

Research Article

Emissions of SO₃ from a Coal-Fired Fluidized Bed under Normal and Staged Combustion

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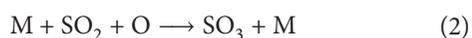
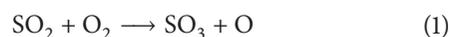
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This paper reports the measurements of SO₃ emissions with and without limestone under unstaged and staged fluidized-bed combustion, carried out on a 0.3 × 0.3 m² and 2 m high stainless-steel combustor at atmospheric pressure. The secondary air was injected 100 cm above the distributor. SO₃ emissions were monitored for staging levels of 85 : 15, 70 : 30, and 60 : 40, equivalent to a primary air/coal ratio (PACR) of ~0.86, 0.75, and 0.67. Experiments were carried out at 0%–60% excess air level, 1–2 m/s fluidizing velocity, 800–850°C bed temperature, and 20–30 cm bed height. During unstaged combustion runs, SO₃ emissions were monitored for a wide range of Ca/S ratios from 0.5 to 13. However, for the staged combustion runs, the Ca/S ratio was fixed at 3. SO₃ was retained to a lesser extent than SO₂, suggesting that SO₂ reacts preferentially with CaO and that SO₃ is involved in the sulphation process to a lesser degree. The SO₃ emissions were found to be affected by excess air, whereas the fluidizing velocity and bed temperature had little effect. SO₃ was depressed on the addition of limestone during both the staged and unstaged operations, and the extent of the reduction was higher under staged combustion.

1. Introduction

The presence of SO₃ in flue gas corrodes the equipment and ducts of combustion system and therefore needs to be removed [1]. In order to control emissions of SO₃, more studies on its formation and dissociation are required under air-fired and oxy-fired combustion conditions. The simulation study of Zheng and Furimsky [2] shows that SO₃ emissions would be unaffected during oxy-fuel combustion, being governed only by oxygen concentration. The kinetics of reactions occurring in the combustor were studied by Burdett et al. [3] using a TGA microbalance. They proposed the following mechanisms for the formation of SO₃.

1.1. SO₂/SO₃ Homogeneous Gas Phase Reaction. SO₂ may be oxidized to SO₃ by two reactions:



where M is a chaperon third body molecule. The large temperature dependence of reactions (1) and (2) ensures that the rate of production falls rapidly with decreasing gas temperature, and, in fact, 90%–95% of SO₃ is formed in the bed and freeboard and the remaining 5%–10% in the region between the freeboard and sampling point. SO₃ increases sharply with temperature, but the homogeneous reaction cannot account for all the SO₃ produced.

1.2. Heterogeneous Catalysis of SO₂ on Bed Particles and Heat Transfer Surfaces. In a coal burning combustor, a more effective catalytic material, iron oxide, is present in fly ash. While the SO₃ formation in this process is important, the experimental data is insufficient to quantify the SO₃ formation.

Dennis and Hayhurst [4] used an 80 mm diameter fluidized-bed combustor and mass spectrometer for measuring the concentration of SO₃. They confirmed the amount of SO₃ formed at atmospheric pressure to be very low and much less than the equilibrium concentration. The rate measured

was 100 times faster than expected for oxidation in the gas phase.

William and Gibbs [5] measured SO_2 and SO_3 emissions from a 0.3 m^2 fluidized-bed combustor. They reported that the higher the excess air level, the lower the SO_2 emissions (on removal of the dilution effect, SO_2 increased with an increase in excess air, reaching a limiting value of 1300 ppm above 30% excess air), and that SO_3 emissions increased slightly with the increase in excess air. Barnes [6] also observed the similar effect of excess air on SO_2 and SO_3 emissions. William and Gibbs [5] and Barnes [6] found that the higher the sulphur content of fuel, the higher the SO_2 emissions, while the SO_3 was unaffected by the fuel sulphur content [5]. Coal fed to the bed caused SO_2 and SO_3 emissions to increase than when fed to the surface [5]; SO_3 shows a weak dependence on bed temperature [5, 6].

