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Influence of SO₂ on the NO/N₂O chemistry in fluidized bed combustion

2. Interpretation of full-scale observations based on laboratory experiments

Kim Dam-Johansen, Lars-Erik Åmand* and Bo Leckner*

Department of Chemical Engineering, Technical University of Denmark, Denmark

*Department of Energy Conversion, Chalmers University of Technology,

S-412 96 Göteborg, Sweden

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In circulating fluidized bed combustion of coal, significant interactions between desulfurization by limestone and emissions of nitrogen oxides (NO, N₂O) are generally observed. In order to facilitate interpretation of experimental results obtained in a 12 MW circulating fluidized bed boiler, a series of laboratory tests have been carried out. The tests include homogeneous CO and HCN oxidation in the presence of different concentrations of SO₂, CO oxidation catalysed by calcined and partly sulfated limestone particles, and formation and reduction of NO and N₂O over bed material containing char. The presence of SO₂ in the gas is shown to decrease the rate of homogeneous CO and HCN oxidation and thereby change the product distribution of the nitrogen-containing species. Unlike sulfated limestone, calcined limestone (CaO) is shown to be a good catalyst for oxidation of CO. A lower mean CO concentration is therefore expected in fluidized bed combustors during injection of limestone. This indirectly influences the nitrogen chemistry. Finally, the influence of NO and O₂ on the formation of N₂O from char was studied.

(Keywords: combustion; fluidized beds; sulfur)

Sulfur retention on lime under fluidized bed combustion (FBC) conditions has been the subject of intensive laboratory research¹⁻¹⁴, but the interaction between sulfur capture on lime and nitrogen oxides is less well known. This interaction is the subject of the present paper, which describes the results of a laboratory study performed to complement the observations made in a large-scale research boiler, presented in part 1 of this work¹⁵.

The interaction between sulfur capture and emission of NO observed in FBC has led to laboratory studies of the decomposition of ammonia catalysed by CaO or CaSO₄ surfaces¹⁶⁻²⁰. Other aspects of the interaction between desulfurization and nitrogen chemistry, such as NO reduction on CaO and CaS, have been studied by Furusawa *et al.*^{16,21}, and N₂O formation and reduction by de Soete²² and Hansen *et al.*²³.

In part 1¹⁵, it was suggested that a connection may exist between the SO₂-CO-NO and N₂O chemistry in FBC, both directly through homogeneous gas phase reactions and/or indirectly through oxidation of CO on lime. At present there is no knowledge of the influence of SO₂ on the oxidation of CO or whether CaO or CaSO₄ act as oxidizing catalysts for CO at temperatures of relevance for FBC.

Hulgaard *et al.*²⁴ showed that volatile nitrogen in the form of HCN, rather than NH₃, may be an important

precursor of homogeneous N₂O formation in the presence of CO at FBC temperatures. This is in agreement with calculations performed by Kilpinen and Hupa.²⁵ The N₂O is primarily formed by a reaction between NO and nitrogen-containing radicals produced from HCN. However, no information is found in the literature about the influence of SO₂ on this formation route of N₂O under FBC conditions.

EXPERIMENTAL AND RESULTS

Apparatus

Homogeneous as well as heterogeneous reactions were studied. For the homogeneous reactions a quartz plug-flow reactor was used (*Figure 1*). The reactor has been described previously^{24,26}. The reaction tube (6) was cylindrical, with a diameter of 0.51 cm and a length of 14 cm. Its lower end was connected to an outlet tube cooled by air. Inert gas, primarily N₂, was led through the main stream inlet (2) and preheated before entering the reaction tube, where it was mixed with the reactants injected separately through four injectors (1). The temperature in the reactor was measured by a thermocouple shielded by a quartz tube.

Another quartz flow reactor was used for the heterogeneous reactions (*Figure 2*). The reactor was similar to that used by Dam-Johansen and Østergaard¹.

