

## First year operation experience with a 1 t/h CO<sub>2</sub> absorption pilot plant at Esbjerg coal-fired power plant

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### Abstract

As a part of the EU integrated project CASTOR a 1 t/h CO<sub>2</sub> absorption pilot plant has been erected at the 400 MW coal-fired Esbjerg power plant. The main purpose of the pilot plant is to demonstrate the post combustion capture technology in conjunction with a coal-fired power plant. Additionally, the pilot plant will be used to test the performance of new energy efficient solvents and to verify modelling work. The design of the pilot plant is based on a commercial CO<sub>2</sub> production plant using alkanolamine as the absorbent. The pilot plant operates on a slipstream of the flue gas taken after the deNO<sub>x</sub> and desulphurisation plants. During the pilot plant's first year, two 1000 hours test campaigns using monoethanolamine were conducted. Among others, the campaigns included tests to verify plant functionality and performance as well as a more detailed parametric study. This paper summarises the initial operation experience and some of the first results obtained during these campaigns.

Keywords: CO<sub>2</sub> absorption, clean coal combustion, post combustion capture, pilot plant

### 1. Introduction

CASTOR is an integrated project under the Sixth Framework programme under Sustainable Energy Systems, "Capture and sequestration of CO<sub>2</sub>, associated with cleaner fossil fuel plants" under the European Commission. One of the key targets of CASTOR (See homepage: [www.co2castor.com](http://www.co2castor.com)) is to reduce the costs of post-combustion capture by development of an absorption solvent with lower energy requirement and optimise the integration of a CO<sub>2</sub> absorption plant into a modern power plant. A major element of the CASTOR project is to construct and operate a CO<sub>2</sub> absorption pilot plant that operates on coal derived flue gas. The purpose of the pilot plant is to test novel solvents developed in the CASTOR project and to gain hands on experience with the CO<sub>2</sub> capture technology on real coal flue gas.

Furthermore, the pilot plant will be used to validate the modelling and laboratory results obtained by the different CASTOR partners. Within the framework of the CASTOR project, DONG Energy and Vattenfall Nordic have carried out the tasks of engineering, purchasing, installation and commissioning of a CO<sub>2</sub> absorption pilot plant as well as planning and conducting tests at the facility.

The CO<sub>2</sub> absorption pilot plant is placed at the Esbjerg power station, Esbjergværket (ESV). ESV is owned and operated by DONG Energy Generation and is located at the West coast of Denmark. ESV is a 400 MW pulverised coal-fired power plant equipped with deNO<sub>x</sub> (high dust SCR), electrostatic precipitator and desulphurisation unit (wet limestone scrubber). The pilot plant operates on a slipstream of the flue gas, taken downstream the desulphurisation unit. In late 2005, the CO<sub>2</sub> absorption pilot plant was erected and commissioned. During the pilot plant's first year of operation, two 1000 hours test campaigns were conducted using a conventional solvent, 30 %-weight monoethanolamine (MEA) solution. In this paper selected test results from the two MEA campaigns are presented.

## **2. CO<sub>2</sub> absorption pilot plant at ESV**

The design of the pilot plant is based on the design of a standard amine-based CO<sub>2</sub> production plant with minor modifications. A flow diagram of the pilot plant is shown in Figure 1.

A slipstream of the flue gas from ESV is taken at a position immediately after the SO<sub>2</sub> scrubber. The flue gas does not undergo any pre-treatment before supplied to the CO<sub>2</sub> absorption plant. The flue gas enters the absorber tower at the bottom in a counter-current flow with the solvent. The fan is placed downstream the absorber, which implies that the absorber is operated at a pressure slightly below atmospheric pressure. The CO<sub>2</sub> content of the absorber in- and outlet is continuously monitored by CO<sub>2</sub> analysers.

