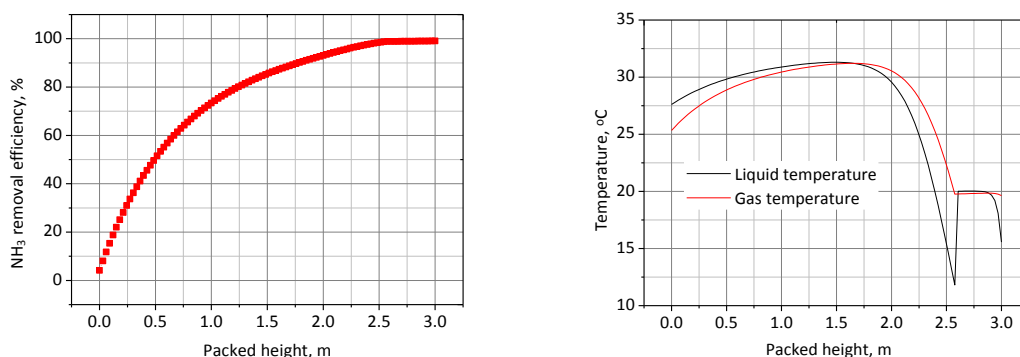


Figure 3 (a) NH₃ reuse efficiency and SO₂ removal efficiency and (b) concentration of major SO₂ containing species after pretreatment column as a function of number of cycles

3.1.1 Wash column

To gain insight into how the NH₃ is removed, the wash column profiles with respect to NH₃ removal efficiency and temperature were modelled. The vaporised NH₃ from the CO₂ absorber was scrubbed in the wash column using the circulated ammonium sulphite solution. As shown in the Figure 4 (a), the NH₃ removal efficiency increased along the packed column and the value can reach over 99.9% under studied conditions. This means that almost all the slipped NH₃ was scrubbed in the solution in the forms of free NH₃ and NH₄⁺ ion species.

It is well known that the NH₃ absorption by solution is an exothermic process leading to an overall temperature increase of the solution (Figure 4 (b)). This will reduce the driving force for NH₃ absorption and subsequently lower the NH₃ solubility in the solvent. The low temperature is required for the NH₃ absorption process, as the low temperature can improve the NH₃ solubility in the solvent. In this simulation, 10 °C wash water was used to obtain a high NH₃ removal efficiency. The liquid temperature profile along the column experienced two peaks. One was caused by the exothermic process of NH₃ absorption by the wash solution. The other was caused by the 10 °C fresh makeup water that was introduced into the top of washing column. The makeup water played two roles: One was to maintain the H₂O balance of the system; the second was to achieve a very high rate of NH₃ removal. The temperature ranged between 10 °C to 32 °C, which provided the reference for the following actual experiment.



(a) (b)
Figure 4 (a) NH₃ removal efficiency and (b) temperature profile as a function of packed height

3.1.2 Pretreatment column

After the slipped NH₃ was absorbed in the wash column, the solution was sent to the pretreatment column which played three significant roles in the proposed SO₂ removal and NH₃ recycle process. The first was to cool down the high temperature flue gas arriving from the power station. As shown in Figure 5 (a), the flue gas temperature experienced a sharp decrease after contact with the relatively low temperature solvent along the packed column. The outlet gas temperature from the pretreatment column decreased to 43.6 °C and the gas could be directly transported to the CO₂ absorber without further cooling. Our simulation has confirmed that flue gas temperatures from 15 °C to 50 °C had little influence on the CO₂ absorption process including absorption rate and NH₃ vaporization rate. This is due to the fact that the latent heat in the high temperature flue gas has been released and transferred to the solvent, which led to a decrease of flue gas temperature and a small heat content.

The second role was to remove the SO₂ pollutant in the flue gas. The result in Figure 5(b) shows that SO₂ removal efficiency increased sharply in the first 1.0 m packed height, and then stabilized at 99.9% with the further increasing packed height. This can be accounted for by the pH profile along the column (Figure 5(c)). In the SO₂ absorption process, the basic solution which contained ammonium sulphite and free NH₃ was used to scrub the SO₂. The solution can quickly absorb SO₂ due to the fast reaction rate between SO₂ and H₂O, and the generated acid species HSO₃⁻ was quickly neutralized by the OH⁻ in the base solution, resulting in the drop of solution pH as the solvent flowed from the top to the bottom.