Barnes [6] studied the effect of sand particle size, fluidizing velocity, and bed depth on SO_2 and SO_3 emissions. Barnes' findings indicate that fine sand (0.300 mm) produces high SO_2 and SO_3 emissions. Increasing fluidizing velocity from 1 to 2 m/s caused reduced formation of SO_2 in the bed and freeboard. An increase in bed depth increased SO_3 emission; a deep bed (30 cm) and fine sand resulted in a slight decrease in SO_2 emission.

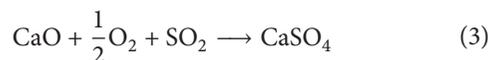
Oxygen availability and fluidizing characteristics within the bed also affect SO_3 formation. Ahn et al. [7] found that for pulverized coal, concentrations of SO_3 and SO_2 were significantly higher for oxy-fired conditions as compared to air-fired conditions. In circulating fluidized bed, SO_3 concentrations were notably higher for oxy-fired conditions too. For higher sulfur coal, SO_3 concentrations were 4–6 times greater on average. Their findings contradict the finding of Barnes [6].

Hindiyarti et al. [8] investigated the reaction of SO_3 with H, O, and OH radical. The revised rate constant calculated by them suggests that SO_3 and O reaction is found to be insignificant during most conditions. According to them, $\text{SO}_3 + \text{H}$ is the major consumption reaction for SO_3 .

Stanger and Wall [9] reviewed published work on SO_3 concentrations and emissions under oxy-fuel firing. Their conclusion is that the conversion of SO_2 to SO_3 is considerably variable.

William and Gibbs [5] found that coal char had a very significant removal effect on SO_3 emissions because SO_3 above the bed was 50% greater than in the exit. Barnes [6] said that unburnt char does not have a major effect on SO_3 as carbon carryover increases under the given operating conditions. Her results showed that the quantity of inert particles (30 cm deep bed) resulted in an increase in heterogeneous catalytic reaction of SO_2 to form SO_3 .

Burdett et al. [3] carried out experiments in a microbalance to study the effect of limestone on SO_3 emissions. According to Burdett et al. [3], the reaction of CaO, O_2 , and SO_2 , in order to yield CaSO_4 (reaction (3)), must occur in two separate steps. Two possibilities exist, either SO_2 reacts with CaO and CaSO_3 is formed, which is then oxidized, or else the formation of SO_3 in gas phase or on a stone surface is followed by an attack on the CaO. Consider the following:



Route 1



Route (2)



It is not possible, due to the lack of available experimental data, to say which of these mechanisms is operative under given conditions, although both may be important. A most interesting comparison between the SO_x level detected with and without limestone (proposed by Burdett et al. [10]) is the SO_2/SO_3 ratio. Without limestone, the ratio they found was 2200/33 (or 67:1), and with limestone it increased to 350/1.5 (or 230:1). It is clear that SO_3 is depressed to a greater extent than SO_2 on the addition of limestone, and they attribute this to the higher reactivity of SO_3 compared with that of SO_2 . Burdett [11] and Burdett et al. [10] have assumed that SO_2 oxidizes to SO_3 in the particles at a rate dependent on local SO_2 and O_2 concentrations, with SO_3 diffusing through the CaSO_4 shell and reacting with CaO. The rate of formation of CaSO_4 may be linked to different rates of production of SO_3 at different locations within the stone. At a high oxygen level, oxidation increases preferentially at the edge of a particle. High utilization is achieved when SO_2 diffusion in the interior of the stone is maximized, and this, in turn, implies a low SO_3 formation rate. Barnes [6] reported a decrease in SO_2 conversion to SO_3 with oxygen concentration and an increase with SO_2 concentration.

