Report: 2011/18 December 2011

IEAGHG SPECIAL WORKSHOP ON OXYFUEL COMBUSTION: ADDRESSING SO₂, SO₃, Hg AND CORROSION ISSUES IN OXYFUEL COMBUSTION BOILER AND FLUE GAS PROCESSING UNITS



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DISCLAIMER AND ACKNOWLEDGEMENTS

IEAGHG supports and operates a number of international research networks. This report presents the results of a workshop held by one of these international research networks. The report was prepared by Stanley Santos of IEAGHG as a record of the events of that workshop.

IEAGHG would like to thank the participants and also the UK Technology Strategy Board for supporting this meeting. Likewise to Doosan Babcock for the co-ordination of the TSB Project.

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Executive Summary

This workshop was established to address specific issues arising from Oxyfuel Combustion processes, namely the corrosion associated with $SO_2 SO_3$ and Hg. It is recognised that the Oxyfuel process could involve higher concentration of these species in the flue gas, and the captured CO_2 stream.

The meeting was well attended, with 80 participants from 17 countries around the world. This mix of participants ensured that discussions encompassed as many different views and regional variants as possible, while tackling the issues present on the agenda.

The IEA Greenhouse Gas R&D Programme (IEAGHG) has been proactive in providing a forum to key players and stakeholders in the development of Oxyfuel Combustion technologies for some years, with two previous meetings taking place in Sweden (2008) and Germany (2009) to discuss the various issues relevant to CO_2 specifications and its impact to the operation of oxyfuel combustion power plants.

As a result of these previous workshops, the agenda of this workshop was aimed at tackling 3 main issues; the fate of sulphur and mercury during Oxyfuel combustion, discussions on the acceptable techniques for measurements of these compounds, and discussions on the corrosion mechanisms present in different boiler options within Oxyfuel.

The meeting consisted of two full days of presentations, including 3 keynote addresses presented by Prof. Terry Wall (Newcastle University), John Pavlish (EERC) and Prof. Axel Kranzmann (BAM). These addresses served to review the current understanding on the fate of sulphur and Hg, and corrosion issues during Oxyfuel combustion. The subsequent presentations covered topics such as:

- Fundamental research on:
 - The fate of sulphur measurement techniques and discussion on sulphur balance,
 - Corrosion issues in high and low temperature regime during Oxyfuel boiler operation,
 - Corrosion and ash deposition during Oxyfuel combustion,
 - Performance of the flue gas processing units in control of NOx, SOx and Hg,
- Large scale pilot testing experience.



Addressing SO₂/SO₃/Hg/Corrosion Issues in Oxyfuel Combustion Boiler and Flue Gas Processing Units

Rembrandt Hotel, London, UK 25th and 26th January 2011

1. Background

The CO_2 captured from an Oxyfuel combustion power plant differs significantly to the CO_2 captured from pre- or post- combustion technologies. Depending on the clean up process applied, it is possible that CO_2 / flue gas from oxyfuel combustion processes could have higher levels of inert gases and oxygen; some quantity of sulphur and nitrogen species; and smaller amounts of minor species like trace metals and halogens.

An important consideration to any oxyfuel combustion power plant is to define the CO_2 specification required by the CO_2 transport and storage, in addition to the specifications required for the safe operation of the oxyfuel combustion boiler, coal mills and the CO_2 processing unit.

It should be noted that various processes are available for the removal of the non- CO_2 components from the flue gas of an Oxyfuel combustion boiler. This includes techniques such as the reduction of non- CO_2 components in the furnace during oxyfuel combustion; treatment of the flue gas during the flue gas processing steps (i.e. ESP, FGD, FGC) and additional removal of non- CO_2 components in the CO_2 processing unit and during compression.

It is therefore recognised that an important aspect in the design, engineering and operation of an oxyfuel combustion power plant with CO_2 capture, is to understand the different operating parameters that could impact the quality of the flue gas produced, both from the Oxyfuel combustion boiler and subsequent flue gas and CO_2 processing units.

The IEA Greenhouse Gas R&D Programme (IEAGHG) has been active in providing a forum to key players and stakeholders in the development of oxyfuel combustion for power generation with CO_2 capture. Two previous meetings were held in Stockholm, Sweden (2008) and Cottbus, Germany (2009) to discuss the various issues relevant to the CO_2 specifications and its impact on the operation of the oxyfuel combustion power plant.

