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Simulating combined SO₂ and CO₂ capture from combustion flue gas

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ABSTRACT. The requirement to pre-treat flue gas prior to the CO₂ capture step is an economic challenge when using aqueous amine absorbents for capturing CO₂ from coal-fired power station flue gases. A potentially lower cost alternative is to combine the capture of both the CO₂ and SO₂ from the flue gas into a single process, removing the requirement for the desulfurization pre-treatment step. The CSIRO's CS-Cap process uses a single aqueous amine absorbent to capture both of these acid gases from flue gas streams. This paper covers the initial simulation of this process applied to both brown and black coal flue gases. Removal of absorbed SO₂ is achieved via reactive crystallization. This is simulated here using a 'black box' process, resulting in a K₂SO₄ product. Different operating conditions have been evaluated that increase the sulfate concentration of the absorbent in the SO₂ capture section of the process, expected to increase the efficiency of the reactive crystallization step. This paper provides information on the absorption of SO₂ into the

amine solution, and heat and mass balances for the wider process. This information will be required for further detailed simulation of the reactive crystallization step, and economic evaluation of the CS-Cap process.

1.0 Introduction

The most technologically advanced method for capturing CO₂ from combustion flue gases is to use aqueous solutions of amines and amine blends. This technology is currently being employed at two commercial-scale CO₂ capture plant: The Shell-Cansolv process at SaskPower's Boundary Dam Power Station, and Mitsubishi Heavy Industry's KM-CDR process at the NRG-owned WA Parish Power station. One of the challenges when using amine-based absorbents for CO₂ capture is the requirement to treat the flue gas prior to the CO₂ capture plant. This includes cooling the flue gas, removing particulate, and removing other acid gas components present in the flue gas, such as SO₂. This is because SO₂ will bind more strongly with the absorbents than CO₂, and is not released under the conditions used to strip CO₂ from the rich absorbent solution. This leads to a build-up of sulfate salts in the absorbent, reducing its ability to capture CO₂.

In most countries, flue gas desulfurization (FGD) is employed on power stations to lower the SO₂ content in the flue gas. However, the inlet requirements for amine-based CO₂ capture plant are likely to be more stringent than current emissions regulations. This means that either an additional spray bank will need to be added to existing FGD units, or a polishing step (e.g. caustic scrubber) will be required. Another option is to develop a process that removes both SO₂ and CO₂ from the flue gas stream as a combined capture process. Combined capture processes have the potential to be cheaper overall than a conventional amine-based process with FGD. Examples include amino acid processes (such as Siemen's PostCap), ammonia and potassium

carbonate (which remove SO₂ as ammonium and potassium sulfate), and also some amine-based processes (e.g. Shell Cansolv). The CSIRO have been developing an amine-based combined capture process, CS-Cap. This concept uses a single amine solution to capture both the SO₂ and CO₂ from the flue gas stream, as outlined in Figure 1. The CS-Cap process essentially consists of separate SO₂ and CO₂ capture loops. The CO₂ capture loop follows the standard liquid absorbent-based CO₂ capture process. In the SO₂ capture section, a slip stream of the CO₂ rich absorbent is recirculated to capture the SO₂ from the flue gas upstream of the CO₂ capture circuit. This generates an amine absorbent rich in sulfate. This high level of sulfate builds up more quickly than conventional degradation products, and requires removal for the process to be continuous. Possible methods for removing this sulfate from solution have been reviewed as part of an ongoing research project,^{1,2} with identified technologies evaluated in the laboratory. One such technology of interest is reactive crystallization. Ongoing work has focused on simulating the CS-Cap process to allow an economic comparison to be made between the CS-Cap process and a standard liquid-absorbent based CO₂ capture process incorporating FGD. This paper covers the results of that simulation work. The results of the economic analysis will be provided in a future paper.

Figure 1. Flow diagram of the CS-Cap process²

2.0 Methods

The ProTreat® simulation software has been shown to effectively replicate amine-based CO₂ capture processes evaluated at pilot scale.^{3,4} The ProTreat® simulation software was used here to simulate both a standard CO₂ capture plant in addition to the combined capture concept. These simulations were completed for both a brown and black coal-fired power stations. The flue gas

compositions evaluated are provided in Table 1. ProTreat uses full mass and heat transfer rate models in its simulations. The Deshmukh–Mather thermodynamic model was used for acid gas equilibrium calculations in amine solutions.¹¹

Table 1. Flue gas compositions used in the simulations (USPC – ultra supercritical pulverised coal)