Fieldes et al. [12] reported the achievement of a high fractional sulphation (0.36) when coal is burnt in the bed. It appears that the fraction of the sulphur gas phase, which is SO_3 , has a substantial effect on the fractional sulphation of the limestone. Ash also appears to remove SO_3 selectively. The mechanism of this hypothesis supposes that the direct reaction of CaO with SO_3 is faster than a reaction via the CaSO_3 intermediate. This paper examines the factors responsible for formation and reduction of SO_2 and SO_3 from a coal-fired fluidized bed under varying operating conditions.

2. Apparatus and Procedure

The main features of the fluidized-bed combustor and ancillaries are presented in Figure 1. The bed consisted of silica sand of mean size 0.700 mm. Fluidizing air was supplied by a fan and metered and introduced through a distributor plate. For staged combustion, the secondary air was introduced into the combustor through a stainless-steel pipe 100 cm above the bed surface. In staged combustion mode, the total combustion air is separated into a primary air stream supplied

TABLE 1: Typical analysis of Linby and Daw Mill coal.

	Weight (%)	
	Linby	Daw Mill
Proximate analysis (dry basis)		
Ash	9.3	4.0
Volatile matter	31.0	37.8
Fixed carbon	59.7	58.2
Ultimate analysis (dry basis)		
Carbon	72.3	77.3
Hydrogen	4.9	5.1
Oxygen	10.17	10.44
Nitrogen	1.5	1.3
Sulfur	1.53	1.66
Moisture	8.0	6.3
Gross calorific value (MJ/kg)	30.24	31.54

TABLE 2: Chemical composition of Ballidone and Penrith limestone.

	Ballidone	Penrith
CaCO ₃	97.8%	95.6%
CaO	55.5%	52.5%
CO ₂ (at 1000°C)	42.3%	43.1%
SiO ₂	1.9%	2.8%
Fe ₂ O ₃	0.09%	0.4%
TiO ₂ -Al ₂ O ₃	0.08%	0.8%
MnO	0.3%	0.2%

to fluidize the bed and a secondary air stream injected above the bed to complete the combustion. For example, in 70 : 30 staging, 30% of the total air is injected as secondary air.

The bed was preheated by a propane burner that was fixed above the bed, and the fluidizing airflow rate was adjusted to the lowest level to minimize heating time. Coal was fed in the combustor when the bed temperature reached 550°C. When the bed temperature reached 800°C, the desired coal feed rate was adjusted to a constant value, the propane burner was switched off, and the fluidizing air was adjusted to the required level. The bed temperature was maintained constant by using an adjustable cooling coil with circulating water. Concentrations of O₂, CO, CO₂, and SO₂ were recorded continuously by an ADC-RF infrared gas analyzer.

The experiments were carried out at bed temperatures of 800–850°C, fluidizing velocities of 1–2 m/s, and excess air levels of 0%–60%. Static bed height was 20–30 cm. Two types of coal bituminous Linby and Daw Mill of 3–16 mm (large) diameter in size and two types of limestone, Ballidone and Penrith of <3 mm mean diameter in size, were used. In both cases, the coal was premixed with the limestone and fed overbed at 42 cm above the distributor (see Table 1 for proximate and ultimate analyses of the coal and Table 2 for chemical composition of the limestone). The Ca/S ratio was 3 : 1 mole per mole or otherwise as indicated. Three levels of staging (15%–40% secondary air) were used to investigate the effect of fluidizing velocity, bed temperature, and excess air on SO₃ reduction during air staging.

3. Sampling of SO₃

An SSL/MEL SO₃ analyzer, developed by Severn Science Labs/Marchwood Eng. Labs, was used for continuous monitoring of the SO₃. A detailed account of the principles and operating procedures of an SSL/MEL analyzer can be found in Jackson et al. [13] and Hotchkiss et al. [14].

The representative sample of flue gas was extracted from the sample point located near the exit of flue gas to the cyclone (200 cm above the distributor), where the gas temperature was around 550°C. Under these conditions, the use of a quartz sampling probe was found to be adequate. This enabled the extraction of the acid-containing gas directly into the filter-contactor of the SO₃ analyzer. The temperature of the sampled gas was maintained (by keeping the length of the tube as short as possible) at a value in excess of the acid dewpoint and below the temperature at which significant dissociation to SO₃ occurred; accurate determination of the acid gas content in the gas could then be made precisely over a significant period of time. This procedure effectively eliminates any interaction between the SO₃ in the gas sample and other species within the sampling probe itself.