The absorber tower consists of four consecutive packed-beds for CO<sub>2</sub> absorption and an additional bed for water wash at the top. The absorber has an internal diameter of 1.1 meter. Each bed for CO<sub>2</sub> absorption is 4.25 meters in height and filled with IMTP50 random packing. The bed for water wash is 3.0 meters in height and filled with Mellapak 250Y structured packing. At the top of each bed there is installed a liquid distributor plate to ensure an even liquid flow profile. The lean solvent can be fed to each of the four beds individually or simultaneously, however, for the tests presented in this paper, the entire solvent has been fed to the top bed. The water wash functions as a closed loop. The wash water from the bed is collected on a tray below the bed and is cooled in a water-cooler before returned to the wash section. Make up water is added to the wash section, in order to control the amine build-up. The overflow from the wash section runs into the absorber.

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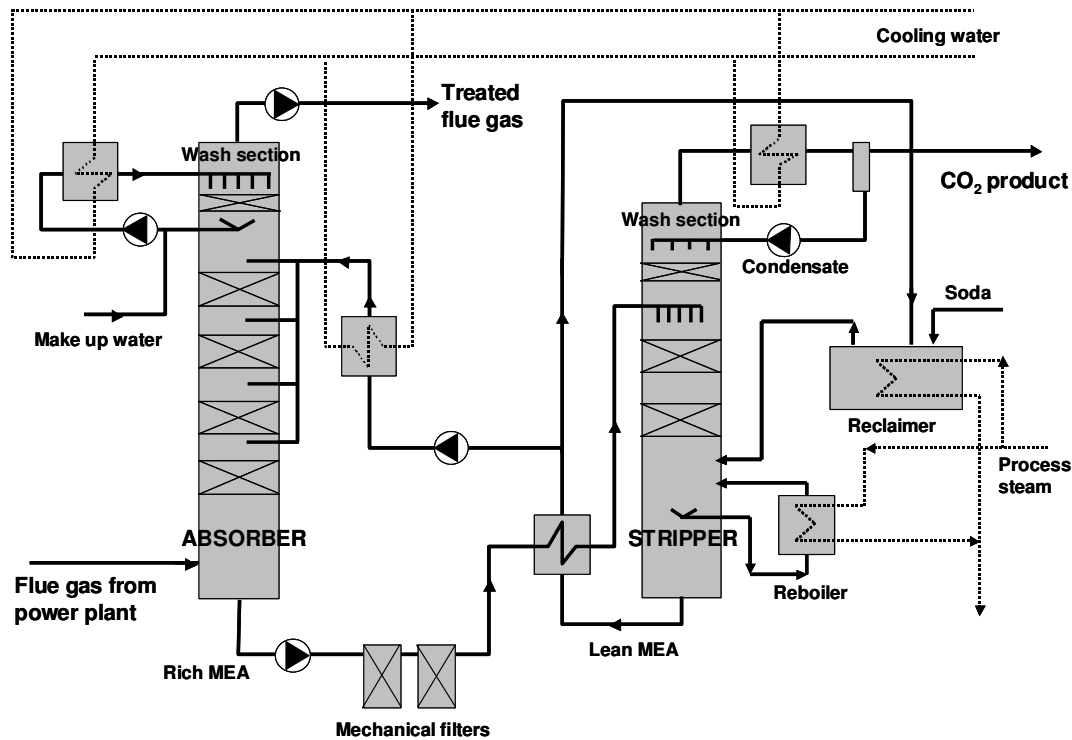


