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Andersson, S., Olausson, M., Phother Simon, J. et al (2019). High Temperature Corrosion and Dioxin Abatement Using Sulfur Recirculation in a Waste-to-Energy Plant. *Detritus*, 5(Mars): 92-98. <http://dx.doi.org/10.31025/2611-4135/2019.13784>

N.B. When citing this work, cite the original published paper.

HIGH TEMPERATURE CORROSION AND DIOXIN ABATEMENT USING SULFUR RECIRCULATION IN A WASTE-TO-ENERGY PLANT

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Article Info:

Received:
30 November 2018
Revised:
31 January 2019
Accepted:
20 February 2019
Available online:
31 March 2019

Keywords:

Sulfur recirculation
Waste-to-Energy
High temperature corrosion
Dioxin primary measure

ABSTRACT

Sulfur Recirculation is a novel technique for reducing the high temperature corrosion and dioxin formation in Waste-to-Energy plants by recirculating sulfur from the wet flue gas cleaning back to the boiler. This is achieved by separating SO₂ from the flue gas in a wet scrubber downstream of a HCl scrubber. H₂O₂ dosed into the scrubber reacts with SO₂ in the gas and produces a 15-25 wt% H₂SO₄ solution, which is injected into the boiler producing SO₂, thus creating a sulfur loop. The first permanent full-scale installation has been in operation in one of the two commercial full-scale Waste-to-Energy boilers at Maabjerg Energy Center (MEC) in Denmark since October 2016. The recirculated sulfur increased the gas concentration of SO₂ by a factor of 2-3 in the boiler, thereby enhancing the sulfation of corrosive alkali chlorides to non-corrosive alkali sulfates. The chlorine content of the superheater deposits decreased by 85%, and the superheater corrosion rate decreased by 40-90% during the first year of operation. The dioxin concentrations upstream of the dioxin removal system decreased by 75% and the dioxin emissions decreased by 72% with Sulfur Recirculation in operation. Furthermore, the sulfate containing effluent water was almost eliminated due to the increased sulfation of the ashes and deposits.

1. INTRODUCTION

The flue gases from Waste-to-Energy and biomass combustion are much more corrosive compared to fossil fuels, due to their higher concentrations of chlorine, alkali and heavy metals. In order to keep the resulting high temperature corrosion rates in the heat transfer surfaces at a manageable level, the steam parameters are lowered, resulting in not only lower corrosion rates but also a drop in electrical efficiency. Hence, in order to increase the green electricity production from these corrosive fuels, more effective corrosion mitigation techniques are needed.

The beneficial effect of sulfur addition on high temperature corrosion is well documented. This is primarily done by decreasing the presence of alkali chlorides. A successful way to minimize the corrosiveness of alkali chlorides is to sulfate them to corresponding alkali sulfates (Karlsson et al. 2011, Pettersson et al. 2011). This can be done by using elemental sulfur, sulfur-rich additives or by co-combustion with a suitable fuel, e.g. sludge or coal. (Kassman et al. 2013, Vainio et al. 2013, Viklund et al. 2009) Higher sulfur dioxide concentration also suppresses PCDD/F formation (Gullett et al. 1992). The suggested mechanism is deactivation of the Cl₂ forming catalyst Cu(II) by reacting it with

SO₂ to form CuSO₄. PCDD/Fs are formed in the boiler deposits by chlorination of aromatic structures in the unburnt soot particles (Hunsinger et al. 1997, 2002). Permanently high SO₂ concentrations in the flue gas cause sulfation of the boiler ash deposits, which leads to low chlorine concentrations in the deposits, which in turn suppresses the dioxin formation by de-novo synthesis in the deposits (Hunsinger et al., 2007).