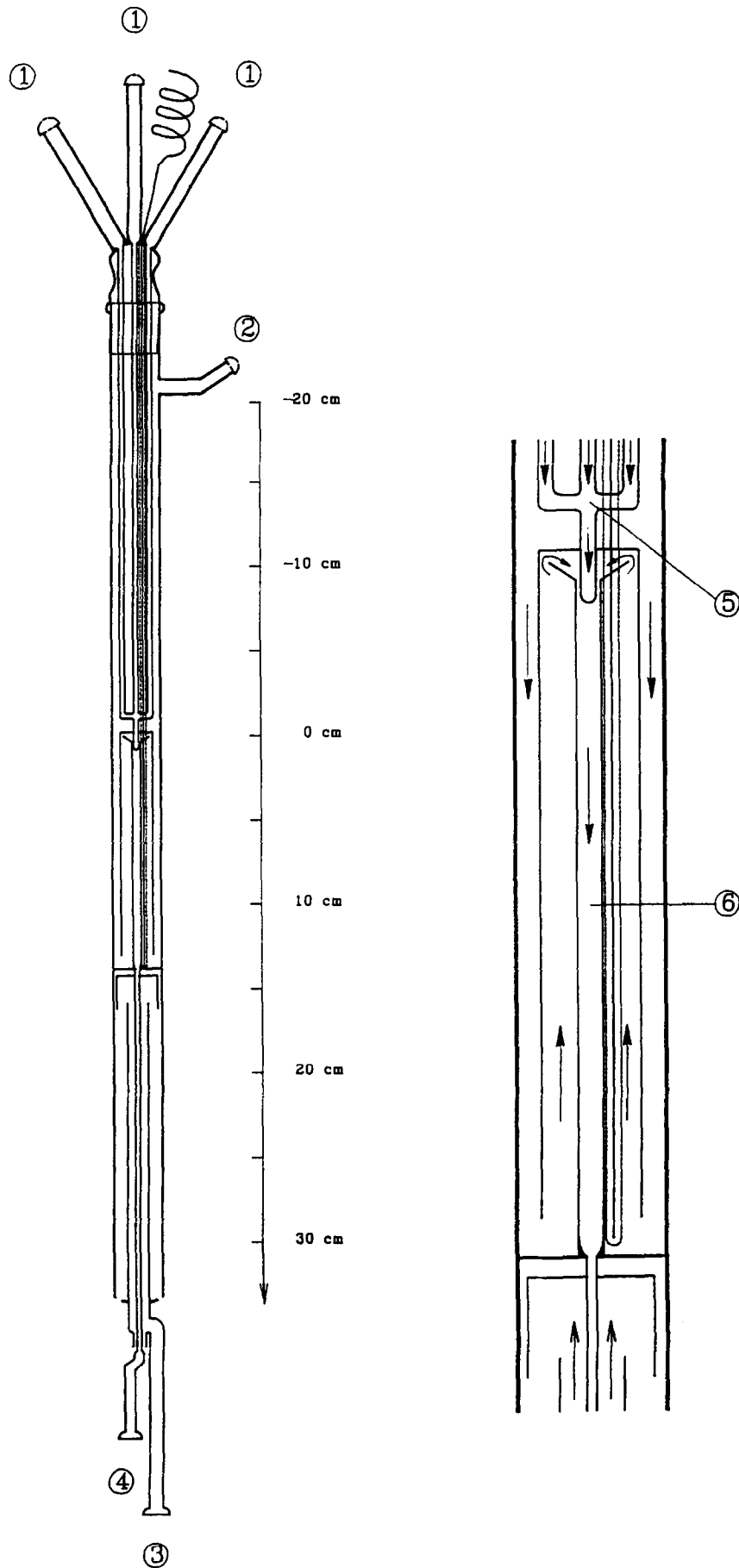


Figure 1 Quartz flow reactor for homogeneous reactions: (1) injectors for reactive gases; (2) main stream inlet for inert gases; (3) inlet for cooling air; (4) outlet; (5) mixing volume for reactants; (6) reactor tube