4. Results and Discussion

4.1. SO₃ Emissions without Limestone under Unstaged Combustion. The effect of some operating variables on the SO₃ emissions under unstaged combustion is presented in Figure 2. The results are corrected to 5% oxygen in the flue equivalent. The flue gas SO₃ emissions ranged from 5 to 10 for the Linby coal. SO₃ emissions decrease with excess air when corrected for dilution, increase slightly with excess air, reach limiting values, and then gradually decrease. The fluidizing velocity also affects emissions to some extent. The effect of bed height on SO₃ emissions was associated with the size of sand particles. As the fine sand produces more reducing environment, the oxygen stoichiometry influences the rates of oxidation of SO₂, and as a result, the generation of SO₃ is reduced. The SO₃ emissions were also less sensitive to change in bed temperature. The rise in emissions was typically 0.5/10°C.

In another set of experiments, the Daw Mill Coal was tested for SO₃ emissions. The flue gas SO₃ emission ranged from 4 to 19.5 ppm for Daw mill coal. Changing the operating parameters resulted in a maximum change in SO₃ emission of 13 ppm. Increasing the bed depth resulted in higher SO₃ emission, the fluidizing velocity also affected emission depending on the size of bed material used. The SO₃ emission increased as the bed size was varied from coarse to fine with the bed depth. SO₃ emissions were weakly dependent on temperature typically rising 0.7 ppm/10°C.

4.2. Comparison to Reported Work. Dennis and Hayhurst [4] have reported that the SO₃ formation under atmospheric pressure was very low (e.g., mole fraction of SO₃ in off gas = 7×10^{-5} for mole fraction of SO₂ gas entering a bed = 2.3×10^{-3} at 875°C). The oxidation rate in the gas phase was 100 times faster than expected. An Eley-Rideal mechanism was