Adding sulfur to the fuel, however, increases the amounts of residual products from the flue gas treatment considerably. The Sulfur Recirculation technique was developed in order to reduce the corrosion rate of the superheaters and the dioxin formation in Waste-to-Energy plants without the drawbacks of adding sulfur. Instead, the beneficial increase of the sulfur concentration in the boiler is achieved by recirculating sulfur from the wet flue gas cleaning back to the boiler. The recirculated sulfur will increase the gas concentration of SO₂ in the boiler and decrease the Cl/S ratio of the deposits and ashes, thus producing a less corrosive environment for the superheaters, which enables higher steam parameters and electrical efficiency (Hunsinger et al. 2014). Sulfur Recirculation was first developed and tested in pilot scale by Hans Hunsinger at Karlsruhe Institute of Technology (DE). The corrosion

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rates were not measured but the PCDD/F concentrations downstream of the boiler decreased slowly from several ng TEQ/Nm³ d.g. down to approximately 0.1 ng TEQ/Nm³ d.g. during a 10-day pilot scale test campaign with Sulfur Recirculation (Hunsinger et al., 2007). Subsequent full-scale tests for 1000 hours at the Renova Waste-to-Energy plant (SE) demonstrated a decrease in high temperature corrosion rates using corrosion probes, while the PCDD/F concentrations downstream of the boiler only decreased by some 25% (Andersson et al., 2014). The Sulfur Recirculation process is patented and Babcock & Wilcox Vølund AB (previously Götaverken Miljö AB) has a world-wide exclusive license.

Following the pilot and full-scale tests, the first permanent installation with the Sulfur Recirculation technology was taken into operation in 2016. The aim with this work is to study the long-term impact on deposit chemistry, corrosion rates and dioxin formation at this installation by comparing two full-scale Waste-to-Energy lines using fuel from the same bunker, one operating with Sulfur Recirculation and one without. This has been achieved by analysing boiler deposits, regular superheater corrosion measurements, test materials welded into the superheaters and flue gas dioxin measurements downstream of the boiler as well as in the stack.

2. MATERIALS AND METHODS

Babcock & Wilcox Vølund AB (previously Götaverken Miljö) has installed the Sulfur Recirculation technology in one of the two Waste-to-Energy lines at Maabjerg Energy

Center (MEC) in Denmark in order to combat high temperature corrosion, decrease dioxin formation and eliminate the sulfate waste water. This is the first commercial installation of this kind and it has been in operation since mid-October 2016.

2.1 MEC (Måbjerg Energy Center) Waste to Energy plant

The Waste-to-Energy plant produces electricity and district heating and consists of two grate fired incineration lines supplied by Babcock & Wilcox Vølund A/S with a capacity of 10 tons/h of waste each. The fuel consists of mainly household waste, with some addition of industrial waste and sludge. Both lines are operating with the same fuel mix, fed from one bunker. A third straw fired line is not covered in this paper. The mean fuel composition was 29 wt% household waste, 63 wt% industrial waste 5 wt% sludge and 3 wt% wood waste, during the first year of operation (October 2016-September 2017).

The flue gas treatment consists of an Electrostatic Precipitator, a HCl scrubber and a multistage scrubber for SO₂ removal, dioxin removal using ADIOX (Andersson et al. 2005) and flue gas condensation for producing additional district heating. The flue gas treatment has been operating with this configuration since 2004 and the PCDD/F emission limit has been met at all measurements so far.

2.2 Sulfur Recirculation installation at MEC

In 2016, Sulfur Recirculation was installed at Line 1 (L1) shown in Figure 1. The Sulfur Recirculation installation consists mainly of a storage vessel and dosage system