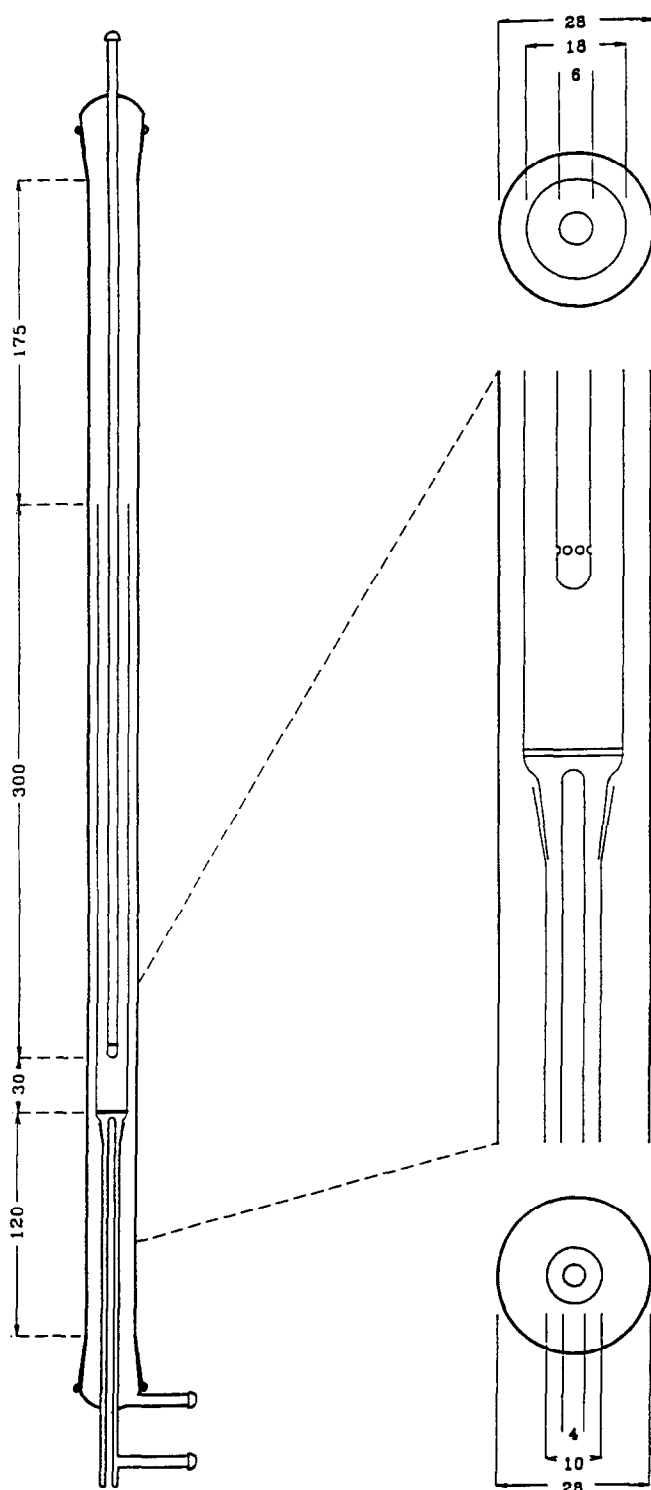


Figure 2 Quartz fixed bed reactor for heterogeneous reactions (measurements are in mm)

The bottom and the inner section of the reactor could be removed; the solid to be investigated was placed on a porous quartz plate, and the inner section was put back without changing the temperature. The temperature was measured just below the quartz plate by a thermocouple shielded by a quartz tube.

Both reactors were placed in a three-zone electrically heated oven which maintained the reaction zone at a constant temperature, within a few degrees.

A gas of well defined composition was mixed from pure gases or gas mixtures in a panel of precision flow

controllers. Continuous monitors were used to measure SO_2 , NO , N_2O , CO , CO_2 and O_2 concentrations in the outlet of the reactors. Prior to the experiments, the monitors were evaluated with respect to linearity and cross-sensitivities. The only monitor that was significantly influenced by the presence of the other components was the N_2O analyser (Perkin-Elmer, Spectran 647). To avoid cross-sensitivities, especially in the N_2O analysis, water was removed by condensation in dry ice and SO_2 was absorbed in a sodium carbonate solution²⁷.