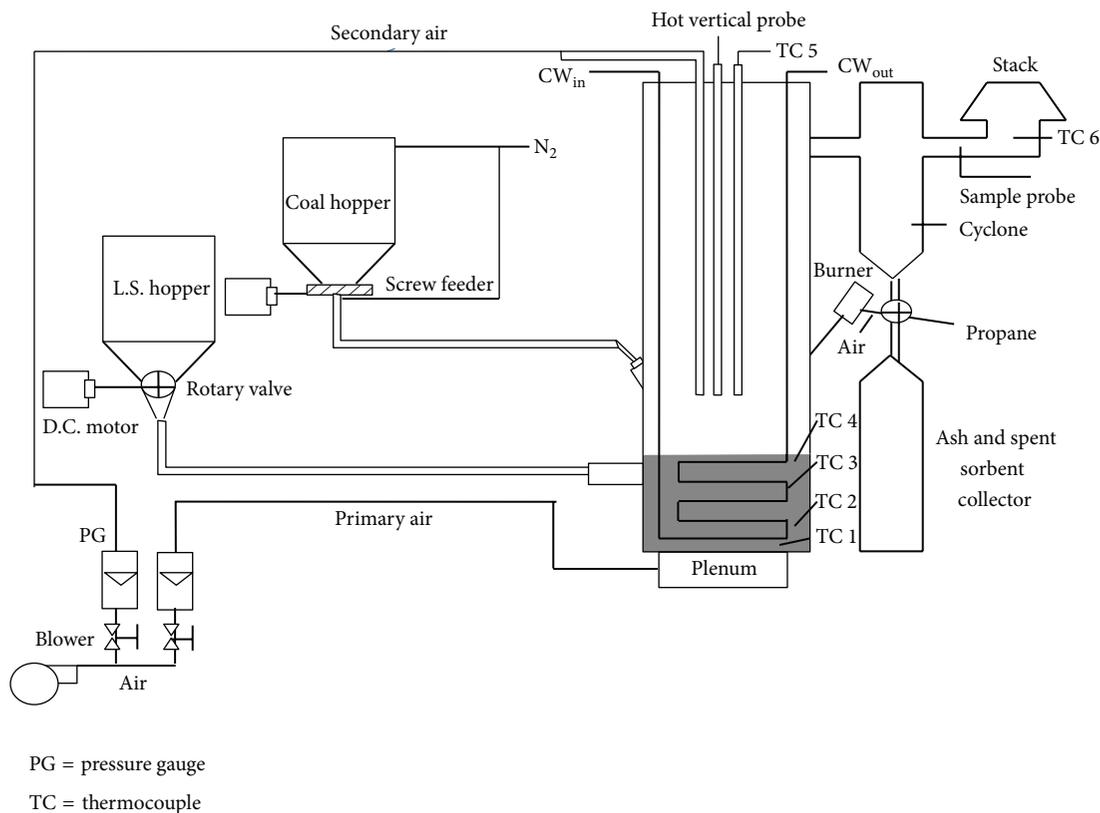


FIGURE 1: Main features of fluidized-bed combustor and ancillaries.

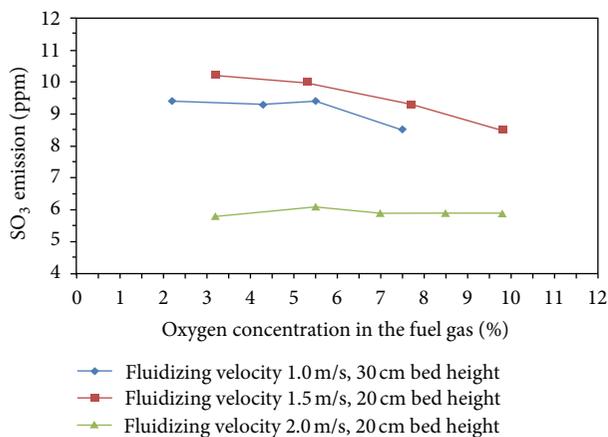


FIGURE 2: Influence of fluidizing velocity, excess air, and bed height on SO_3 emissions during unstaged combustion at 850°C bed temperature and coarse sand (corrected to 5% in flue equivalent).

proposed in which O_2 and SO_2 competitively chemisorb on the surface, and the rate of reaction is controlled by gas-phase molecule of SO_2 reacting with adsorbed O atom.

William and Gibbs [5] have tested many coal types for SO_3 concentration without limestone in $750\text{--}900^\circ\text{C}$ temperature range. Their findings suggest that ash (having traces of Ca, Mg, Na, K, etc.) is the principle removing species of SO_3 . In another experiment, when pure SO_2 was introduced,

the SO_3 reacted with added char at 850°C in the absence of oxygen to give SO_3 of 7 vpm in the outlet, which suggests that char is important in the removal of SO_3 . He also observed a 50% reduction in SO_3 in the freeboard. According to William, the reduction was due to the reaction of SO_3 with unburnt char.

SO_3 emissions are dependent on the oxygen and sulfur dioxide concentrations and were found to follow a similar trend. William and Gibbs [5] found that in contrast to the effect on SO_2 emissions, fine coal produced lower SO_3 emissions. In this study, SO_3 emissions were slightly higher when fine sand was used and tended to increase with bed height. This suggests that unburnt char does not have a significant effect on SO_3 emissions. The results of this study indicate that the amount of particles in the bed could have a significant effect on SO_3 emissions, resulting in an increase in the heterogeneous catalytic reaction of SO_2 to form SO_3 as the quantity of bed particles increases. Higher bed height, therefore, will also result in high SO_3 emissions. The oxygen concentration and fluidizing velocity will also affect SO_3 formation.