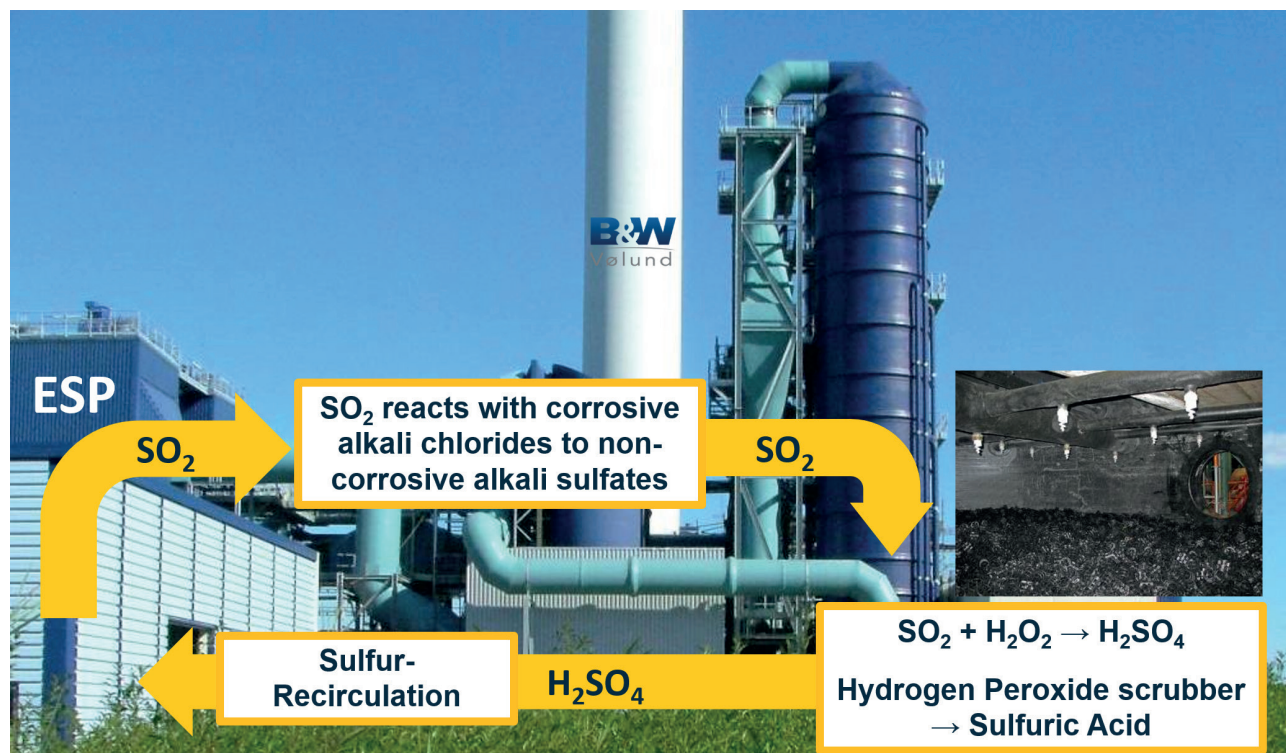


FIGURE 1: The flue gas treatment of MEC consists of an ESP (Electrostatic Precipitator), a HCl scrubber and a multistage scrubber for SO₂ removal, dioxin removal using ADIOX and flue gas condensation.

for hydrogen peroxide (H₂O₂), a raw gas analyzer for SO₂, equipment for transport and dosage of sulfuric acid into the furnace. SO₂ is now separated in the wet scrubber using H₂O₂, producing a 15-25 wt% H₂SO₄ solution, which is injected into the boiler producing SO₂, thus creating a sulfur loop. The sulfuric acid is sprayed through nozzles with atomization air, which produces a fine mist which evaporates rapidly. The dosage rate is controlled by a regulator, which maintains a fixed SO₂ setpoint. The recirculated sulfur will increase the gas concentration of SO₂ in the boiler and decrease the Cl/S ratio of the deposits and ashes, thus lowering chlorine content of the boiler deposits, in order to decrease the high temperature corrosion and the dioxin formation rates as well as dioxin emissions.

2.3 Deposits analysis

Deposits were collected during the planned plant shut-downs. The ICP-MS analyses of the deposits were performed by Eurofins Environment Testing Sweden AB, which are accredited by Swedac SS-EN ISO/IEC 17025. The sample preparation was made according to EN 14780-11/EN 15443-11/SS 187114-92/SS 187117-97. The chloride concentration was analysed according to SS 187185.

2.4 Corrosion rate measurements

Thickness measurements on the actual superheaters produced in 16Mo3, including different material samples welded into the superheaters, were performed in the two almost identical Waste-to-Energy lines at MEC. The material thickness of the superheaters and welded samples was measured with an Olympus 38DL Plus ultrasonic thickness gauge with a 0.01 mm resolution.

Materials from three different groups were selected: Low alloyed steels are represented by 16Mo3 and 13CrMo4-5; stainless steels to TP347H and W1.4841 (314); High alloyed to Sanicro 28 and 3RE28 (310). The material composition of the test materials is shown in Table 1.

2.4.1 Corrosion rate measurements in superheater coil samples

In order to perform long term exposures under realistic conditions, a number of different test materials were welded into the superheaters. In total, 8 test pieces, each with 6 different materials were welded into the two waste fired boilers. Two test pieces for each position times two superheater positions times two lines sums up to eight pieces. After half a year of operation, half of the samples were replaced with new 16Mo3 tubes and analyzed. After one year, the rest of the samples were analyzed.