To follow these two workshops, this special workshop was organised to specifically address three relevant issues:

- Fate of sulphur and Hg during Oxyfuel combustion,
- Discussion of acceptable techniques in the measurements of SO₃ and Hg during Oxyfuel combustion,
- Discussion of the corrosion mechanisms concerning different boiler operating regimes of oxyfuel combustion.

The meeting was held at the Rembrandt Hotel, London on the 25th and 26th of January 2011. This workshop is partly supported by UK Technology Strategy Board under the TSB Project.



1.1 Workshop Overview

The workshop consisted of two full days, with 3 keynote addresses presented by Prof. Terry Wall (Newcastle University), John Pavlish (EERC) and Prof. Axel Kranzmann (BAM) which review the current understanding on the fate of sulphur and Hg, and corrosion issues during oxyfuel combustion.

The keynote addresses were followed by 24 presentations covering the following topics:

- Fundamental research on the fate of sulphur measurement techniques and discussion on sulphur balance
- Fundamental research on corrosion issues in high and low temperature regime during oxyfuel boiler operation.
- Fundamental research on corrosion and ash deposition during oxyfuel combustion
- Fundamental research on performance of the flue gas processing units in control of NOx, SOx and Hg.
- Large scale pilot testing experience

This report summarises the different issues discussed during the workshop.

2. Participants

The workshop brought together 80 participants from industry, research institutes and universities covering at least 17 countries worldwide. The delegate list is appended as Appendix 1.

3. Agenda

The agenda of the meeting is presented in Tables 1a and 1b.



Table 1a: Day 1 Agenda of the Special Workshop

Agenda (Day 1):	Rembrandt Hotel, London, 25 th January 2011
09.45 - 10.00	Welcome and Other Announcements
09.43 - 10.00	Stanley Santos, IEA Greenhouse Gas R&D Programme, UK
	Stancy Sanos, ILA Oremiouse Gas Red Hogramme, OK
	Keynote Session:
Look	ng at the Challenges of Understanding SO2/SO3/Hg and Corrosion Issues During Oxyfuel Combustion Conditions
10.00 - 10.45	Keynote Address 01:
	"Challenges in the Understanding of Sulphur Evolution and Capture During Oxyfuel Combustion"
	Terry Wall, Newcastle University, Australia
10.45 - 11.30	Keynote Address 02:
	"Understanding the Fate of Hg during Oxyfuel Combustion"
	John Pavlish, Energy & Environmental Research Center, University of North Dakota, USA
11.30 - 12.15	Keynote Address 03:
	"The Challenge in Understanding the Corrosion Mechanisms Under Oxyfuel Combustion Condition"
	Axel Kranzmann, Federal Institute of Materials Research, Germany
10.15 10.20	
12.15 - 13.30	Lunch and Coffee
	Session 01:
Em	adamental Research in the Fate of Sulphur during Oxyfuel Combustion – Measurement Experiences and Results
13.30 – 13.55	"Experiences and Results of SO ₃ Measurements Performed under Oxy-Coal Fired Conditions"
15.50 15.55	Jeorg Maier, IFK University of Stuttgart, Germany
13.55 - 14.20	"SO ₃ Measurements under Oxy-Coal Conditions in Pilot-Scale PC and CFB Combustors"
10100 11120	Eric Eddings, Jiyoung Ahn, Liyong Wang and Ryan Okerlund, Utah University, USA
	Andrew Fry, Reaction Engineering Ltd., USA
14.20 - 14.45	"Experiments and Modelling of the Fate of Sulphur in Oxy-Coal Combustion"
	Klas Andersson, Chalmers University, Sweden
14.45 - 15.10	"SO ₂ /SO ₃ /Hg and corrosion Issue Results From DOE/NETL Existing Plants Oxy-combustion projects"
	Timothy Fout, NETL, USA
15.10 - 15.30	Coffee Break
	Session 02:
	ndamental Research in the Fate of Sulphur during Oxyfuel Combustion – Measurement Experiences and Results "SO ₃ Control in Oxyfuel Applications"
15.30 - 15.55	Bhupesh Dhungel, Connie Ellul, Jacqueline Gibson and Dave Fitzgerald, Doosan Babcock, UK
15.55 - 16.20	"Sulfur trioxide on-line measurement technique for power plants"
15.55 - 10.20	Alexander Fateev and Sønnik Clausen, Risø DTU, Denmark
16.20 - 16.45	"Bench-Scale Fluid-Bed Experiments of SO ₂ /SO ₃ formation and Sulfur Capture in N ₂ /O ₂ and CO ₂ /O ₂ Environments"
10.20 - 10.43	Eric Eddings, Utah University, USA
16.45 - 17.10	Sulphur Issues for Oxyfuel FBC Technology
10.15 17.10	Edward Anthony, Canmet Energy, Canada
19.00	Workshop Dinner – This is partially supported by UK Technology Strategy Board)