Figure 1: Flow diagram of the CASTOR pilot plant at the Esbjerg power plant

The rich solvent from the absorber is pumped through two mechanical filters in series and a plate heat exchanger (heat exchanged with lean solvent from the stripper) before fed to the stripper. The stripper has an internal diameter of 1.1 meter and consists of two 5.0 meters in height beds filled with random packing IMTP50 and an additional bed for water wash at the top (3.0 meters of random packing IMPT50) where the internal diameter is reduced to 0.8 meter. The stripper pressure is controlled by an overhead regulation valve. A steam driven reboiler supplies the heat input to the stripper. The steam (2.5 bar(g) saturated) is supplied by ESV and the reboiler temperature controls the steam flow. The CO<sub>2</sub> gas and vapours from the stripper pass through a water-cooled condenser and a gas/liquid separator. The condensate from the separator is returned to the stripper wash section and the resultant gas, which essentially is pure CO<sub>2</sub> saturated with water, is returned to the ESV flue gas duct. The CO<sub>2</sub> product quality is monitored online by an analyser. The regenerated solvent from the stripper is cooled to its final set point temperature by a water-cooler after it has been heat exchanged with the rich solvent. A slipstream of approximately 10% of the solvent flow is passed through a carbon filter in order to remove organic degradation products.

Also part of the pilot plant is a reclaimer vessel where the impurities can be concentrated and re-moved from the solvent. The reclaimer works as a simple batch distillation unit – water and amine are boiled off whereas higher molecular weight organic degradation products, inorganic salts, and particulates (termed heat stable salts) are largely retained in the bottom product. Sodium hydroxide is added to the reclaimer in order to ease the evaporation of amine and liberate amine that has been

captured by acidic compounds. The reclaiming is driven by up to 6 barg saturated steam from ESV.

In order to monitor energy and cooling water consumptions as well as the general plant performance, the pilot plant is fitted with temperature sensors, pressure gauges and flow meters through out. All of the measurements are continuously registered by a data acquisition system and logged on a PC.

The key design and performance figures for the pilot plant are shown in Table 1. The flue gas purity criteria were defined in order to avoid excessive solvent degradation or other adverse impacts on the pilot plant operation, e.g. foaming and fouling.

Parameter	Design value
Flue gas capacity	5000 Nm <sup>3</sup> /h $\approx$ 0.5% of ESV flue gas flow
CO <sub>2</sub> production (at 12 % CO <sub>2</sub> )	1000 kg/h
Absorption degree	90%
Max solvent flow	40 m <sup>3</sup> /h
Max reboiler steam flow	2500 kg/h (2.5 bar(g))
Max stripper pressure	2 bar(g)
Flue gas conditions	47°C (sat.), <10 ppm SO <sub>2</sub> , <65 ppm NO <sub>x</sub> , <10 mg/Nm <sup>3</sup> dust (wet basis, actual O <sub>2</sub> )

Table 1: CO<sub>2</sub> absorption plant design specifications.

During the test campaigns, typical solvent analyses have been performed at the laboratory at ESV. The amine concentration was determined by a standard titration procedure. The CO<sub>2</sub> loading was determined by the barium chloride method, Ma'mun et al. (2005). A number for the total acid equivalents, i.e., heat stable salts, was determined by cation-exchange and subsequent titration. For selected samples, the sulphur and iron contents were determined by elemental analysis.

### 3. Operation experience and results

#### 3.1. CASTOR test programme

Within the time frame of the CASTOR project, the activities at the pilot plant have been divided into four major test campaigns:

1. 1000 hours operation on a reference solvent: 30% MEA (completed)
2. 1000 hours operation on a reference solvent: 30% MEA (duplicate of campaign 1, completed)
3. 1000 hours operation on a "new" solvent: CASTOR 1 (2007)
4. 1000 hours operation on another "new" solvent: CASTOR 2 (2007)

The first test campaign, 1000 hours operation on 30% MEA, was conducted in January through March 2006, Knudsen et al. (2006). MEA was chosen as the reference solvent because of its well-known performance and the general availability of kinetic and thermodynamic data for simulations. During the first test campaign it was discovered that some of the vital measurement devices were not working properly. Additionally, the pilot plant was initially operated at conditions recommended by the supplier, which proved not to be optimal with respect to CO<sub>2</sub> capture from power plants. Thus, it was decided to conduct a second test campaign using 30% MEA to improve the data quality and the overall process performance.