2.4.2 Corrosion rates in full-scale superheaters

The tube wall thickness in the actual superheaters is measured periodically, twice per year, by Babcock & Wilcox Vølund A/S in order to assess the corrosion rates and plan maintenance need and super heater exchange which has previously been done approximately every 3 years.

2.5 Dioxin measurements

The dioxin emissions are normally measured two times per year using EN1948 and two samples per line. Eurofins

A/S in Denmark performed the sampling and Eurofins GfA in Germany (accred. Number DAkKS D-PI-14629-01-00) During the first two years of operation with ADIOX, 2004-2005, the dioxin concentration was additionally measured downstream of the ESP, upstream of the scrubbers in order to measure the dioxin removal efficiency of ADIOX.

3. RESULTS AND DISCUSSION

Both incineration lines, with and without Sulfur Recirculation (denoted Ref. and Rec. respectively) were operated with the same fuel mix (see section 2.1) from the same bunker. The process conditions for the first year of operation are summarised in Table 2.

The steam flow was almost identical for the two lines in the respective period. The measured mean SO₂ concentration was 2.3 times higher with Sulfur Recirculation. The difference between the calculated SO₂ dosage into the boiler, and measured SO₂ concentration at the boiler outlet is quite small and is partly due to measurement inaccuracy and partly due to the fact that some of the sulfur ends up in the water treatment and is not recirculated back to the boiler.

At this plant, Sulfur Recirculation almost entirely decreased the need for costly road transports of effluent sulfate water for the Sulfur Recirculation line, since most sulfur from the waste now ends up in the ashes instead of creating a surplus dilute Na₂SO₄ solution. The sulfur

TABLE 1: Composition of the investigated materials in wt.% (Fe bal.).

Material	C	Cr	Ni	Si	Mn	Others
16Mo3	0.16	0.16	0.15	0.28	0.70	P 0.01; S 0.002; Mo 0.31; Cu 0.27; V 0.003; Al 0.027
13CrMo4-5	0.13	0.9	0.03	0.26	0.53	P 0.006; S 0.004; Mo 0.48; Sn 0.002; Cu 0.04
TP347H	0.07	17.4	10.2	0.27	1.09	P 0.033; S 0.007; Nb 0.85
W1.4841 (314)	0.06	24	20	1.9	1.7	P 0.02; S 0.0004
Sanicro 28 (Composite)	0.009	26.6	30.5	0.42	1.64	P 0.017; S 0.0008; Mo 3.32; Co 0.097; Cu 1.01; Al 0.03; N 0.051
3RE28 (Composite 310)	0.014	25.5	21.35	0.43	1.75	P 0.013; S <0.001; Mo 0.02; Ti 0.003; Cu 0.02; Al 0.034; Nb <0.01; N 0.097

TABLE 2: Summary of process conditions for the superheater material test with and without Sulfur Recirculation (denoted Rec and Ref respectively).

		Exposure time (h)	Steam flow (t/h)	SO ₂ dosage (mg/Nm ³ , d.g.)	SO ₂ conc. (mg/Nm ³ , d.g.)
Ref	first ½ yr	4053	40.7	0	225
Ref	second ½ yr	3283	41.0	0	287
Rec	first ½ yr	4008	40.7	477	530
Rec	second ½ yr	4188	40.9	615	668

content of the waste varies with time, which may create periods of sulfur surplus and deficit respectively. Most of these variations are being evened out by a storage vessel for H₂SO₄.

Analysis of boiler deposits showed that Sulfur Recirculation decreased the chlorine concentrations, thus creating a less corrosive environment in the boiler. Furthermore, the lower chlorine content of the boiler deposits also significantly decreased the dioxin formation rates as well as dioxin emissions.

3.1 Superheater deposit composition

The chlorine concentration decreased by 85% in the deposits from superheater 2B with Sulfur Recirculation (Table 3). The difference in deposit chlorine concentration was much larger than the difference in SO₂ concentration. This result indicates that a simple equilibrium does not adequately describe the deposit sulfation, which is not surprising considering the complex nature and multitude of mechanisms involved in deposit formation. One hypothesis is that the effect of higher SO₂ concentrations is two-fold: Firstly, higher in-flight sulfation decreases the gas phase alkali chloride concentration and thereby the alkali chloride condensation onto the colder superheater surfaces. Secondly, the already lower alkali chloride concentrations in the deposits are further sulfated by SO₂. This two-fold effect may explain the observed results.