Table 1b: Day 2 Agenda of the Special Workshop

•	Rembrandt Hotel, London, 26 th January 2011
08.10 - 08.15	Administrative Announcements Stanley Santos, IEA Greenhouse Gas R&D Programme, UK
	Session 03:
	damental Research in Behaviour of Mercury during Oxyfuel Combustion – Experimental and Modelling Results
08.15 - 08.40	"Advanced AQCS for Oxy-fuel Combustion System; Controlling Mercury & SO ₃ " Hirofumi Kikkawa, Kure Research Laboratory, Babcock-Hitachi K.K.
08.40 - 09.05	"Mercury Speciation & Emission from Pilot-Scale PC Furnaces under Air- & Oxy-fired Conditions"
00.10 07.05	Andrew Fry*, Brydger Van-Otten, Brad Adams, Reaction Engineering International, USA;
	Larry Bool, Praxair, Inc., USA
09.05 - 09.30	"Modelling of Hg/SO ₂ /SO ₃ Behaviour in Oxyfuel Combustion Systems"
00.20 0.55	Maryam Gharebaghi, Kevin J. Hughes, Lin Ma, Richard T. J. Porter and Mohamed Pourkashanian, Leed University, UK
09.30 - 9.55	"Impact of Oxyfuel Operation on Emissions, Deposition and Ash properties based on Experience with E.ON's 1MWth combustion Test Facility"
	Robin Irons and David Couling, E.On New Build and Technology Ltd., UK
09.55 - 10.20	"Deposition Behavior and Superheater Corrosion under Coal Fired Oxyfuel Conditions"
	Jeorg Maier and Gosia Stein-Brzozowska, IFK, Univ. Stuttgart
10.20 - 10.35	Coffee Break
	Session 04:
10.35 - 11.00	Fundamental Research in Corrosion and Ash Deposition – Measurement Experiences and Results "Corrosion in an Oxyfuel boiler - first results from Vattenfall pilot plant"
10.55 - 11.00	Alexander Gerhardt, Vattenfall Research and Development, Germany
11.00 - 11.25	"Fire-Side Corrosion Rates of Heat Transfer Surface Materials for Air- and Oxy-coal Combustion"
11.00 11.25	Andrew Fry*, Brad Adams, Kevin Davis, Dave Swensen, Shawn Munson, Reaction Engineering International, USA;
	William Cox, Corrosion Management, UK
11.25 - 11.50	"Experimental Study Results on Corrosion Issues in Oxyfuel Combustion Process"
	Toshihiko Yamada and Takashi Kiga, IHI, Japan
11.50 - 12.15	"Fireside Corrosion in Oxy and Air-Firing Combustion Environments" Steve Kung, Babcock and Wilcox, USA
12.15 - 13.25	Lunch and Coffee
	Session 05:
	Fundamental Research in Corrosion and Performance of Flue Gas Processing Units
13.25 - 13.50	"Impact of Oxyfuel Operation on Corrosion in Coal Fired Boilers Based on Experience with E.ON's 1MWth Combustion
	Test Facility"
10.50 14.15	Colin Davis, E.On New Build and Technology Ltd., UK
13.50 - 14.15	"Corrosion Performance of Boiler Materials Under CaCO ₃ -CaSO ₄ deposit" S. Tuurna, P. Pahianna, VITT Taghnigal Research Cantra of Finland, Espace, Finland
	S. Tuurna, P. Pohjanne, VTT Technical Research Centre of Finland, Espoo, Finland E. Coda Zabetta, Foster Wheeler Energy Oy, Varkaus, Finland
14.15 - 14.40	"Performance of a wet Flue Gas Desulphurisation Pilot Plant under Oxy-fuel Conditions"
	Brian Hansen, DTU; Søren Kiil, Dong Energy, Denmark
14.40 - 15.05	"Desulfurization Options for Oxyfuel Combustion"
	Wuyin Wang, Jörgen Grubbström, Alstom, Sweden
15.05 - 15.30	"Pilot-scale Testing of an In-situ Process for Control of SO ₂ , NOx and Mercury Emissions from Oxy-Coal Combustion" Kourosh Zanganeh, CANMET Energy, Canada
15.30 - 15.45	Coffee Break
	Carrier OC
	Session 06: Experiences in Large Scale Combustion Test Facilities
15.45 - 16.10	"Sulphur Behaviour in Flue Gas Recycle and Gas Cleaning during Oxyfuel Combustion of Lignite"
	Jinving Yan, Vattenfall Research and Development, Sweden
16.10 - 16.25	"Sulphur Balance Assessment During Demonstration of the Doosan Power Systems 40MWt OxyCoal [™] Burner"
	Jim Rogerson, Euan Cameron, David Sturgeon, Clive McGhie and Dave Fitzgerald, Doosan Babcock, UK
16.25 - 16.50	"SO ₃ emissions from a tangentially-fired pilot scale boiler operating under oxy-combustion conditions"
	Michael Clark, James Kenney, Armand Levasseur, Shin Kang, Alstom Power, USA
1	
16.50 - 17.00	Closing Remarks
	Stanley Santos, IEA Greenhouse Gas R&D Programme