The second MEA campaign was conducted from mid December 2006 to early February 2007. Each of the two MEA campaigns consisted of a series of parameter variation tests as well as a longer period (500 hours) of continuous operation. The objective of the parameter variation test was to locate the optimum operating conditions for MEA with respect to energy consumption and to generate various different data points for model validation. The objective of the 500 hours test was to gain information on the behaviour and performance of the CO<sub>2</sub> absorption plant in the longer term.

### *3.2. Results – Optimisation of solvent flow rate*

In this test, the idea was to locate the optimum solvent flow rate at 90% CO<sub>2</sub> removal. The test was conducted at a fixed stripper pressure of 1.85 bar(a) and at the nominal flue gas flow ( $\approx 5000 \text{ Nm}^3/\text{h}$ ). Measurements were conducted at five different solvent flow rates in the interval of 12.5 to 23 m<sup>3</sup>/h. At a given solvent flow rate, the plant was tuned to a CO<sub>2</sub> recovery of 90% by adjusting the reboiler temperature.

In Figure 2, the specific steam demand and the CO<sub>2</sub> recovery have been plotted as a function of the solvent flow rate. It appears from Figure 2 that the CO<sub>2</sub> recovery has been close to 90% in all of the tests. In addition, Figure 2 indicates that the lowest specific steam demand, 3.6 GJ/ton, was measured at 14.8 m<sup>3</sup>/h. However, it should be noticed that taking the experimental uncertainty into account, the specific steam demand has been nearly constant in the flow range of 12.5 to 19.0 m<sup>3</sup>/h. At the higher flow rate (23 m<sup>3</sup>/h), the specific steam demand is clearly seen to increase and at lower flow rates it was barely possible to reach the specified 90% recovery even though the maximum steam input was applied to the reboiler. Altogether, the solvent flow optimisation test indicates that the optimum flow range is quite broad for MEA at the Esbjerg pilot plant.

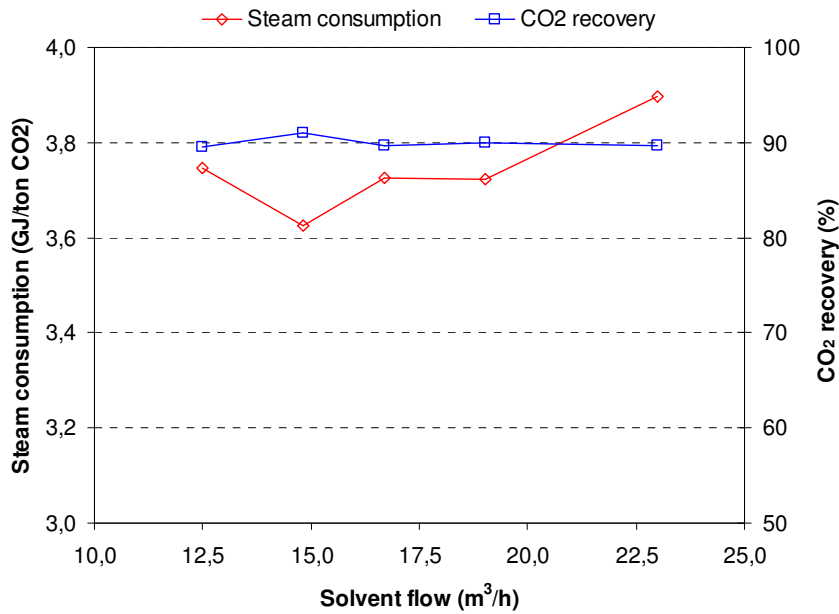


Figure 2: Specific steam consumption and CO<sub>2</sub> recovery as a function solvent flow

CO<sub>2</sub> mass balances are shown in Figure 3. It is seen that there is relatively good agreement between the CO<sub>2</sub> uptake in the absorber and the CO<sub>2</sub> release from the stripper based on gas side measurements. The CO<sub>2</sub> uptake based on solvent analyses are seen to be systematically lower than either of the gas side measurements. This is presumably related to minor offsets in the calibration of the liquid flow meters.