3.2 High Temperature Corrosion of superheaters – impact of Sulfur Recirculation

The results show that the sulfur recirculation has a very positive effect by drastically reducing the corrosion rate of 347H stainless steel and considerably reducing the corrosion rate of the lower alloyed steel 16Mo3 and 13CrMo4-5 used in the tests.

3.2.1 Corrosion rates in superheater coil samples

In order to measure the corrosion rate in the same conditions as the full scale superheaters, different material samples were welded into the actual superheaters and extracted after ½ and 1 year of operation respectively. The corrosion rate of the fixed installed superheater test mate-

rials in mm/year was measured by an ultrasonic thickness gauge at 4 different positions per ring: wind, lee and both sides. The standard deviation of the initial thickness measurements was in the range of 0.1 mm, the measured corrosion rates varied substantially between different rings, even at the same position. Therefore, mean values were formed for all rings, grouped by material type in Figure 2: Low alloyed corresponds to 16Mo3 and 13CrMo4-5; stainless steels to TP347H and W1.4841 (314); High alloyed to Sanicro 28 and 3RE28 (310). The low alloyed mean corrosion rate was some 50% lower and max some 40% lower in the Sulfur Recirculation line. The stainless steels mean corrosion rate was almost 80% lower, while the max values were approximately 35% lower. It was difficult to draw any conclusion from the high alloyed materials since the corrosion was in the order of the standard deviation of the initial thickness, which is reflected by the large number of measurements with apparent material growth.

The effect of sulfur on corrosion of low alloyed steels and on alkali chloride induced corrosion on stainless steels has previously been investigated. Four different mechanisms have been identified that may explain the results. Increasing the amount of SO₂ in the superheater region may:

- Transform alkali chlorides to alkali sulfates. This gives less reactive alkali on the superheater breaking down the Cr rich protective scale of stainless steels. A longer incubation time before breakaway may explain the larger effect on stainless steels (Jonsson, et al., 2009, Pettersson et al 2005, Pettersson et al., 2011);
- Transforming alkali chlorides to alkali sulfates gives less Cl on the superheaters both on low alloyed and stainless steels. Chlorine is known to increase the corrosion rate of steels and stainless steels (Folkesson, et al., 2011, Nielsen, 2000, Spiegel, 2006, Grabke et al., 1995);
- Iron oxide grows slower in the presence of small amounts of SO₂ in low alloyed steels and in stainless steels after breakaway oxidation (Jonsson et al., 2007, Järnäs et al., 2003);
- Formation of iron sulfide at the metal/oxide interface may increase spallation of the oxide scale (Jonsson et al., 2007);
- The results from the investigation of the superheater coil samples are well in line with three of the proposed mechanisms. The largest impact could be observed on the stainless steels where all three mechanisms are expected to be at play. However, no increased tendency for spallation in the presence of an increased SO₂ level could be identified during the exposures.

3.2.2 Corrosion rates in full-scale superheaters

TABLE 3: Percentage (wt%) of chlorine in the superheater 2B deposits after ½ year of operation.

	Superheater 2B deposits
Reference	1.84
Recirc.	0.27

The mean corrosion rates in the actual superheaters were calculated from material thickness measurements at 32 positions after 1 year of operation prior to Sulfur Recirculation operation (here denoted Reference) and after 1 year of operation with Sulfur Recirculation in the same boiler. The corrosion rates are calculated from the decrease in material thickness over a given period of time, and the results from the Sulfur Recirculation line is shown in Figure 3. The corrosion rates of the low alloyed superheaters are

comparable to the low alloyed superheater test material results in Figure 2. Superheaters 2A and 2B are the ones with the shortest life time, and the measured decrease in corrosion rates of 40-50% means that the life time is extended from 3 years to 5-6 years. Alternatively, the steam temperature can potentially be raised without increasing the corrosion rates, resulting in a higher electrical efficiency (Hunsinger and Andersson, 2014).