Materials

The limestones studied were Ignaberga limestone, also used in the full-scale experiments in part 1, and, for comparison, Stevns chalk. Ignaberga limestone is a Swedish limestone of intermediate reactivity towards sulfur retention. Stevns chalk is a Danish, very porous chalk of high sulfur reactivity. The two types of limestone have previously been characterized as desulfurization agents under oxidizing conditions^{1,2} under alternating oxidizing and reducing conditions more closely representing the reaction conditions in FBC^{6,7}.

Bed material samples

The experiments performed are listed in Table 1 for both the homogeneous and the heterogeneous reactions.

Bed material was withdrawn from the centre of the dense bed of the full-scale plant, approximately 65 cm above the bottom plate, during a test with the reference conditions defined in part 1 (bed temperature 850°C , excess air ratio 1.2, primary air stoichiometry 0.75, no limestone addition, low-sulfur coal). The mean char content of this bed sample was determined to be about 4.2%. A small fraction of this sample together with selected char particles from the sample, 0.1–3 mm in size, were used in the tests.

Homogeneous oxidation of CO

The experimental conditions are summarized in the first column of Table 1. The reactor shown in Figure 1 was used for the experiment. Without H_2O present, the rate of CO oxidation was low and no influence of SO_2 could be detected because of the low overall CO conversion in the reactor. In the presence of H_2O and O_2 , CO was oxidized rapidly at 850°C and a high conversion of CO was observed even at residence times below 50 ms. The presence of SO_2 moderately inhibited the CO oxidation.

Homogeneous conversion of HCN

The experimental conditions are summarized in the second column of Table 1. The reactor shown in Figure 1 was used for the experiment. N_2 , O_2 and H_2O were introduced through the main stream inlet and were preheated before being mixed with HCN, CO, NO and SO_2 separately introduced through the four injectors.

Figure 3 shows the normalized CO, NO and N_2O concentrations in the reactor outlet versus the temperature in the presence of 50, 500 or 2000 ppm SO_2 . The N_2O concentration is normalized with the inlet HCN concentration.

As in the homogeneous CO oxidation experiments, the presence of SO_2 inhibits the conversion of HCN and CO and results in a temperature shift towards higher values. A significant amount of N_2O is formed from HCN at

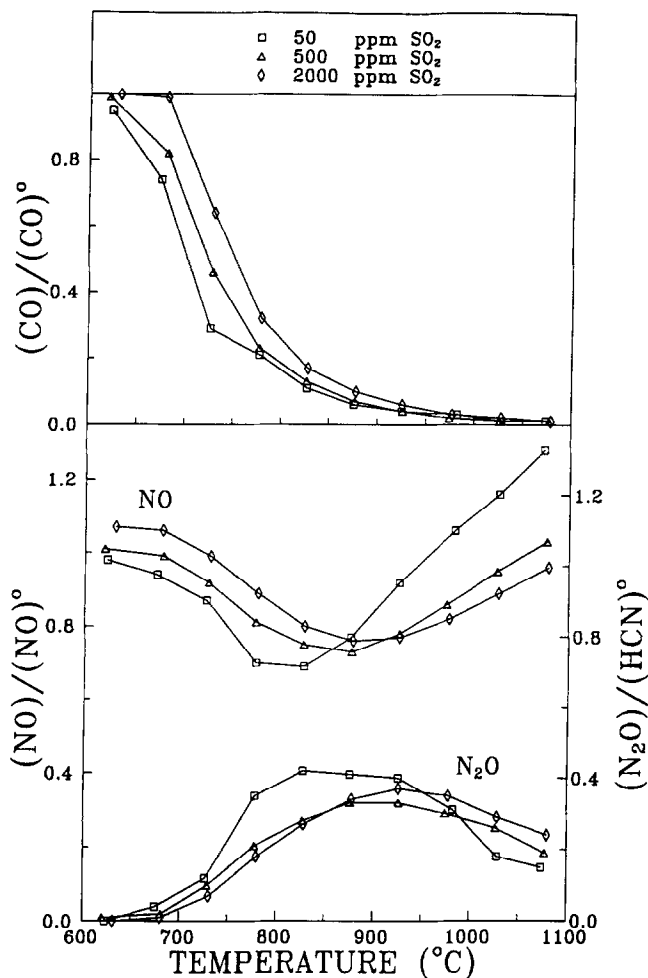
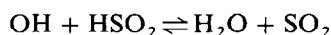
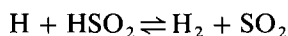
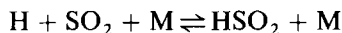