The third role was to recycle the scrubbed NH₃ to the CO₂ absorber by making use of the latent and sensible heat in the flue gas. As shown in Figure 5(b), the NH₃ recycling efficiency increased steadily along the column and reached the maximum of 99.9% at the top of column. In the pretreatment column, the solution was heated by the high temperature flue gas and released the NH₃ vapor. Figure 5(d) describes the N/S ratio profile in the liquid phase (the molar ratio of N-containing species to the S-containing species) as a function of packing height. Initially, the solution at the column top contained the free NH₃ and had a high N/S ratio. With the liquid falling down along the column, the ratio decreased gradually due to the release of molecular NH₃ from the solution. The vaporized NH₃ was then recycled back to the CO₂ absorber for re-capturing CO₂. It is worth mentioning that the N/S ratio can be below 2.0 at the bottom stage. This implies that all the free NH₃ was recycled and part of (NH₄)₂SO₃ was decomposed to release the NH₃ vapor. The decomposition of (NH₄)₂SO₃ occurring at the column bottom was partly attributed to the high temperature flue gas in the bottom stage (Figure 5(a)).

In summary, the multi-function pretreatment column acted as: (1) a cooler to cool down the high temperature flue gas; (2) a heater to recycle almost all the escaped NH₃ to the CO₂ absorber; and (3) an efficient desulphurization facility to achieve a high rate of SO₂ removal. Accordingly, this advanced process would hold the advantages of (1) saving the energy consumption for flue gas cooling; (2) reducing the energy and capital cost for the NH₃ recovery system; and (3) simplifying the flue gas desulphurization (FGD) process which is particularly important in Australia as FGD systems are not installed in any Australian power plants.