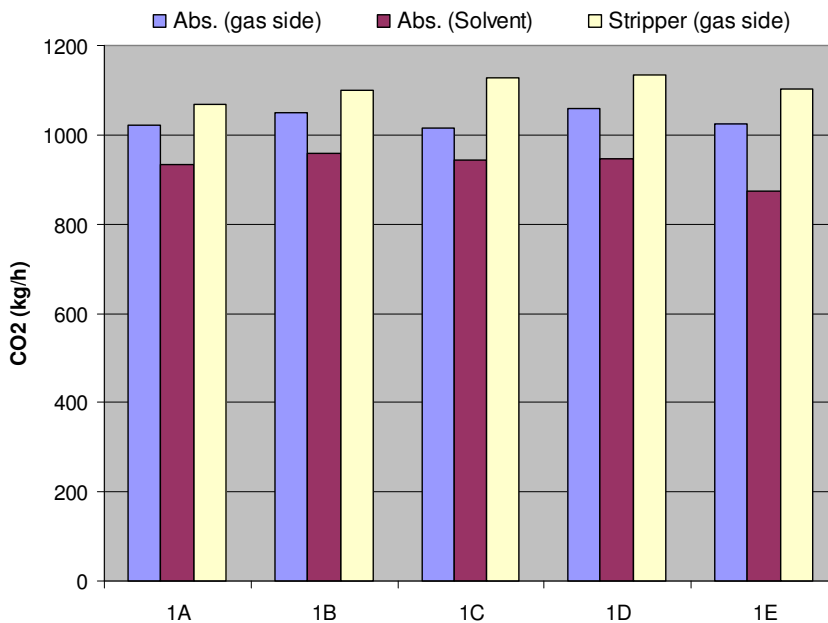


Figure 3: CO<sub>2</sub> mass balances – Solvent flow optimization test

Energy balances are shown in Table 2. It appears that the energy balances close within 0-10%. This confirms that the values estimated for the reboiler steam consumption are reliable. Table 2 furthermore indicates that little heat has been transferred from the system to the flue gas, i.e. almost no net heating or cooling of the flue gas in the absorber. This is a consequence of the objective of operating the plant at close to neutral water balance.

Parameter	Unit	1A	1B	1C	1D	1E
Solvent flow rate	m <sup>3</sup> /h	23.0	19.0	16.7	14.8	12.5
Steam consumption	GJ/ton CO <sub>2</sub>	3.9	3.7	3.7	3.6	3.7
Cooling water	GJ/ton CO <sub>2</sub>	3.9	3.6	3.4	3.2	3.2
Heat from flue gas	GJ/ton CO <sub>2</sub>	-0.01	-0.04	-0.08	-0.08	-0.09
Total heat loss	GJ/ton CO <sub>2</sub>	0.02	0.12	0.24	0.31	0.41
Total heat loss	% of input	0	3	6	8	10

Table 2: Energy balances – Solvent flow optimization test

In addition, it appears from Table 2 that the heat loss, i.e. closure on the energy balance, increases as the solvent flow is reduced. This is presumably related to the fact that the cooling-water flow meters under predict the actual flow rate at low flow rates, because they operate in the low range of their scale.

### 3.4 500 hours test

The objective of 500 hours test was to operate the pilot plant continuously for 500 hours at the optimised conditions and achieving 90% CO<sub>2</sub> removal on average. Corrosion coupons were installed at various positions in the plant in order to quantify the corrosion rates of selected steel materials. The corrosion investigation was conducted by one of the CASTOR partners, Kittel et al. (2006). In addition, it was the aim to gain information on solvent consumption and build-up of degradation products and to gain experience on the overall process stability.