4.3. SO_3 Emissions with Limestone under Unstaged Combustion. SO_3 emissions decrease in the presence of limestone and the reduction is temperature sensitive. The SO_3 reductions were less sensitive than the reductions achieved for SO_2 at similar conditions [15, 16]. At a temperature around 850°C , the SO_3 reductions were only 28% of the SO_2 reductions, but

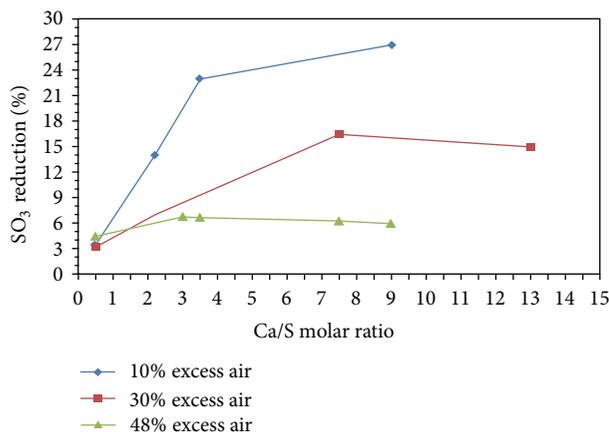


FIGURE 3: Influence of limestone addition on SO_3 reduction during unstaged combustion 25 cm height, 1.0 m/s fluidizing velocity, 850°C bed temperature, and 12 cm limestone addition height.

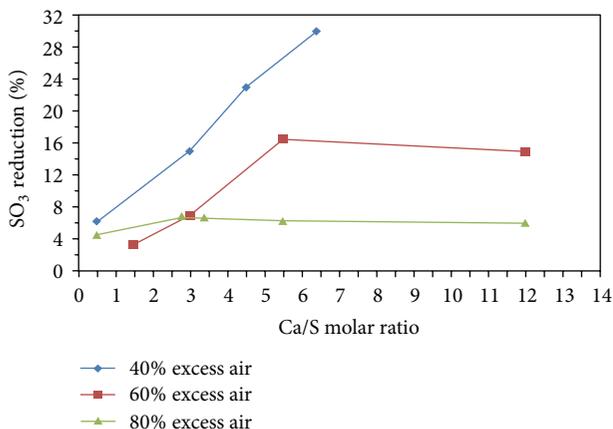


FIGURE 4: Influence of limestone addition on SO_3 reduction during unstaged combustion 25 cm height, 1.0 m/s fluidizing velocity, 850°C bed temperature, and 42 cm limestone addition height, as well as coarse sand.

at 800°C , the reductions reached 70% of the SO_2 reduction level. The results corresponding to the operating conditions are shown graphically in Figures 3 and 4. It should be noted that the SO_3 reduction shown in Figure 3 was obtained when limestone was injected 12 cm above the distributor, and Figure 4 represents the results when limestone was injected 42 cm above the distributor.

At a temperature of 850°C , some of the SO_2 will always be converted to SO_3 via reaction (6). The conversion decreases with oxygen concentration and increases with sulfur dioxide concentration. An increase in temperature enhances the rate of SO_3 formation. SO_3 can react with CaO to form CaSO_4 via reaction (7). The rate of this reaction is temperature dependent. Yilmaz et al. [17] studied the thermal dissociation of SO_3 in the range of 800 – 1200°C under atmospheric pressure. At the location in the flame where the net SO_3 formation rate is zero, he determined a rate constant of $6.9 \times 10^{10} \text{ cm}^3 \text{ mol}^{-1} \text{ s}^{-1}$

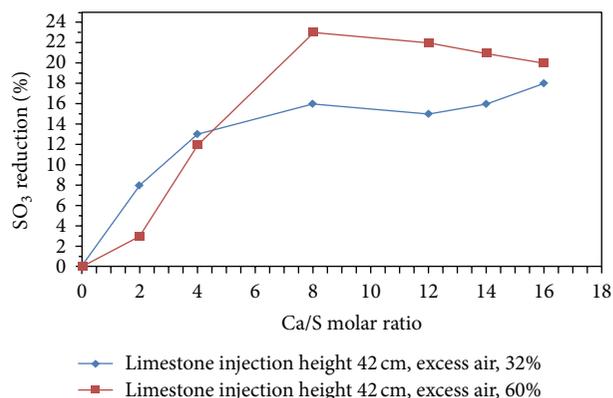


FIGURE 5: Influence of limestone addition on SO_3 level in the flue under staged combustion.