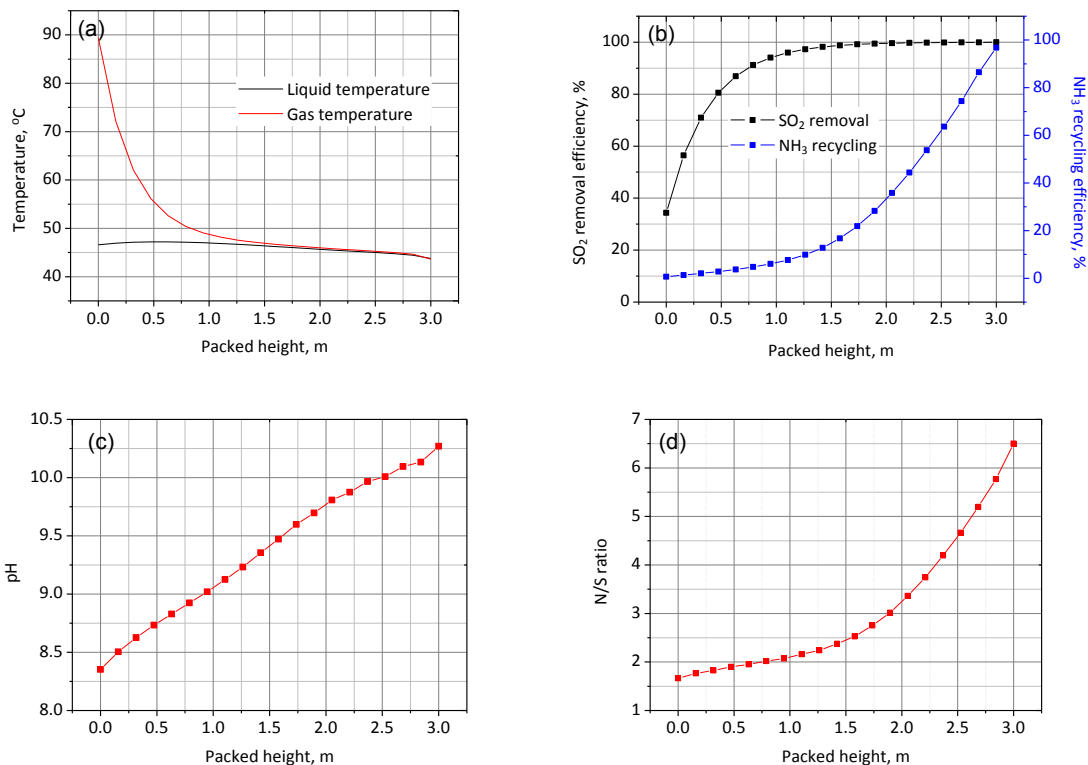


Figure 5 (a) liquid and gas temperature profile, (b) SO₂ removal efficiency and NH₃ recycling efficiency, (c) solution pH profile, (d) N/S ratio profile as a function of the packed height

3.2. SO₂ and NH₃ absorption experiments

Based on the modelling results, the experimental work using bubble column was conducted to provide an initial verification on the prediction results, including SO₂ absorption and NH₃ absorption by aqueous (NH₄)₂SO₃ solution.

3.2.1 NH₃ absorption

As discussed in section 3.1.1, the temperature range along the length of the column during the NH₃ absorption process varied between 10 °C and 32 °C. Therefore 10 °C and 25 °C were selected to represent the temperature swing during this absorption process. From Figure 6 (a), it is evident that the NH₃ removal efficiency decreased with the increasing (NH₄)₂SO₃ concentration. NH₃ scrubbing efficiency was able to maintain over 85% at 10 °C, while at 25 °C the absorption efficiency dropped significantly especially at high (NH₄)₂SO₃ concentrations. This is mainly because of the NH₃ partial pressure increasing with an increase in the (NH₄)₂SO₃ concentration (Figure 6 (b)). As a result, less NH₃ was absorbed and more NH₃ came out from the column. The gas NH₃ concentration at the outlet of the bubble column ranged from 40 to 693 ppmv which cannot satisfy the limiting requirements for NH₃ emission [2]. However, it should be mentioned that the bubble column used in the experiment allowed only a very short contact time between gas and liquid phase. If a packed column with a high surface area was used for the NH₃ absorption, a higher NH₃ absorption efficiency is likely to be achieved.

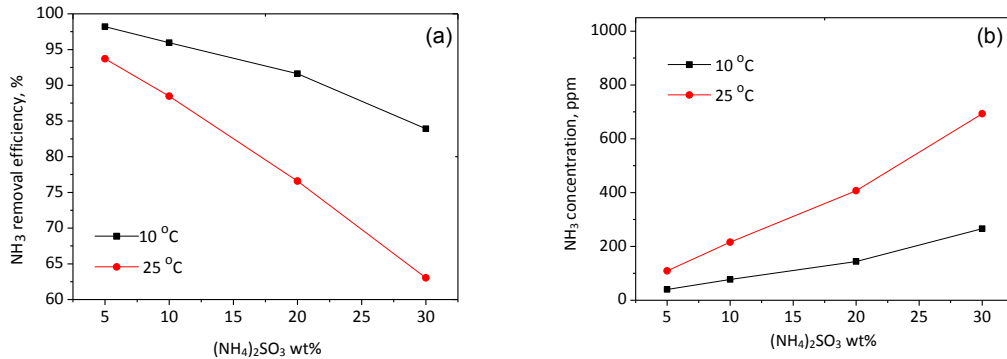


Figure 6 Effect of $(\text{NH}_4)_2\text{SO}_3$ concentration and temperature on the (a) NH_3 removal efficiency and (b) NH_3 concentration in the column outlet

3.2.1 SO_2 absorption and outlet NH_3 concentration

The modelling results in Figure 5 suggest that the SO_2 absorption process was performed in the liquid temperature range of 42–48 °C. In the experiment, the temperature of 40 °C was used to test the SO_2 absorption process. For a comparison, 25 °C was also included. The SO_2 inlet concentration varied from 58 ppmv to 833 ppmv, which covers the typical SO_2 level in the flue gas from Australian coal-fired power stations [1]. As shown in Figure 7(a), the $(\text{NH}_4)_2\text{SO}_3$ solution had excellent sulphur removal for the high SO_2 level flue gas, while a relatively low SO_2 removal efficiency for the low SO_2 level flue gas. During the experiment, it was observed that the outlet SO_2 concentration from bubble column varied little from 15 ppmv to 20 ppmv despite the inlet SO_2 concentration having increased from 58 ppmv to 833 ppmv. This means the ammonium sulphite solution has a high SO_2 absorption capacity. It is worth mentioning that the FTIR gas analyser has an SO_2 detection limits of <20 ppmv where the SO_2 concentration measurement is likely to be influenced by the other gas component present in the gas such as moisture. So it is difficult to determine the SO_2 concentration accurately at a concentration below 20 ppmv and the experimental results with low SO_2 level may be not valid.