Based on the results from the parameter variation tests, the optimised conditions were defined as:

- Flue gas flow: 5000 Nm<sup>3</sup>/h (wet)
- Solvent flow: 15.5 m<sup>3</sup>/h
- Stripper pressure: 1.85 bar(a)
- Reboiler temperature: 120.5 °C
- Plant operated at neutral water balance

The 500 hours test lasted from January 15<sup>th</sup> to February 7<sup>th</sup> 2007, a total of 550 running hours. During the test period, no incidents or malfunctions occurred at the CO<sub>2</sub> pilot plant, which resulted in shut down, however, at the Esbjerg power station two incidents occurred which triggered a momentary shut down of the pilot plant with a total outage of 3 ½ hours.

In Table 3, the average, minimum and maximum values of a series of key parameters obtained during the test are shown.

Parameter	Unit	Average	Min*	Max*
Solvent flow	m <sup>3</sup> /h	15.5	15.5	16.8
Flue gas flow	Nm <sup>3</sup> /h, wet	5020	4870	5230
Reboiler temperature	°C	120.4	119.1	120.6
Stripper pressure	bar(a)	1.89	1.87	1.94
CO <sub>2</sub> content flue gas	%-vol. (dry)	12.4	9.3	13.9
CO <sub>2</sub> recovery	%	88	77	97
CO <sub>2</sub> production	kg/h	1000	850	1070
Steam consumption	GJ/ton CO <sub>2</sub>	3.7	3.6	5.4
Cooling water	GJ/ton CO <sub>2</sub>	3.4	2.8	4.3
Total heat loss	% of input	6	0	16
Water accumulation	kg/h	19	-60	115

Table 3: Summary of results Test 2 (550 hours average values). \* Excluding the two outages

It appears from Table 3 that the average performance achieved by the CO<sub>2</sub> absorption plant during the test has been very close to the target parameters as listed above. On average, a CO<sub>2</sub> removal efficiency of 88 % was achieved. This is 2 %-points below the target of 90% removal. The main reason for the deviation is that there is no active control loop, which controls the CO<sub>2</sub> capture percentage. Therefore it is practically impossible to keep a fixed CO<sub>2</sub> removal degree over an extended period of time as fluctuations in the flue gas composition, i.e. CO<sub>2</sub> concentration, will affect the CO<sub>2</sub> capture percentage. The CO<sub>2</sub> concentration of the feed gas changed quite frequently during the test campaign as a response to load changes at the power plant.

Table 3 furthermore indicates that the pilot plant was operated at a near neutral water balance. This was achieved by maintaining the effluent gas from the absorber approximately 1°C above the flue gas inlet temperature by the means of the wash water cooler. The average CO<sub>2</sub> production (average of absorber and stripper measurement) during the test has been very close to the nominal capacity (1 t/h) of the plant.

In Figure 4, the history of the flue gas flow to the pilot, the CO<sub>2</sub> recovery and the specific steam demand are shown for the entire duration of the 550 hours test. It appears that, except for the two outages the 23<sup>rd</sup> and 25<sup>th</sup> of Jan. the pilot plant has been in continuous and stable operation. The CO<sub>2</sub> recovery typically fluctuates between 85 and 95% throughout the period.

The average steam demand for solvent regeneration during the 550 hours was approximately 3.7 GJ/ton CO<sub>2</sub> (Table 3). The dynamic energy consumption is shown in Figure 4. It appears from Figure 4 that the steam demand has been relatively constant around 3.7 GJ/ton CO<sub>2</sub> with some narrow peaks reaching 4.2-4.3 GJ/ton. The



peaks appear to be related to a sharp increase in the CO<sub>2</sub> recovery. In fact, the peaks are caused by the sudden decrease in CO<sub>2</sub> production when the CO<sub>2</sub> concentration in the flue gas decreases, whereas the absolute heat input to the reboiler remains unchanged.