Figure 3 Normalized outlet concentrations of CO, NO and N₂O versus the reactor temperature for SO₂ contents of 50, 500 and 2000 ppm. Inlet concentrations of CO, NO and HCN were about 1780, 475 and 300 ppm respectively. (The case SO₂ = 0 is very close to SO₂ = 50 ppm and is not shown.) The experimental conditions are summarized in the second column of *Table 1*

FBC temperatures. HCN may either reduce NO or form NO, depending on the temperature and the gas composition. In the present experiments NO was reduced in the temperature range of 750–950°C, but without NO present initially Hulgaard²⁸ observed a net formation of NO. Furthermore, Hulgaard demonstrated how the curves are moved towards lower temperatures by an increase in the residence time and/or a high CO concentration. The reason for the shift of NO concentration towards higher temperatures in the presence of SO₂ may be the catalytic effect of SO₂ on the radical recombination reactions²⁹:



where M symbolizes a third body.

Heterogeneous oxidation of CO

The experimental conditions are summarized in the third column of *Table 1*. The reactor shown in *Figure 2* was used.

Three types of experiment were carried out in the presence of NO: (1) oxidation of CO catalysed by

calcined Ignaberga limestone and Stevns chalk; (2) oxidation of CO catalysed by partly sulfated Ignaberga limestone with and without SO₂ present; (3) oxidation of CO catalysed by Ignaberga limestone and Stevns chalk during sulfation.

NO was not reduced in any of the experiments in the presence of O₂.

The particles of Ignaberga limestone introduced to the reactor had a diameter of 1.0–1.4 mm. During the rapid heat-up period they fragmented, resulting in a much lower mean diameter. This may be one of the reasons for the relatively high catalytic activity towards CO oxidation observed for Ignaberga compared to Stevns chalk.

The results of CO oxidation during sulfation of Stevns chalk are shown in *Figure 4* in the form of CO, NO and SO₂ concentrations in the outlet of the reactor versus time. Initially, the chalk was introduced into the reactor where it reacted with SO₂ in the mainly oxidizing atmosphere, forming CaSO₄. CO was catalytically oxidized by the CaO surface and NO was hardly influenced. As the sulfation proceeded, the reactivity towards SO₂ removal and catalytic CO oxidation decreased, indicating a very small or no catalytic activity of the solid product CaSO₄.