For the base case simulation, the flue gas was sourced downstream of the FGD unit. This simulation matched that used in a recently completed IEAGHG study (900 MW gross USPC).⁵ For the cases with SO₂, the flue gas was sourced upstream of the FGD unit used in the IEAGHG study. This resulted in a flue gas SO₂ concentration of 700 ppm entering the CS-Cap plant. For the brown coal cases, the flue gas composition is a rough average of that measured at the CO₂ capture pilot plant located at the AGL Loy Yang Power Station.⁶ In addition, a flue gas concentration of 200 ppm SO₂ was also evaluated. This represents the typical flue gas SO₂ concentration observed in power station flue gas streams in Australia.³

The CO₂ capture facility was simulated with 30 wt% monoethanolamine (MEA), including 2 wt% formate as indicative of a typical heat stable salt (HSS). The production of formate was taken into account, with the thermal reclamation vessel sized to remove the HSS at the rate at which it forms, thus achieving a constant steady state level of 2 wt% formate in the absorbent. In a previous IEAGHG study⁷ the average degradation rate of MEA was determined to be 0.46 kg/tCO₂. This rate was used here as the formation rate of formate in the absorbent, assuming 1 mol MEA degrades to form 1 mol formate. For all simulations, 90% capture of the inlet CO₂ was achieved.

2.1 Standard CO₂ capture process

The standard CO₂ capture process consists of a pre-treatment cooler, CO₂ absorber, and CO₂ stripping column. To simplify the simulation, a wash stage was not simulated downstream of the CO₂ capture stage. In practice, this stage is used to capture water and amine carried over in the exiting flue gas, maintaining the water balance of the process and limiting the amount of absorbent make-up required. Here, the simulation control-block was used to add water and amine back into the simulation lost in the gas stream exiting the absorber, maintaining steady state conditions.

A slip stream of lean absorbent is removed from the CO₂ capture section and sent to a thermal reclaimer. Thermal reclamation is often a batch process. To simulate this step here as a continuous process in the steady state model, an equilibrium flash vessel is used, **operating at 149 °C and 185 kPa-a**. Na⁺ (to replicate NaOH) is added to the absorbent stream as a 10 wt% solution. Steam (150 °C, 450 kPa-a) is then added until the MEA remaining in the waste stream is 5% of that in the inlet. The hot vapor stream exiting the reclaimer is returned to the stripping column.

A line diagram of the standard CO₂ capture process, including thermal reclamation, is provided in Figure 2

Figure 2. Flow diagram of standard liquid-absorbent based CO₂ capture process simulated. Flue gas sourced downstream of the FGD unit

2.2 The CS-Cap process

The first step of the process incorporates a pre-treatment cooler to reduce the flue gas temperature, and condense excess water present in the flue gas stream. The cooled flue gas then enters the SO₂ capture step where the CO₂ rich absorbent from the CO₂ capture stage is used to remove the SO₂ from the flue gas. In the simulation, the SO₂ and CO₂ capture steps have been simulated as separate columns. These may be combined into a single column in the real process. Pilot-scale evaluation has shown that the majority of absorbed SO₂ is quickly converted to sulfate.^{2,8} To replicate this in the simulation, a component splitter (1 in Figure 3) is used to remove 95% of the SO₂ from the recirculating absorbent. An inlet stream with a flow multiplier is then used to add an equimolar amount of sulfate back into the absorbent solution. SO₂ lean flue gas then enters the CO₂ capture section, where sulfur free amine absorbent is used to capture CO₂ from the flue gas. A stripping column is used to remove the CO₂ from the solution and regenerate the absorbent.

A portion of the recirculating sulfate-rich absorbent is separated from the SO₂ capture loop and enters the crystallization step. Here, K⁺ (to replicate KOH) is added in 10% excess of that required to react with the sulfate present in solution. A ‘black box’ approach was then used to simulate the reactive crystallization step. Experimental results achieved in the laboratory were used to determine the level of sulfate removal that could be achieved in this stage.¹⁰ A component splitter (2 in Figure 3) was then used to remove K₂SO₄ in the required quantity. The absorbent remaining after the crystallization step is then split, with a portion of the sulfate-reduced stream being recirculated to the SO₂ capture loop. The remainder of the sulfate-reduced absorbent is then sent to a thermal reclamation step. The thermal reclamation step is the same as that described above for the standard process. However, experimental work completed in the laboratory evaluating thermal reclamation has shown that pH above 10 was required for recovery

of MEA from sulfate rich solutions.⁹ For simulations of the CS-Cap process, which result in high sulfate loaded absorbents, sufficient Na^+ was added to increase the pH of the stream entering the reclaimer to 11.