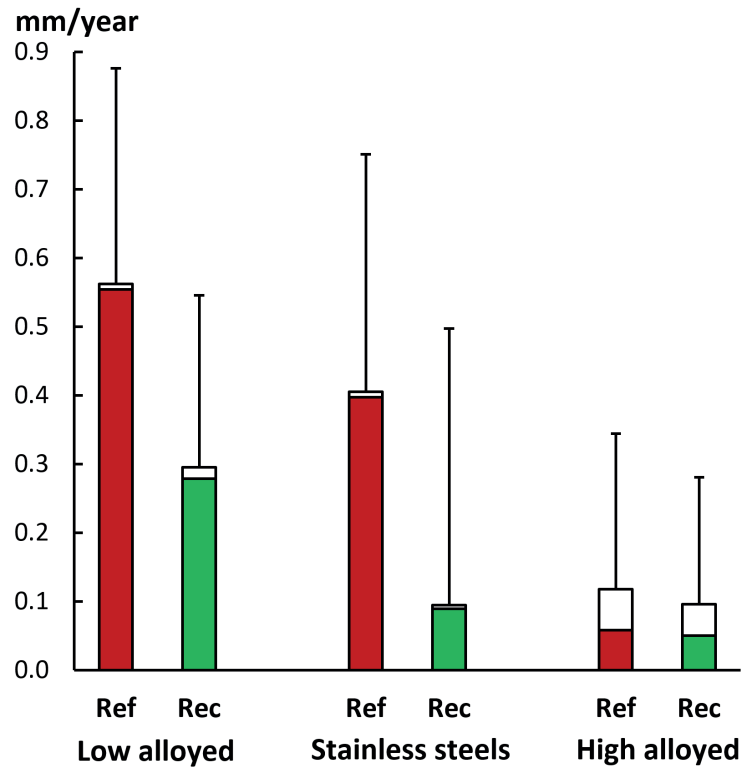


FIGURE 2: The filled bars denote the mean corrosion rate of fixed installed superheater test materials in mm/years. The entire bars denote the corrosion rate when all measurements with apparent material growth was discarded. The top error bar reading denotes the mean value of the max corrosion rate per ring. Ref = Reference line, Rec = Sulfur Recirculation line.

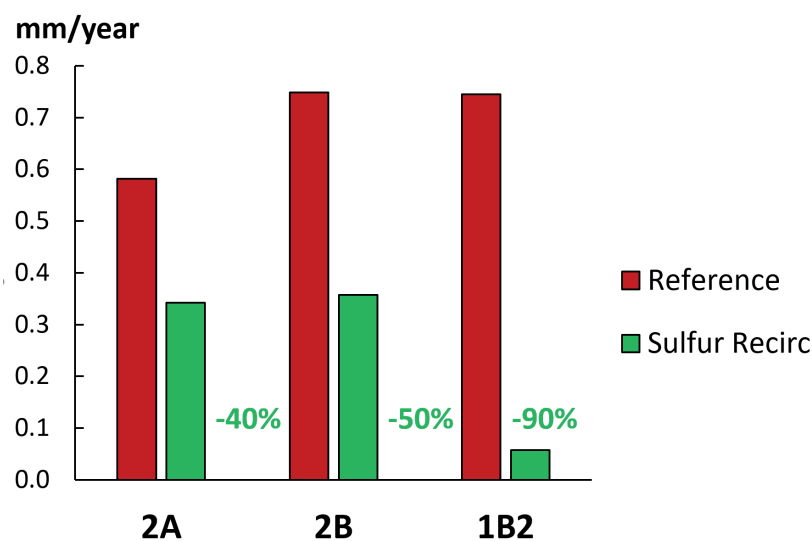


FIGURE 3: Mean corrosion rates in the actual superheaters for 1 year prior to Sulfur Recirculation operation (here denoted Reference) and 1 year of operation with Sulfur Recirculation in the same boiler.

3.3 Dioxin formation and emissions

With Sulfur Recirculation in operation, the mean dioxin stack concentration decreased from 0.020 ng TEQ/Nm³ d.g. @11% O₂ to 0.0055 ng TEQ/Nm³ d.g. @11% O₂ as seen in Table 4., corresponding to a decrease of 72%. Notably, all 8 stack concentrations measured during Sulfur Recirculation were below 0.008 ng TEQ/Nm³ d.g. @11% O₂ compared to only 2 out of 32 for normal operation. In order to compare the raw gas concentration upstream of the dioxin removal technology ADIOX, two measurements were performed with Sulfur Recirculation in operation. The mean raw gas concentration decreased from 1.87 ng TEQ/Nm³ d.g. @11% O₂ to 0.47 ng TEQ/Nm³ d.g. @11% O₂, corresponding to a decrease of 75% according to Table 5.