The data from *Figure 4* and three similar experiments

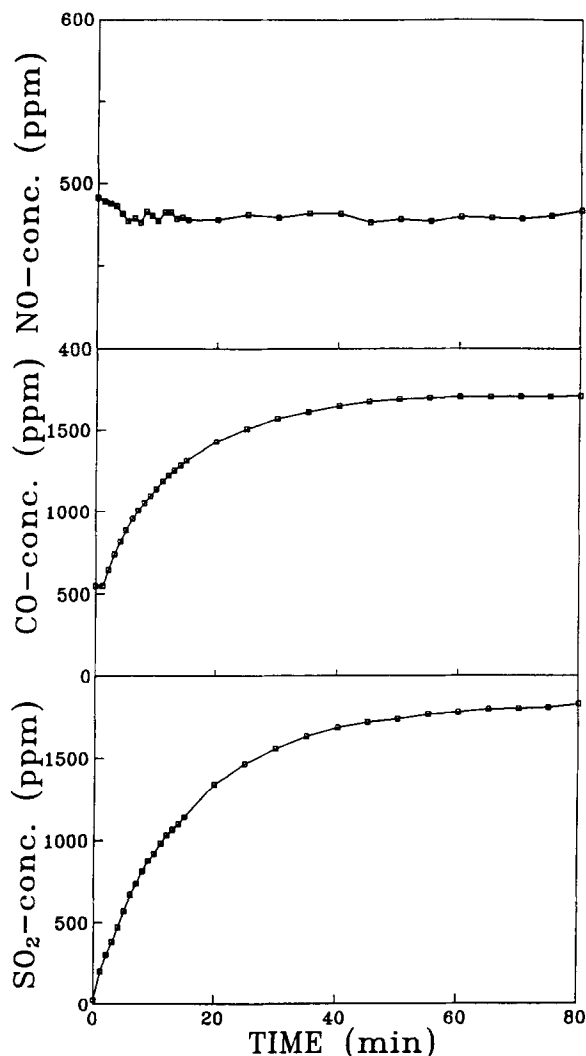


Figure 4 Outlet concentrations of NO, CO and SO₂ versus time for CO oxidation over Stevns chalk during sulfation

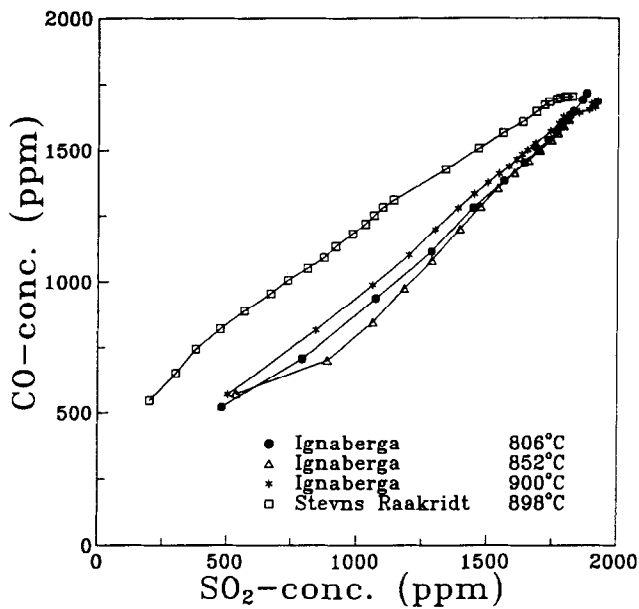


Figure 5 Outlet concentration of CO versus the outlet concentration of SO₂ for CO oxidation over Ignaberga limestone and Stevns chalk during sulfation

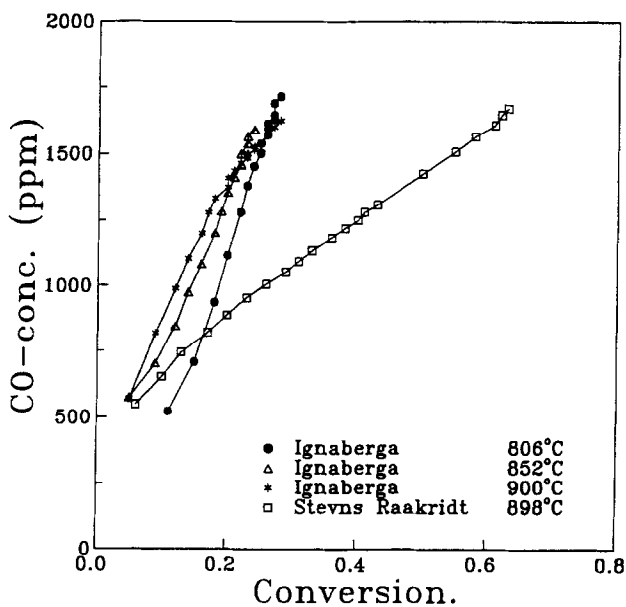


Figure 6 Outlet concentration of CO versus the CaO conversion to CaSO₄ for Ignaberga limestone and Stevns chalk

with Ignaberga limestone are shown in Figure 5 as outlet CO concentration versus outlet SO₂ concentration. A linear relationship is observed. Integration of the amount of SO₂ reacted, by means of a procedure described by Dam-Johansen³⁰, gives the CaO conversion to CaSO₄ as a function of time. The outlet CO concentration from the four experiments is plotted versus the CaO conversion in Figure 6, showing linear relationships. Similar experiments, where the SO₂ stream to the reactor was stopped during sulfation, verified that the presence of SO₂ had only minor or no influence on the CO oxidation, the CaO conversion to CaSO₄ being responsible for the observed decrease in the CO conversion.