A line diagram of the CS-Cap process is provided in Figure 3.

Figure 3. Flow diagram of the simulated CS-Cap process

Initial simulations of the CS-Cap process have already been completed incorporating the SO_2 capture loop of the process only. This provided insight into the operating conditions of this novel section of the process.¹⁰ These simulations were recently upgraded to include the full process, as outlined in Figure 3. The results of these full simulations are provided in this paper.

3.0 Results

Proof-of-concept of the CS-Cap process was completed at the Loy Yang CO_2 capture pilot plant, using a CO_2 rich MEA absorbent in the pre-treatment column for SO_2 removal. This was completed as a batch process. Initially all SO_2 was removed from the incoming flue gas. The pH of the recirculating absorbent decreased to a pH ~ 7 to 8, where it entered a buffering region. Once the absorbent was saturated with sulfur, the pH of the solution was noted to drop rapidly, and break-through of SO_2 into the flue gas leaving the column was observed.² The pilot-scale operation used ~ 2.5 mol/L MEA in the pre-treatment column to capture SO_2 from the flue gas. The batch operation resulted in saturation of the MEA solution with absorbed sulfur. This was determined by ion chromatography to be predominately sulfate,⁹ with a concentration of approximately 10 wt% sulfate (or loading of 0.4 mol sulfate/mol MEA). Assuming the same

loading is possible for higher concentrations of amine, suggests that for 30 wt% MEA (5 mol/L) a sulfate concentration up to 20 wt% should be possible.

Increasing the proportion of recycling absorbent up to 98% was possible for the simulations completed here. This led to an increase in sulfate concentration up to 15 wt% in the recirculating absorbent stream, for the simulation cases with high SO₂ concentration flue gas streams. This sulfate concentration was below the upper limit predicted from the pilot-scale operation. The pH of the recirculating absorbent was in the range 7.7 – 8. It is anticipated that increasing the recirculating absorbent flow rate (i.e. minimizing the slip stream taken off for crystallization) increases the concentration of sulfate in the solution, increasing the efficiency of the downstream separation step. Before the process is operated in reality, consideration will also have to be given towards the allowable corrosion rates of the process. Increasing sulfate loading lowers the solution pH, increasing corrosion rates.

Laboratory scale experiments of reactive crystallization with sulfate loaded 30 wt% MEA solutions has shown the removal of sulfate, as K₂SO₄, to increase with increasing amount of precipitant addition (in this case KOH). Where KOH is added 10% in excess of that stoichiometrically required to react with the sulfate present in solution, between 70-75% of the sulfate is removed from solution at 20-40 °C.⁹ These laboratory experiments were completed in a structured environment as a batch process. Thus the removal of sulfate from solution might not be as efficient when the process is run continuously in a real plant. To be conservative, a lower fractional removal of 40% of the sulfate as K₂SO₄ has been assumed for all cases simulated here.

To observe the impact of increasing the recycle rate of absorbent in the SO₂ capture loop (increasing sulfate concentration), simulations were completed for two different recycle rates for

the brown coal case with 700 ppm SO₂ in the inlet flue gas (90 and 98% of absorbent recycled). The results of these two simulations are provided in Table 2. The properties of the **pre-treatment cooler**, CO₂ absorber and stripper were found to be unaffected by the change in operating conditions of the SO₂ loop. Increasing the recirculating absorbent flow rate increased the concentration of sulfate in the absorbent solution, and also requires a larger SO₂ absorption column. Hence the improved efficiency of the crystallization step comes at a slightly increased capital cost.

Further simulations were completed with 98% of the absorbent recycled in the SO₂ capture loop. These results, for both black and brown flue gases, are provided in Table 3. **90% CO₂ capture was achieved for all cases. This resulted in CO₂ concentrations of 1.1 (brown) or 1.3 mol% (black) in the purified flue gas exiting the absorber column. In all cases, no gaseous SO₂ was observed in this stream.**

Table 2. Effect of absorbent recirculation rate in SO₂ absorption section

Table 3. Properties of standard CO₂ capture plant and plant employing CS-Cap process for both brown and black coal flue gases

The higher moisture content of the brown coal flue gas is the main difference between the brown and black coal cases. This moisture was removed in the **pre-treatment cooler** to avoid dilution of the downstream SO₂ absorption loop. This resulted in a larger **pre-treatment cooler** requirement for the brown coal case, and a smaller downstream flue gas flow rate then entering the SO₂ and CO₂ absorption columns. As the flue gas SO₂ concentration increased, the concentration of sulfate in the absorbent in the SO₂ capture loop also increased. It is likely that

this will affect the efficiency of the crystallization step, but that was not investigated as part of the simulations completed here.