Figure 7(b) shows the effect of $(\text{NH}_4)_2\text{SO}_3$ concentration on the NH_3 vaporization at different temperatures. At high temperature and high $(\text{NH}_4)_2\text{SO}_3$ concentration, the NH_3 vaporized drastically indicating that part of $(\text{NH}_4)_2\text{SO}_3$ was decomposed to release the gas NH_3 . However, it was observed that the measured SO_2 concentration at the column outlet was always less than 10 ppm (out of detection range). This indicates that the SO_2 in the $(\text{NH}_4)_2\text{SO}_3$ solution was very stable and difficult to be released even at high temperature. This phenomenon was consistent with the modelling results, which is beneficial for the NH_3 recycle and SO_2 removal in the real process. The experiments proved the concept of the combined capture process and demonstrated the feasibility of SO_2 removal and NH_3 recycling through the application of the proposed process configuration.

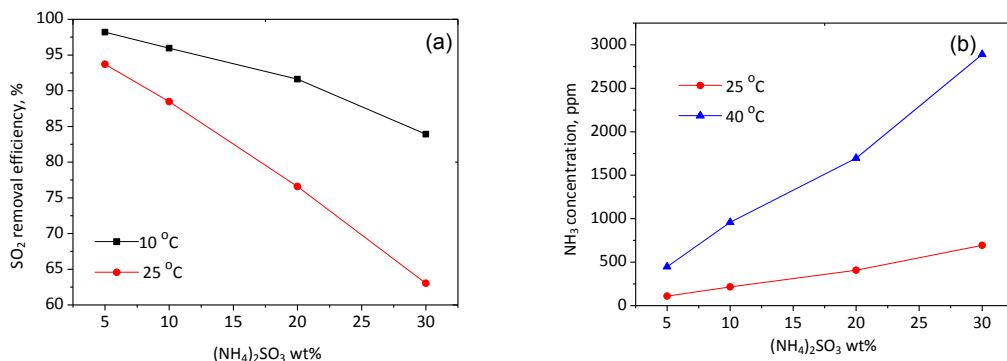


Figure 7 Effect of $(\text{NH}_4)_2\text{SO}_3$ concentration on the (a) SO_2 removal efficiency and (b) NH_3 concentration in the column outlet

4. Conclusion and ongoing work

The combined process of SO₂ removal and NH₃ recycling was modelled using Aspen Plus. The modelling results showed that more than 99.9% of the SO₂ in the flue gas can be removed and over 99.9% of the slipped NH₃ from the CO₂ absorber can be recycled by the application of the proposed process configuration. The experiment using a simple bubble column was carried out to provide a preliminary test of the technical feasibility of the combined SO₂ removal and NH₃ recycling process. The (NH₄)₂SO₃ solution had a high NH₃ absorption capability at low temperature and a high SO₂ removal efficiency at high inlet SO₂ concentration. The experimental results qualitatively confirmed the simulated results and the technical feasibility of the combined SO₂ removal and NH₃ recycling process.

Future work will focus on model validation including the thermodynamic model and the rate based model using laboratory or pilot plant results. The validated model can help to identify conditions under which SO₂ is selectively removed in preference to CO₂ by NH₃ (flue gas pre-treatment) and conditions under which NH₃ can be fully recovered by SO₂ solution (flue gas post-treatment). The combined SO₂ removal and NH₃ recycling process will be integrated with a typical CO₂ capture plant. Further work is also required to determine the potential problems of the combined SO₂ removal and NH₃ recycling process, such as the oxidation of SO₃²⁻ to SO₄²⁻ and the formation of aerosols as a result of NH₃ volatility.

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