Nitrogen chemistry on bed material

The experimental conditions are summarized in the last column of Table 1 and the reactor used is that shown in Figure 2. The results are only of a qualitative nature due to the problems in taking samples of bed material that contain a constant amount of char, and problems caused by the entrance of small amounts of oxygen into the reactor during loading with bed material.

When no NO or N₂O was present in the inlet gas (N₂) almost no formation of these nitrogen oxides was detected.

In the tests when 500 ppm NO or 100 ppm N₂O were added to the reactor a considerable reduction of these gases was observed. There was no N₂O measured during reduction of NO in this case with no O₂ in the gas. This is in agreement with other recent investigations³¹⁻³³, where it was shown that only insignificant amounts of N₂O were formed during NO reduction on char.

The addition of O₂ resulted in considerable CO production until the char was completely burned up. Without NO and N₂O present as reactants, both NO and N₂O were measured with a ratio between the N₂O and NO of about 0.05. Introducing 500 ppm NO, reduction of NO took place on the char. As the char was burned, its reducing effect diminished and the concentration of NO increased gradually towards its inlet value. Some N₂O was measured during the entire period of char combustion. The quantity of N₂O formed was small but it was greater with than without NO added.

Comparison of the on-line N₂O measurement with off-line gas chromatography showed that the on-line method may overestimate the N₂O concentration due to the presence of methane and a hitherto unidentified

Table 1 Conditions for the laboratory experiments

Parameter	Homogeneous oxidation of CO	Homogeneous conversion of HCN	Heterogeneous oxidation of CO	Nitrogen chemistry on bed material
Number of experiments	73	50	32	15
Residence time (s)	0.04-0.1	0.052-0.077	-	-
Temperature (°C)	850 ± 5	625-1125	800-900	800
Type of limestone	-	-	Ignaberga/Stevns chalk	-
Particle diameter (mm)	-	-	1.0-1.4 ^a , 0.85-1.0	<3
Bed material	-	-	-	Char, sand and ash (3 g)
Inlet concentration				
CO (ppm)	400-2500	1780 ± 30	1950 ± 50	1000
H ₂ O (%)	0-5	3.3 ± 0.2	-	-
HCN (ppm)	-	300 ± 10	-	-
NO (ppm)	0-500	475 ± 50	500 ± 10	0, 500
O ₂ (%)	0-4.5	2.0 ± 0.1	4.2 ± 0.1	0, 1.6
SO ₂ (ppm)	0-2000	0, 50, 500, 1250, 2000	1850 ± 75	-
N ₂ O (ppm)	-	-	-	0, 100

^aIgnaberga lime fragmented during heat-up

reaction product. Attention will be paid to this observation in further work.

DISCUSSION

Effects of excess lime feeding on NO emission

The results given in *Figures 4 to 6* show that CaO acts as a catalyst for CO oxidation. In part 1¹⁵, excess limestone was fed to the boiler to illustrate the effect on the emission of NO (part 1, *Figure 2*). The limestone catalytically decreases the CO concentration in the combustor and thereby removes CO from the catalytic reduction of NO over char. As a result, the NO emission increases. Even if this explanation contributes to the understanding of the interaction between sulfur retention and the observed increase of the NO emission, other factors, such as a change in the rate and the products of volatile N oxidation, can still be relevant. For example, it has been shown^{16–18,20} that volatile N in the form of NH₃ is rapidly oxidized to N₂ and NO catalysed by CaO. This oxidation of NH₃ is also catalysed by char and bed ash³⁴, but in a bed containing both char and an excess of CaO, a shift in the oxidation reactions towards a higher production of NO, leading to an increase in NO emission, could be expected. This alternative requires further investigation using bed material from a boiler with different contents of char and CaO.