The higher absorbent recirculation rates has a small effect on the SO₂ absorber column size. Despite the increase in sulfate concentration, the pH in the recirculating absorbent remained within the buffering region observed during the pilot plant operation. At the pilot plant, breakthrough of SO₂ into the flue gas exiting the SO₂ absorption section was not observed until the absorbent was fully saturated with sulfate, and a sharp drop in pH was observed. Whilst in the buffering region, all SO₂ was removed from the flue gas. This is observed in the simulations here with SO₂ in the flue gas exiting the SO₂ absorption section being below 0.1 ppm in all cases.

The flow rate entering the thermal reclaimer was sized such that it removed the formate anticipated to be formed during the CO₂ capture process. As the concentration of sulfate in the solution increased, a higher mass flow of absorbent was sent to the reclaimer for both the brown and black coal cases. This then required a higher energy input to the reclaimer to meet the criteria of only 5% of the inlet MEA in the waste stream. In the simulations completed here, this higher energy input is recovered by recycling the reclaimer vapor stream to the stripping column, resulting in the overall thermal energy requirement being roughly similar for the 200 and 700ppm flue gas cases.

As the concentration of sulfate in the absorbent increased, the K⁺ addition necessarily also increased. Some of the excess, unreacted K⁺ then passed to the thermal reclamation circuit, resulting in a lower amount of Na addition to achieve pH 11 in the absorbent sent for reclamation. However, as KOH is typically a higher cost commodity than NaOH, the reduced NaOH usage resulting from KOH addition may not be favorable. An economic assessment is

required to determine if the increased operating costs of the CS-Cap process outweigh the reduced capital costs achieved by the process. This economic comparison is to be the focus of a future paper.

Finally, it should be noted that as only 95% conversion of SO₂ to sulfate was assumed, this left some residual unreacted SO₂ entering the thermal reclaimer in the simulation. This SO₂ was released with the vapor stream, entering the CO₂ stripping column. Thus, despite the flue gas being SO₂ free, SO₂ can still enter the CO₂ capture loop if any unreacted SO₂ remains in the absorbent sent to the reclaimer. If unreacted SO₂ is able to enter the CO₂ capture section, it will react with the amine in this loop, again forming a sulfate salt, and build up in the absorbent in this section. This sulfate will similarly require removal to ensure the effectiveness of the CO₂ capture section of the process. Pilot-scale evaluation of the full CS-Cap process is recommended to verify whether any unreacted SO₂ will be released downstream of the thermal reclamation step.

4.0 Conclusions

Combined capture of CO₂ and SO₂ from flue gases is being explored as a potentially lower cost method for reducing gaseous emissions from coal-fired power plant. This could be advantageous in countries such as Australia, that don't currently employ flue gas desulfurization (FGD) at coal-fired power stations. Removing the requirement for FGD could impart significant capital savings for the overall capture process.

The CS-Cap process combines the removal of CO₂ and SO₂ from flue gas streams. This process will result in the production of an amine absorbent rich in sulfate that requires regeneration to allow for a continuously operating process. A possible method for recovering the

sulfate from amine absorbents is to use reactive crystallization. Simulation of the CS-Cap process has been explored in this paper to provide information that will allow an economic comparison to be made to a standard CO₂ capture process combined with flue gas desulfurization in the future. Results show that increasing the recirculation rate of absorbent in the SO₂ capture loop will increase the concentration of sulfate in the absorbent, anticipated to improve the efficiency of any downstream removal step. The overall energy requirement of the CO₂ capture process does not appear to be significantly affected by the addition of the SO₂ capture stage. There will however be an increase in the operating costs as KOH addition is required to remove the sulfate from the aqueous amine absorbents.