4. Presentations Highlights

4.1 Keynote Presentations

The three keynote presentations provided an overview of the current state of understanding of the fate of sulphur, mercury and corrosion during oxyfuel combustion.

4.1.1 Sulphur Impacts during Pulverised Coal Combustion in Oxyfuel Technology for CO2 Capture and Storage [1]

Prof. Terry Wall, Newcastle University, NSW, Australia

This presentation is an update to the recent review published in the Progress in Energy and Combustion Science (Vol. 37, pp. 69-88). It provides a review of various laboratory and pilot work to identify the impact of the sulphur impurities throughout the Oxyfuel combustion process.

The presentation discussed the fate of sulphur and its impact:

- During combustion which could lead to higher concentration of COS and H₂S in the low oxygen combustion regime (i.e. reducing condition) of the furnace that are typical to any low NOx burners,
- Possible effect of the sulphur (due to higher concentration of SO₂ and SO₃) on the slagging and fouling propensity in the radiant and convective sections,
- Possible impact on the performance of the SCR and SNCR (if operational) due to higher SO₃ concentrations,
- Operational enhancement in the ESP (due to higher SO₃) or possible competition of sulphur species (especially SO₃) with Hg removal in the fabric filter,
- Considering the determination of the dew point; specifically relevant to the handling of the secondary RFG,
- Considering the removal of H₂SO₄ in the flue gas condenser,
- Understanding the SOx and NOx reaction during compression

The presentation concluded with some important questions which highlight the different issues that need to be addressed:

- Reliability of the published data especially on SO₃.
- Questions regarding the closure of the sulphur balance; are we satisfied with at least 20% error?
- Determination of the acid dew point; can we rely on the correlation of acid dew point, H₂O and SO₃ used typically in any conventional air blown combustion?
- Optimal removal of the sulphur species (where, and to what extent?).

4.1.2 Understanding the Fate of Hg During Oxyfuel Combustion [2]

John Pavlish, Energy & Environmental Research Center, N. Dakota, USA

This presentation provided an overview on the fate of Hg during air fired conditions. Using this as a starting point, the different parameters that could impact the behaviour (i.e. speciation) of Hg during combustion was reviewed. Based on this understanding, the changes in the behaviour of Hg under oxyfuel combustion conditions can be identified by comparing the flue gas characteristics from the oxyfuel combustion and the air fired case.

In this presentation, it was highlighted that during oxyfuel combustion, the form of Hg and its total concentration could vary throughout the system because of the flue gas recycle loops. The



presentation presumed that behaviour of Hg may be similar to the behaviour in a high-acid-gas environment. The presentation further emphasised that the unique flue gas characteristics of oxyfuel combustion flue gas may:

- Change the partitioning and emission of trace elements, including mercury, selenium, etc.,
- Require validation and/or modification of existing mercury-sampling protocols,
- Require mercury control strategies to be re-evaluated for application to oxyfuel combustion.

The presentation provided the following preliminary conclusions:

- Flue gas with varied and/or highly concentrated CO₂ may require modifications to CMMs for proper measurement,
 - Measurements were affected in relation to mass flow controllers specific to CMMs.
 - Precipitation occurred in the impinger solution (wet chemistry method), resulting in a biased measurement of gas-phase mercury.
- Flue gas dilution appeared to improve measurement accuracy of total gas-phase mercury, however, the mercury speciation measurement is not reliably accurate,
- Depending on the flue gas-recycling scenario and enrichment of acid gases, mercury oxidation and Hg(g)-to-Hg(p) (gas-to-particulate) are affected,
- Bench-scale sorbent breakthrough tests under enriched-CO₂ conditions show similar performance to those observed under coal air combustion conditions with high-acid-gas concentrations.