Homogeneous effects of SO₂ on NO and N₂O emissions

It was shown in part 1 (*Figure 3*) that an addition of SO₂ to the combustion air increased the CO emission and decreased the NO emission. The result of the laboratory test (column 1 of *Table 1*) shows the same trend as the full-scale experiments; the CO oxidation is moderately inhibited by addition of SO₂. The increased CO concentration resulting will lead to a greater reduction of NO on char surfaces.

The second test shown in *Figure 3* (this part), on the other hand, does not give a straightforward answer. At low temperatures, the results trend in an opposite direction compared with the boiler measurements; an increase in the CO concentration as a result of the addition of a high concentration of SO₂ is accompanied by a smaller reduction of NO than in the case of a low concentration of SO₂. However, at high temperatures the situation for the NO is reversed to the same order as the boiler results, whereas CO is already almost consumed. As shown by the experiments of Hulgaard²⁸, higher residence times and higher inlet concentrations of CO may shift the set of curves towards lower temperatures. Since the operating conditions of the boiler (850°C) were close to the intersection of the curves in *Figure 3*, a definite conclusion is difficult to draw, except that SO₂ does influence the NO and the N₂O emissions homogeneously.

NO reduction and N₂O formation

In part 1 the N₂O formation during the NO supply test (part 1, *Figure 5*) was interpreted as an effect of the NO reduction on char surfaces. The results obtained in the laboratory tests only quantitatively indicate that NO may play a role in the formation of N₂O in connection with combustion of char and they are not sufficient to prove that such a formation is the cause for the increase in N₂O when NO was added to the combustor, shown

in *Figure 5* (part 1), or the reason for the rise in the N₂O concentration with combustion chamber height, which was observed in previous tests³⁵.

In part 1, *Figure 8*, the simultaneous changes of the N₂O and the NO concentrations are plotted for the cases investigated in the boiler. It is seen that the two 'homogeneous' cases, where a considerable change in the SO₂ concentration took place, imply a greater interdependence than the case of NO supply to the bed of silica sand when no change in the SO₂ concentration was involved. This leads to a suspicion that SO₂ may play a role in N₂O formation. However, the effects may have independent origins, as can be suspected for the 'heterogeneous' lime transient in part 1, *Figure 8*. Also, in this case the SO₂ concentration was almost constant, but the interrelation between N₂O and NO is stronger than in the case of NO supply to a bed of silica sand, and the factors influencing the concentrations of NO and N₂O could be independent of each other. As mentioned in part 1, the decomposition of N₂O on accumulated CaO surfaces is such an independent factor. This decomposition has been shown by Hansen *et al.*²³ to be almost independent of the CO concentration. The N₂O reduction thereby gradually increases as the CaO accumulates in the bed, in spite of the falling CO concentrations.

CONCLUSIONS

The tests carried out in the 12 MW CFB boiler and the complementary laboratory investigations lead to the following conclusions.

1. CaO has a catalytic effect on CO oxidation which can explain the increase of NO taking place when CaO is accumulated in the bed. Other factors which may also influence the NO emission, such as an increase of the formation of NO from volatile N oxidation on CaO surfaces, have to be further investigated for CFB conditions.
2. SO₂ influences both the CO oxidation homogeneously and the homogeneous formation of N₂O from HCN and NO through a catalytic effect of SO₂ on radical recombination reactions. This can explain the effects of changing SO₂ concentrations in the 12 MW boiler, but the surrounding environment, especially that of the SO₂-HCN-NO-CO system, has to be better determined.
3. No N₂O was detected as a result of NO reduction on char surfaces in the absence of oxygen. During addition of oxygen and nitric oxide to the reactor, an enhancement of the N₂O formation was measured, but further work is necessary to quantify this observation. Further attention has to be paid to the on-line gas analysis.

The laboratory results will be used for the development of detailed kinetic models with respect to the influence of SO₂ on the nitrogen chemistry.

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