3.4 Stack emissions and residual products

The stack emissions of both the line with Sulfur Recirculation and the one without fulfilled the strict emission limits set by the EU Industrial Emissions Directive (2010/75/EC) and no increased adverse effects on the environment were anticipated or detected from Sulfur Recirculation as seen in Table 6. The sulfation of the alkali chlorides in the fly ashes and deposits is estimated to increase the HCl concentration in the flue gas at the boiler exit by less than 10%, but the stack concentration was far below the emission limit and practically unchanged due to the extremely high HCl removal efficiency of the 2-stage wet HCl scrubbers. After passing the waste water treatment system, these chlorides end up in the ocean instead of being landfilled, which is preferable from an environmental perspective. The emission values for SO₂ and NO_x were practically unchanged, while CO and Hg were reduced with Sulfur Recirculation.

4. CONCLUSIONS

Results from the first permanent full-scale installation of the Sulfur Recirculation technique are presented in this paper. Sulfur Recirculation changes the deposit chemistry in the MEC Waste-to-Energy plant, and less corrosive species end up on the superheaters. This resulted in 40-90% lower corrosion rates of the low alloyed superheaters, which will increase the superheater life time and decrease the cost for replacements. Alternatively, the steam data and electricity production may be increased in this plant and also in new plants using Sulfur Recirculation. Sulfur Recirculation almost entirely decreased the need for costly road transports of effluent sulfate water for the Sulfur Recirculation line, since most sulfur from the waste now ends up in the ashes instead of creating a surplus dilute Na₂SO₄ solution. The dioxin concentrations upstream of the ADIOX dioxin removal system decreased by 75% and the dioxin emissions decreased by 72% in the MEC Waste to Energy plant with Sulfur Recirculation in operation.

ACKNOWLEDGEMENTS

The Måbjerg Energy Center Waste to Energy plant is gratefully acknowledged for providing the dioxin emission data. This work has been supported by KME (Cooperation Programme Materials technology) and Chalmers HTC

TABLE 4: PCDD/F emissions in ng TEQ/Nm³ d.g. @11% O₂ from the MEC Waste-to-Energy plant (DK) for line L1 with Sulfur Recirculation in operation (L1 Rec) and in normal operation (L1 Ref), as well as for line L2 (L2 Ref).

	L1 Rec	L1 Ref	L2 Ref
2013-11-20		0.029	0.038
2014-01-08		0.030	0.041
2014-02-25		0.021	0.042
2014-02-25		0.019	0.038
2014-09-12		0.013	0.016
2014-09-13		0.013	0.017
2015-03-17		0.014	0.018
2015-03-18		0.014	0.017
2015-11-11		0.005	0.006
2015-11-12		0.004	0.008
2016-03-09		0.013	0.022
2016-03-10		0.010	0.021
2016-11-03	0.005		0.045
2016-11-04	0.005		0.046
2017-01-11	0.006		0.013
2017-01-11	0.006		0.013
2017-09-19	0.007		0.008
2017-09-20	0.005		0.009
2018-03-21	0.006		0.012
2018-03-22	0.005		0.012

TABLE 5: PCDD/F raw gas concentrations in ng TEQ/Nm³ d.g. @11% O₂ (downstream of the ESP) from the MEC WtE plant (DK) for line L1 with Sulfur Recirculation in operation (L1 Rec) and in normal operation (L1 Ref), as well as for line L2 without Sulfur Recirculation (L2 Ref).

	L1 Rec	L1 Ref	L2 Ref
2004-10-26		1.1	
2004-11-17		1.3	0.71
2004-12-08		2.2	1.18
2005-08-02		4.8	2.3
2005-11-08		1.6	1.6
2017-09-19	0.45		
2017-09-20	0.49		

TABLE 6: One year mean values from the two lines with (Rec) and without (Ref) Sulfur Recirculation in mg/Nm³ d.g. @11% O₂ together with the ½h emission limits.

	HCl	SO ₂	NO _x	CO	Hg
Emission limit	10	50	200	100	0.05
Reference	<0.5	2.2	158	10	0.024*
Recirculation	<0.5	2.7	162	6.8	0.011*

* Mean value of 6 measurements per line

(High Temperature Corrosion centre), financially supported by the Swedish Energy Agency and member companies.

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