Additionally, this presentation highlighted the following uncertainties:

- Because of the flue gas recycle loop, it is assumed that Hg concentration would be higher under oxyfuel combustion than air combustion but how much is the increase? Further verification and validation of this assumption is necessary.
- Risk relevant to corrosion caused by Hg (which also causes aluminium embrittlement) should be evaluated under oxyfuel combustion what is the probability? It is important to identify the point of accumulation that could be susceptible to Hg related corrosion.
- Acid gas environment may enhance and exacerbate Hg's amalgamating behaviour to what extent?
- Will existing flue gas Hg control be as effective under higher acid concentration?
- What could be the trace metal partitioning during oxyfuel combustion?
- What impact will Hg have on compression equipment?

The presentation underlined the lack of data which in turn impedes the understanding of the fate of Hg during oxyfuel combustion. It was recommended that R&D activities should be pursued to address these issues.



4.1.3 The Challenge in Understanding the Corrosion Mechanisms under Oxyfuel Combustion Conditions [3]

Prof. Axel Kranzmann, BAM, Germany

During oxyfuel combustion, different materials could be subjected to various temperature regimes which could involve different corrosion mechanisms as illustrated in Figure 1.

Under oxyfuel combustion conditions, the main species (other than O_2) that play a major role in various corrosion mechanisms are: SOx, NOx, CO_2 and H_2O . It should be noted that typical oxyfuel combustion flue gas contains high partial pressure of CO_2 and water; and higher concentration of NO_x , SO₂ and SO₃ when compared to air combustion flue gas.

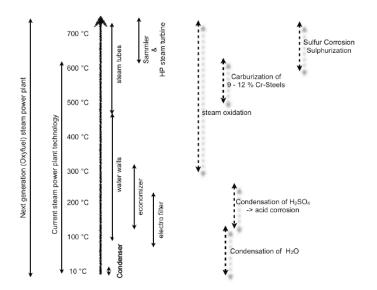


Figure 1: Different Temperature Regimes, and the Potential Corrosive Media and Mechanism

The presentation provided an overview of the different work relating to corrosion under three different temperature regimes. Various types of steel specimen were subjected to different environments simulating the oxyfuel combustion flue gas:

- Less than 200°C
- Greater than 200°C but less than 400°C
- Greater than 400°C

For temperature below 200°C, it was noted that:

- Corrosion was only observed when condensation occurred at low temperatures of 5°C.
- Even at low temperatures, SO₂ is oxidized to SO₃ to form H₂SO₄ in condensates; although equilibrium is not reached. Condensation is dependent on the dew point or the concentration of acid gas species and water in the flue gas.
- At ambient pressure and temperatures of 60°C and 170°C neither visible condensation nor corrosion occurred when different types of steel are subjected to 8,000ppm water and 1.8% O₂, 220ppm SO₂ and 1,000ppm NO₂. With no condensation, no corrosion occurred.

For temperature above 400°C, it was noted that:

- Current work (using steel with 12% Cr VM12) demonstrates that carburisation occurs at temperature above 500°C if CO₂ is the dominant gas constituent.
- The carburisation depth increases with decreasing O₂ fugacity.



• Up to 12% Cr, it was noted that water, O₂ and CO₂ influence the formation of the protective layer. It was observed that formation of the Fe/Cr-Spinel or Cr oxides (which consists of the protective layer) seems to be retarded under oxyfuel condition.

It was concluded that the understanding of the corrosion mechanisms during oxyfuel combustion would require the understanding of the role of water, diffusion of molecules and iron in the oxide region. Essentially, it is important to elucidate the transport processes in oxide in order to develop a predictive model. The following questions were raised during the presentation and require further clarification:

- Diffusion coefficient depends on vacancy density, vacancy mobility or other lattice defects,
- Vacancy density is a function of oxygen fugacity $(f = \phi \cdot p)$. How this can be influenced by an oxyfuel atmosphere?
- CO₂ permeability in oxides enhanced by water?
- Water typically enhances oxidation rates. How?
- What is the effect of SO₂/SO₃ in combination with high CO₂ partial pressure?
- Nucleation and growth of Cr containing phases, which decrease the corrosion rate.