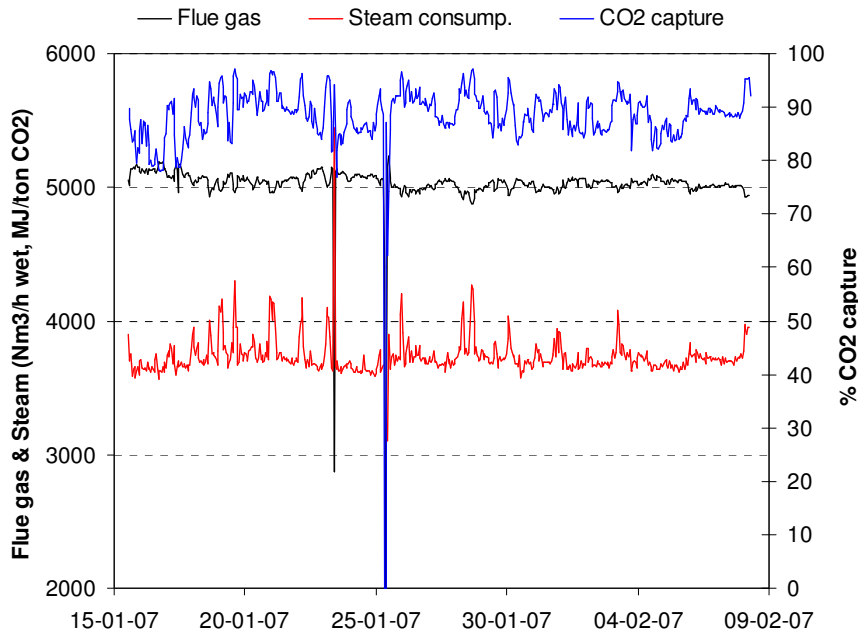


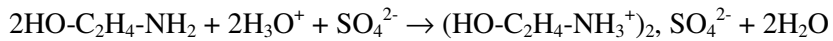
Figure 4: Pilot plant performance during 500 hours test

### 3.3. Solvent consumption and impact of flue gas pollutants

The amine consumption is an important factor in evaluating the operating costs of post combustion capture. In a CO<sub>2</sub> capture plant amine is lost because of slip, chemical degradation and reactions with pollutants, e.g. acidic compounds from the flue gas.

In the case with MEA, oxidative degradation and carbamate polymerization are very significant causes of MEA consumption, Goff and Rochelle (2004). The complex degradation mechanisms of MEA have been investigated in several publications, Goff and Rochelle (2004), Bello and Idem (2005). However, the relative importance of the different degradation mechanisms is still not completely clear. Among the more important degradation products of MEA are carboxylic acids and aldehydes. The degradation of MEA is also reported, Goff and Rochelle (2004), to be catalysed by the presence of certain metallic elements, e.g. V, Fe and Cu. These species may enter the solvent due to the presence of corrosion products, fly ash, and corrosion inhibitors.

SO<sub>2</sub>, NO<sub>2</sub>, and carboxylic acids react with MEA in an acid-base reaction forming heat stable salts (HSS) as illustrated in the reaction below. NO does not react with MEA, Chapel et al. (1999).



The MEA associated in heat stable salts may partly be regenerated in the reclaimer as the above reaction may be reversed upon addition of caustic soda. However, sulphur and nitrogen may also be incorporated in organic compounds.

During the 500 hours tests, the concentration of MEA was found to gradually decrease as results of solvent degradation and emission losses. The MEA consumption during the two 500 hours tests have been estimated by keeping track of the make-up MEA added to the plant in order to maintain the concentration at 30 %-weight. In the 500 hours test of the first MEA campaign, the specific MEA consumption was estimated to approximately 2.4 kg/ton CO<sub>2</sub> removed. In the second MEA campaign, a significantly lower MEA consumption was found, 1.4 kg/ton CO<sub>2</sub>. The reasons for the lower MEA consumption in the second campaign is not completely clear, however, the NO<sub>x</sub> and SO<sub>2</sub> concentrations in the flue gas was significantly lower during the second campaign.

For CO<sub>2</sub> recovery from gaseous (No SO<sub>2</sub>) fuels, the MEA consumption rate has been reported to 1.6 kg/ton CO<sub>2</sub>, Chapel et al. (1999). This value is within the range (1.4-2.4 kg/ton CO<sub>2</sub>) found in this work.