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Table 1. Flue gas compositions used in the simulations (USPC – ultra supercritical pulverised coal)

Unit		Case				
		Base Case USPC Black coal with FGD	USPC black coal with CS-Cap, 200ppm SO ₂ in flue gas	USPC black coal with CS-Cap, 700ppm SO ₂ in flue gas	Brown coal flue gas with CS-Cap, 200ppm SO ₂ in flue gas	Brown coal flue gas with CS-Cap, 700ppm SO ₂ in flue gas
Temperature	°C	56.9	90.83	90.83	170	170
Pressure	kPa-a	108	110	110	110	110
Flow rate	kg/s	275.6	271.23	271.23	271.23	271.23
Concentration						
H ₂ O	mol%	12.1	9.49	9.49	20	20
CO ₂	mol%	13.4	13.75	13.75	10	10
SO ₂	mol%	0	0.02	0.07	0.02	0.07
N ₂	mol%	70.4	72.48	72.43	63.96	63.91
NO, Ar	mol%	0.8	0.87	0.87	0.02	0.02
O ₂	mol%	3.3	3.39	3.39	6	6

Table 2. Effect of absorbent recirculation rate in SO₂ absorption section

	Unit	Case	
		Brown coal flue gas with CS-Cap, 700ppm SO ₂ in flue gas Low sorbent recirculation rate	Brown coal flue gas with CS-Cap, 700ppm SO ₂ in flue gas High sorbent recirculation rate
Fraction sorbent recirculated		90%	98%
SO ₂ column L/G ratio	kg/kg	0.7	1.7
pH of recirculating absorbent		7.9	7.7
SO ₂ column diameter (80% flooding capacity)		7.9	8.4
Sulfate concentration in recirculating absorbent	wt%	8.8	15.4
Waste stream from thermal reclaimer	kg/s	0.27	0.34
Sulfate in reclaimer waste	kg/s	0.06	0.12
NaOH addition required to thermal reclaimer	kg/s	0.3	0.1

Table 3. Properties of standard CO₂ capture plant and plant employing CS-Cap process for both brown and black coal flue gases

Unit		Case				
		Base Case USPC Black coal with FGD	USPC black coal with CS-Cap, 200ppm SO ₂ in flue gas	USPC black coal with CS-Cap, 700ppm SO ₂ in flue gas	Brown coal flue gas with CS-Cap, 200ppm SO ₂ in flue gas	Brown coal flue gas with CS-Cap, 700ppm SO ₂ in flue gas
Pre-treatment cooler packed height	m	5	5	5	5	5
Pre-treatment cooler diameter	m	11.0	9.9	9.8	10.3	10.3
Pre-treatment cooler L/G	kg/kg	2.7	2.2	2.2	2.6	2.6
SO ₂ ABS packed height	m	N/A	4	4	4	4
SO ₂ ABS diameter	m	N/A	8.6	8.7	8.1	8.4
SO ₂ ABS L/G	kg/kg	N/A	1.0	1.5	0.8	1.7
CO ₂ ABS packed height	m	20	20	20	20	20
CO ₂ ABS diameter	m	11.0	11.0	11.0	10.6	10.6
CO ₂ ABS L/G	kg/kg	2.5	2.5	2.5	2.7	2.7
STR packed height	m	12	12	12	12	12
STR diameter	m	6.3	6.3	6.3	5.7	5.7
Reboiler duty	kW	168	145.5	138	120.7	111.8
Total thermal energy	kW	171.4	168.2	167	136.9	136.1
Total thermal energy	MJ/kgCO ₂	3.4	3.3	3.3	3.5	3.5
Absorbent flow to crystallisation	kg/s	N/A	5.2	8.2	4.1	8.5
Sulfate concentration	wt%	N/A	6.4	14.6	8.9	15.4
Absorbent flow to thermal reclamation	kg/s	0.81	1.1	1.5	0.7	1.3
pH absorbent SO ₂ loop		N/A	7.9	7.7	7.9	7.7
pH into thermal reclamation			11	11	11	11
Waste stream	g/s	49.3	256.1	402.3	192.8	340.5
Waste stream H ₂ O	g/s	10.8	69.7	84.6	49.6	69.1
Waste stream CO ₂	g/s	0.0	33.8	12.4	20.9	7.0
Waste stream MEA	g/s	12.8	12.7	13.2	9.9	9.9
Waste stream SO ₄ ²⁻	g/s	0.0	40.6	132.4	36.7	118.7
Waste stream formate	g/s	17.0	17.1	17.2	13.1	13.3
Waste stream Na ⁺	g/s	8.7	43.0	18.0	27.3	9.5
Waste stream K ⁺	g/s	0.0	39.2	124.4	35.4	113.0
NaOH addition	kg/s	0.09	0.43	0.18	0.27	0.09
KOH addition	kg/s	N/A	0.3	1.0	0.3	1.1
K ₂ SO ₄ produced	kg/s	N/A	5.1	18.0	5.5	19.7