4.2 Workshop Discussion and Highlights

After the keynote presentations there were 24 additional presentations subdivided into 6 different sessions. The following issues were discussed:

- Closure of sulphur balance of Oxyfuel Combustion
- SO₃ Measurements Techniques and Acid Dew Point
- Hg Measurement and Modelling
- SO₂, SO₃ and Hg Control
- Ash Deposition and Corrosion

4.2.1 Closure of Sulphur Balance of Oxyfuel Combustion

Due to regulatory requirements (i.e. reporting for Environmental Impact Assessment), the closure on the sulphur balance is important. From this workshop, it could be concluded that closure of the sulphur balance under oxyfuel combustion could be difficult to obtain. Nonetheless, it is still important to evaluate and understand the reasons for the discrepancy in the sulphur balance. Furthermore, this should also aid in the verification of the data gathered in any pilot testing.

Several presentations in the workshop [4-7] indicated that the typical discrepancy in the sulphur balance is between 10 to 35%; and on average, the discrepancy is less than 20%. This is in agreement to several earlier published works by IHI [8] and Chalmers University [9].

From this workshop, the following could be concluded:

- Discrepancy in the sulphur balance was generally higher during oxyfuel combustion,
- 'Missing' sulphur appears to be lost in the furnace and it could be observed that the degree of discrepancy is very specific to the test rig used. This could be due to manner on how ash is deposited within the system. A larger scale test rig (>10MW_t) tends to have a lower discrepancy when compared to a smaller test rig (< 1MW_t),
- It was agreed (albeit with high uncertainty) that 'missing' sulphur is likely due to the retention of the sulphur in the furnace surface, ash, and affected by the amount of SO₂/SO₃ recycled in the flue gas.
- The injection of absorbent for in-furnace SO₃ reduction tends to increase the discrepancy of the sulphur balance in both air and oxy combustion cases.



4.2.2 SO₃ Measurement Techniques and Acid Dew Point

Three different techniques on SO_3 measurements were discussed and described in various presentations:

- Controlled condensation method [4, 5, 10-12]
- Severn Science continuous analyser (using isopropanol solution) [11]
- FTIR optical probe (under development) [13]

The controlled condensation method is universally acceptable which involve the sampling of fixed volume of flue gas through a condensing unit held at constant temperature. However, this measurement technique is known to have a negative bias due to interference of the ash. Thus, it is recommended that careful control of temperature and efficient ash separation are needed to prevent any loss of SO_3 during gas sampling.

Spoorl and co-workers [10] recommended the use of quartz wool instead of glass wool for the separation of the ash during gas sampling. This is because quartz wool (being almost pure SiO₂) tends to be more inert thus reducing any side reactions with SO₃ or H_2SO_4 . The use of an inertial sampling probe as demonstrated in the work done by Kerney and co-workers [12] demonstrated better ash separation during gas sampling.

Severn Science continuous SO_3 analyser is based on the use of isopropanol to react with SO_3 to form sulphate ions. The solution is then passed through a porous bed of barium chloranilate which emits light in contact with sulphate ions. The amount of SO_3 can be estimated using a photometer. However, the use of isopropanol to absorb SO_3 is well known to be biased. In addition to the negative bias due to the ash separation, the use of isopropanol also provides a positive bias due to the solubility of SO_2 with water. Another issue of concern is the interference of the CO_2 gas with the isopropanol and SO_3 measurements under oxyfuel combustion conditions which is still to be evaluated.

The last method discussed during the workshop is based on the optical probe with FTIR analysis to measure SO_2 and SO_3 [13]. The current work revolves around resolving the spectra in order to separate the two signals (which overlap). The probe is still under development and will be tested at the DTU's 30kW oxyfuel test rig (in 2011) prior to being tested on a larger scale at Dong Energy's pilot facility.

Uncertainty regarding the determination of the acid dew temperature under oxyfuel combustion was discussed during the workshop. Spoorl and co-workers [10] noted that acid dew point correlation presented by Verhoff/Banchero's equation over predicts dew point temperature; whilst Okkes' equation under predicts dew point temperature under oxyfuel combustion condition and recommended the Zarenezhad equation.

Yan and co-workers [6] attempted the measurements of acid dew point temperature by using an air cooled probe which condenses H_2SO_4 onto the surface thus closing an electrical circuit to produce a signal. With this technique, significant interference due to the ash has been noted, making measurements very complicated.