In Figure 5, the accumulation of total sulphur, iron and heat stable salts in the solvent during the second MEA campaign is shown. As no reclaiming was performed in this period, all impurities trapped in the solvent were accumulating. The sulphur content in the solvent originates from absorption of the few ppm SO<sub>2</sub> that is present in the flue gas, whereas iron originates from dissolved corrosion products.

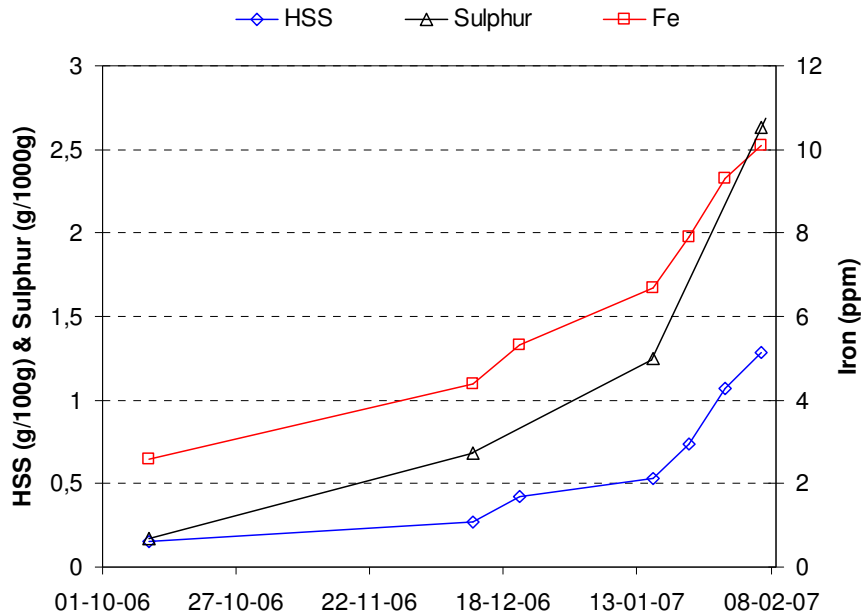


Figure 5. Accumulation of heat stable salts, iron and sulphur in the solvent during the 2<sup>nd</sup> MEA campaign

It appears from Figure 5 that, the accumulation rates of impurities were higher after mid-January. This is related to the fact that the plant was not operated continuously in the period October through December, hence less degradation, corrosion and absorption of SO<sub>2</sub> occurred. Furthermore, it is seen in Figure 5 that in the period from January 15<sup>th</sup> to February 7<sup>th</sup> (500 hours test), the HSS accumulation rate has been close to constant. Considering this period, the sulphur concentration in the solvent increases from 1.25 to 2.63 g/1000 g, which is equivalent to approximately 18 kg of total sulphur. In the similar period, the average SO<sub>2</sub> concentration in the ingoing flue gas was 5.6 ppm, which corresponds to a total sulphur input with the flue gas of 21 kg. This illustrates that the solvent absorbs nearly all ingoing SO<sub>2</sub>.

#### 4. Conclusion

A 1 t/h CO<sub>2</sub> absorption pilot plant at the coal-fired Esbjerg power station has been erected and commissioned. The first year of operation shows that it is possible to maintain stable and reliable operation of the plant and to operate the plant at neutral water balance. The steam requirement for the MEA process was found to be ≈3.7 GJ/ton CO<sub>2</sub> and the MEA consumption 1.4-2.4 kg/ton CO<sub>2</sub>. In the coming years, the pilot plant will be used to gain more experience on post combustion capture and to evaluate the performance of novel solvents developed in the CASTOR project.

#### 5. Acknowledgement

The pilot plant at ESV is sponsored by the CASTOR partners and the European Commission through the CASTOR project. The authors would like to express their gratitude to all of the sponsors and the staff at ESV.

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