for $\text{SO}_3 + \text{N}_2 \rightarrow \text{SO}_3 + \text{O} + \text{N}_2$; that was consistent with other flame results. A high temperature lowers the reaction rate. Therefore, at a high temperature, more SO_3 is produced but less will be consumed in sulphation. As a result, a larger decrease in SO_3 emissions is observed at lower temperature.

The effect of excess air can also be seen in these graphs. SO_3 emissions have been found to increase with excess air, but upon removing the dilution effect, the increase is within a narrow range, indicating that there could be an optimum reduction at a particular excess air beyond which the SO_3 reduction decreases. An increase in the fluidizing velocity has little effect on the overall reduction of SO_3 emissions.

During another set of experiments, Penrith limestone was added to the Daw Mill coal. It was observed that SO_3 emissions were decreased in the presence of limestone, and the reduction was temperature dependent. At the higher temperature of 850°C , the SO_3 reductions were 18%–20% of the SO_2 reduction, but at 800°C the SO_3 reductions reached 55% of the SO_2 reduction level. Figure 5 shows the results of this set.

4.4. Comparison to Reported Work (Conducted on Microbalance or Small Bed of 36–78 mm ID). Burdett et al. [3] have reported that the reaction between limestone and sulfur oxides is highly sensitive to changes in O_2 , SO , and SO_3 concentrations. Absorption of SO_3 by the coal ash cannot be quantified on the microbalance, and the microbalance results are not applicable to fluidized combustor.

Fieldes et al. [12] have reported that extent of SO_2 oxidation to SO_3 varied with SO_2 and O_2 concentration. The coal combustion test showed that the lower SO_3 concentrations are due to its selective removal by ash. They had tested a variety of limestone, and in all the cases the mole fraction of CaO converted to CaSO_4 was affected by inlet oxygen in the same way as Penrith limestone.

Thibault et al. [18] have conducted experiments on a small (6 mm) fixed bed packed with CaO particle. They have tested two grain size of the sorbent and reported that for efficient capture of SO_3 a small grain size and open macropore structure are essential.

4.5. Comparison to Reported Work (Conducted on Pilot Scale). Burdett et al. [19] have reported fractional conversion of SO_2 to SO_3 decreased from about 1.5% in the limestone-free case to around 0.35% when the limestone and alkaline ash were present, which was due to the greater reaction of SO_3 with limestone compared with ash, the absorption occurring both in the bed itself and in the freeboard.

Burdett et al. [10] have reported that combustion of a 3% sulfur coal in a bed burning at 900°C generated 33 vpm of SO_3 and proposed that the effect of O_2 on sulphation capacity results from the formation of SO_3 within the pores of the stone.

4.6. SO_3 Emissions without Limestone under Staged Combustion. Merryman and Levy [20] have conducted staged experiments on a quartz tube methane burner producing stable methane- H_2S flame within desired fuel-air ratio without a sorbent presence under staged combustion conditions. They have reported that when the remaining excess air was injected into these gases, the maximum amount of SO_3 formed was greater than formed when this additional air was included with the initial combustion air, the overall excess of air being the same in both cases. The experimental conditions of Merryman and Levy do not match with our fluidized bed; therefore, their results are not comparable with this study.

During this study, the SO_3 emissions under staged combustion without limestone could not be monitored extensively due to malfunctioning of SO_3 analyzer.