4.2.3 Hg Measurement Techniques and Modelling

John Pavlish [2] in his keynote address presented some preliminary work on the evaluation of Hg sampling techniques for atmospheric CO_2 . He noted that CO_2 affected the mass flow rate of the continuous monitoring measurements (CMM) equipment. He recommended that measuring Hg under high CO_2 concentration requires calibration of the mass flow meter and is dependent on the type of CMMs.



Likwise, John Pavlish and co-workers [2] also noted the acid traps and $SnCl_2$ which are typically used in the wet chemistry method tends to be affected by higher concentration of CO₂, SO₂ and NOx under oxyfuel combustion conditions. They have recommended the dilution of the flue gas to obtain better measurements of total gaseous Hg.

Fry and co-workers [14] compared Hg behaviour across 2 pulverised coal furnaces (40kW & 1.5MW) and a CFB furnace (330kW). For Hg sampling they used the Inertial Flue Gas Probe (mentioned earlier) which absorbs Hg species on activated carbon. In most cases the oxyfuel conditions produce a higher native capture of Hg, which was considered to be due to the higher carbon in the ash. They also found that the CFB produced the highest capture rate but that Hg speciation did not vary across the apparatus' by any appreciable amount.

Gharebaghi and co-workers [15] presented work on Hg & SOx modelling in an oxyfuel environment. They found that Hg oxidation via heterogeneous mechanisms may be lower in oxyfuel, though the model under predicted experimental values. They concluded that there is little data available for model validation of Hg oxidation in oxyfuel.

Kiga and co-workers [16] of IHI presented experimental results indicating that a fabric filter removes mercury, but no units were given on the plots to quantify levels.

The workshop concluded the following on this area of development:

- Difficulties in the measurements of total Hg under oxyfuel combustion condition have been noted. More work is necessary to identify different biases when using the Ontario Hydro (OH) method as reference case. The results from OH method should be compared against other methods. Various CMM would require better calibration. Activated Carbon trap method might significantly be disadvantaged by higher concentrations of the acid gas species of oxyfuel combustion flue gas.
- Uncertainties and significant gaps exist with regard to the measurements of Hg²⁺ or Hg^p under oxyfuel combustion conditions. It was recommended that more fundamental study is required to determine the big difference between the OH and CMM methods.

4.2.4 SO₂, SO₃ and Hg Control

Due to the impact of SOx on corrosion of steel in various parts of the oxyfuel combustion system, it is considered essential to evaluate the different options to reduce SOx from the flue gas. The main question is on where and to what extent SOx should be removed prior to the CO_2 processing unit or what level of SOx in the recycled flue gas should be tolerable to the boiler.

Fundamental work in understanding the fate of SO_2 and SO_3 during oxyfuel combustion in large burner scale (i.e. > 15MWt) have been demonstrated in the work done by Alstom [12], Doosan Babcock [7] and Vattenfall [6] presented during the workshop. The implications of the amount of sulphur recycled in the flue gas that could have an impact on the amount of the SO_2 and SO_3 in the flue gas exiting from the boiler have been presented.

In this workshop, different SOx and Hg control technologies were presented.

• Specific work on the possible performance of the FGD under oxyfuel combustion has been evaluated by Hansen and co-workers [17]. They noted that an increase in CO₂ under oxyfuel combustion results to an increase in residual CaCO₃ but better removal efficiency of SOx. They also noted the impact of the pH due to higher concentration of the acidic components. They concluded that the slurry between having a pH in the range of 5.2 - 5.5 caused a critical change in the dissolution of limestone. These results could correlate well with the results presented by Yan and co-workers [6] showing FGD sulphur capture slightly decreased during oxyfuel firing.



- Dhungel and co-workers [4] presented results on sorbent injection in-furnace and at the exit of the furnace which was mainly focussed on SO₃ control. They found that the sorbent injected post-combustion performed better in regards to SO₃ removal. They also showed that the sorbents performance was not affected by the high CO₂/H₂O concentration. This technique provides a good opportunity to represent a step forward in terms of assuaging the downstream corrosive effects of an oxyfuel environment.
- Hitachi-Babcock [18] presented a novel process illustrated on Figure 3 for controlling Hg and SO_3 , with the addition of a novel catalyst in the SCR and by cooling the flue gas down to 90° C using finned cooling tubes. The novel catalyst (Triple Action Catalyst, TRAC©) has been designed in order to selectively oxidise Hg without oxidising SO₃. The flue gas then passes through a finned tube gas cooler. The concept was to deliberately cool the gas below the acid dew point thereby allowing the acidic mist to be condensed on the fly ash and ash deposits on the tubes. The benefits appear to be that the H₂SO₄ is condensed on the fly ash without competing for the Hg absorption sites, allowing better removal efficiency (though this mechanism is theorised by these authors, rather than proved the presenters).

4.2.5 Ash Deposition and Corrosion

Metal loss associated with sulphur species under oxyfuel combustion conditions would require clarification especially when subjected to temperatures greater than 500°C.

Presentations in the workshop on corrosion and ash deposition [3, 19-24] demonstrated two important mechanisms occurring under oxyfuel conditions. Carburisation of Fe and Cr based alloys is expected to occur due to the higher concentration of CO₂. The expected effects are that it reduces the protective oxide layer that prevents further corrosion in Fe, Cr or Fe/Cr alloys and can reduce the creep strength and welds. The second is sulphatisation, mainly of Fe oxides which are typically associated with corrosion in the water wall and superheaters. Evidence was presented that the corrosion rate increased under high S/high Cl coal firing [20], however, other evidence was presented that showed carburisation corrosion being inhibited by the presence of SOx.

Fry and co-workers [24] evaluated the corrosion potential using an "Electrochemical Noise (EN)" measurement device. It was demonstrated that oxy-fuel conditions produce a greater corrosion potential and that switching from reducing to oxidising produced a spike in signal, particularly in the lower alloyed steels.

5. Concluding Remarks

Below highlights some of the key learning points during the workshop.

- [1] Discussion about the reliability of SO_3 measurements and previously published data on this area of work should be reviewed. It is essential that all SO_3 measurements and published should also include temperature data to be able to established a more consistent database.
- [2] Measurements of SO₃ using "Controlled Condensation Technique" and "Isopropanol" Technique are all acceptable methods that could be used during oxyfuel combustion. However, care should be taken in the control of temperature and ash filtration during any experimental work. Furthermore, biased with the use of the Isopropanol technique should require more evaluation when operating under oxyfuel combustion condition.
- [3] Continuous measurements techniques in a real power plant setting should be develop to address the needs of oxyfuel combustion power plant to monitor SO_3 emissions in all critical areas of operation (i.e. recycled flue gas duct, ESP, etc...).



- [4] Sulphur balance under oxyfuel combustion condition would still require more investigation to enable better understanding of the fate of sulphur during oxyfuel combustion operation.
- [5] Various control for SO_2 and SO_3 has been reported. One of the interesting development presented during the workshop was the reduction of SO_2 and SO_3 by using in-furnace absorption injection which should provide opportunity to control SO_2 and SO_3 without the use of external FGD as an option.
- [6] It was established in the meeting regarding the error caused by the high concentration of CO_2 and acidic components toward measurements of total Hg and its speciation. It is important that work should be pursued in this area to re-validate all Hg measurement techniques used in air fired system, if this could be used under oxyfuel combustion condition. This put into question the reliability of Hg measurements data presented in various oxyfuel combustion experimental campaigns. This therefore requires Hg measurements data to be reviewed. This only re-emphasised that more work should be done to validate the measurement techniques to allow a better understanding of the fate of Hg during oxyfuel combustion operation.
- [7] There is a lot more questions to be answered regarding the understanding of corrosion mechanisms during oxyfuel combustion. Several work in this area is focused on evaluating metal losses for temperature greater than 500°C and they are still on-going. It was recommended during the workshop that a "Working Group" should be organised to review, evaluate and discuss in more details all the data collected during oxyfuel combustion experimental campaign.
- [8] Work on corrosion mechanism between 100°C and 300°C (especially between 150-250°C regions) under oxyfuel combustion condition are very limited. It was recommended that work in this area should be pursued to address this issue and prevent any surprises given that concentration of acidic gases in the flue gas of oxyfuel combustion is much higher as compared to any air fired condition.
- [9] Ash deposition and its interaction with acidic gases on corrosion is another area where more evaluation is needed to support any fundamental work on mechanism of corrosion under oxyfuel combustion conditions. It was also highlighted during the workshop regarding the higher capture rate of sulphur in the ash which consequently could lead to a more significant role of the sulphur species to be involved in the corrosion mechanism during oxyfuel combustion. Only way to elucidate this interaction requires the understanding of the different ash deposition mechanism during oxyfuel combustion.

6. Acknowledgements

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Appendix 1 - List of Participants

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