4.7. SO_3 Emissions with Limestone under Staged Combustion. The concentration of SO_3 emissions at 1.5 m/s and 20% excess air was 17.0 ppm, which decreased to 5.5 ppm in the presence of limestone. The SO_3 emissions at 70/30 staged (1.5 m/s, 850°C) combustion (without limestone) were similar to those of unstaged combustion (without limestone). However, it was observed that in the presence of limestone, staged combustion results in a higher reduction of SO_3 than unstaged. Figure 6 gives the SO_3 emissions as a function of PACR. The emissions at 15% secondary air were 1.5 ppm and increased to 7.2 ppm at 45% secondary air. It is clear that SO_3 is depressed on the addition of limestone during both the unstaged and staged operations, and the extent of reduction was higher under staged combustion.

Figure 6 shows that the maximum removal of SO_3 occurs at a lower staging levels of 85/15, and, as the bed becomes more substoichiometric, the rate of SO_3 removal decreases. This trend indicates the formation of SO_3 in the freeboard which bypasses the limestone and appears in the flue. This increase in SO_3 reduction with the in-bed air ratio is in agreement with Barnes [6] findings.

The results of the staged combustion test with Daw Mill coal in the presence of Penrith limestone indicate that SO_3 emissions varied little with changes in excess air. However, if excess air is coupled with fluidizing velocity, then it had some effects on the emissions. At higher velocity of 2 m/s, the change was up to 4 ppm. The concentration of SO_3 emissions at 1.5 m/s and 30% excess air was 20 ppm which decreased to

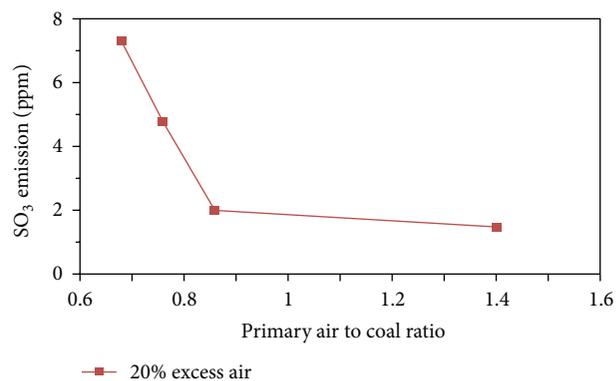


FIGURE 6: Influence of PACR on SO_3 emission at 1.5 m/s fluidizing velocity and coarse sand.

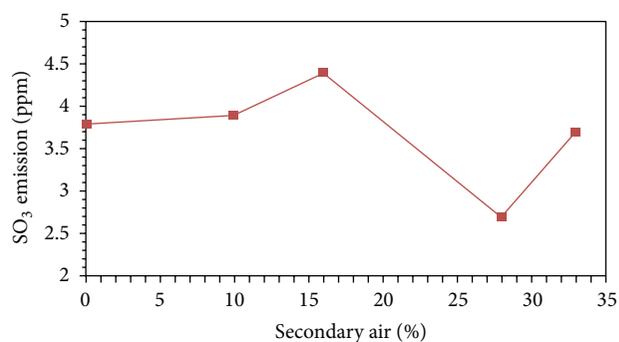


FIGURE 7: Influence of staging SO_3 emission in the flue secondary air injection height 100 cm above the distributor, excess air 30%.

8 ppm under staged combustion in the presence of limestone. The results of Daw Mill coal test are shown in Figure 7.

It should be noted that there is no published work on SO_3 emissions under staged combustion conditions with or without limestone on any scale. Therefore, the results of this study could not be compared.

5. Conclusion

The experimental data shows that during unstaged combustion without limestone, SO_3 emissions are dependent on oxygen and SO_2 concentration. SO_3 emissions increase slightly with excess air, reaching a limiting value, and then slowly decrease. SO_3 emissions are less sensitive to change in bed temperature. However, the fluidizing velocity and bed height affect the emissions.

In the presence of limestone, SO_3 emissions are reduced during both staged and unstaged operations, and the reduction is temperature sensitive. However, during staged combustion, the reduction is enhanced. As staged fluidized-bed combustion is a proven technique to reduce NO_x and SO_2 emissions, therefore, it should be possible to operate a fluidized-bed combustor under a staged mode with limestone to keep SO_2 , SO_3 , and NO_x emissions to a minimum.

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