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Performance of an amine-based CO₂ capture pilot plant at the Fortum Oslo Varme Waste to Energy plant in Oslo, Norway

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ABSTRACT

Fortum Oslo Varme AS (FOV) decided (in July 2018) to build a 1:350 scale pilot plant to demonstrate that the selected Shell's CANSOLV capture technology using solvent DC-103 is suitable for cleaning CO₂ from the exhaust gases of their waste to energy (WtE) plant at Klemetsrud in Oslo, Norway. This paper presents the most important measurement data and results from a pilot plant testing campaign from March to December 2019.

The project stated requirements for the successful completion of the first pilot plant campaign were at least 2000 operational hours and total amine emissions lower than 0.4 ppmv on average over the last 500 h of testing. Emphasis is on the online emission measurement results both with and without an aerosol emissions mitigation system located downstream of the capture unit. The impact of perturbations in the plant operating conditions and inlet gas quality is discussed, in particular the particulates content.

The pilot plant campaign achieved its primary purpose of demonstrating low amine emissions. It also produced important data on absorbent degradation in actual operation on WtE flue gas while confirming other performance parameters such as CO₂ capture efficiency, steam consumption and CO₂ product purity. Thus, it can be concluded that the all requirements according to objectives of the pilot campaign were successfully met.

1. Introduction

This paper gives an overview of all the most significant results obtained during the Fortum Oslo Varme's (FOV's) carbon capture (CC) pilot plant operation with Shell's proprietary amine based solvent DC-103 in 2019. The location of the pilot plant was adjacent to FOV's waste to energy (WtE) plant in Klemetsrud (Oslo, Norway).

As part of the Norwegian government's carbon capture and storage (CCS) strategy, several CO₂ point sources in Norway have been evaluated as suitable candidates for large scale CO₂ capture facilities ([Gassnova webpage, 2020](https://www.gassnova.no)). In 2015, Fortum Oslo Varme's (FOV's) waste to energy (WtE) plant in Klemetsrud (Oslo, Norway) was selected as a potential receiver of government funding to build and operate a CO₂ capture (CC) unit to capture in excess of 400,000 t/y of CO₂. The

Abbreviations: ACF, Activated carbon filter; Am1, Am2 etc., DC-103 solvent component identifiers; AMD, Amine emission mitigation device; CC, Carbon (dioxide) capture; CCS, Carbon (dioxide) capture and storage; DC-103, Shell's proprietary amine-based solvent used in the CANSOLV process; Deg1, Deg2 etc., DC-103 solvent degradation product identifiers; ESP, Electrostatic precipitator; FG, Flue gas; FOV, Fortum Oslo Varme; FTIR, Fourier-transform infrared spectroscopy; IC, Ion chromatography; ICP-MS, Inductively coupled plasma mass spectrometry; K1, K2, K3, Klemetsrud WtE plant incineration line identifiers; LCMS, Liquid chromatography - mass spectrometry; L/G, Liquid to gas ratio; LOQ, Level of quantification; MVR, Mechanical vapour recompression; NIPH, Norwegian Institute of Public Health; OC, Organic compound; PFD, Process flow diagram; PM, Particulate matter; PSD, Particle size distribution; PTR-TOF-MS, Proton transfer reaction - time of flight - mass spectrometry; SCR, Selective catalytic reduction; SF, Stripping factor; TRU, Thermal reclaimer unit; UiO, University of Oslo; WtE, Waste to energy.

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captured CO₂ will be contained and liquefied and transported by ship to the Northern Lights facility (Northern Lights webpage, 2020) for sequestration under the Norwegian off-shore seabed. Additional (up-to-date) information about the Norwegian CCS projects can be found on the referenced Gassnova webpage, including a quality (and cost) assurance report by Atkins and Oslo Economics, published June 25, 2020 (Atkins report, 2020)

FOV evaluated several different CO₂ capture (CC) technologies and selected Shell's CANSOLV amine-based technology as the capture technology for the Klemetsrud project, with TechnipFMC as the engineering partner to deliver the plant. Shell's proprietary DC-103 solvent used in the CANSOLV system has been in large-scale commercial use at SaskPower's coal fired power plant in Boundary Dam since 2014 (St phenne, 2014). However, DC-103 has not been previously tested on WtE flue gases and it is anticipated (based on Norwegian Institute of Public Health (NIPH) recommendations (NIPH, 2011)) that the restrictions on emissions to air will be more stringent than previously encountered in other projects (compared to e.g. 6 ppmv on a 24-h basis at The CO₂ Technology Centre Mongstad (Maree et al., 2013)). Therefore, FOV decided (in July 2018) to build a 1:350 scale pilot plant to demonstrate that the selected CANSOLV capture technology is suitable for cleaning CO₂ from the exhaust gases of the Klemetsrud WtE plant, and in particular to show that the emissions of amines and solvent degradation products to air are within the set requirements.

The duration for a successful demonstration of the first pilot campaign were decided to be at least 2000 operational hours, with total amine emissions lower than 0.4 ppmv on average over the last 500 h of testing (of the first test campaign). All emissions to air were continuously monitored, including solvent degradation products. TechnipFMC delivered and commissioned the pilot plant in February 2019, and the requirements were successfully met with the plant in operation for more than 5000 h with the DC-103 solvent, from March to December 2019.

The pilot plant is a scaled down version of a future full-scale plant (> 400,000 t CO₂/year), in terms of process parameters such as L/G, gas velocity, etc., operating on a mixture of flue gas (FG) from three separate incineration lines from the existing Klemetsrud WtE plant. The pilot plant was designed and built to replicate the process conditions in the full-scale plant as well as possible.

2. Description of the waste to energy plant and the carbon capture pilot plant

2.1. Carbon capture pilot plant overview

In July 2018 it was decided to build a CO₂ capture pilot plant at

Table 1
List of participants to the FOV Klemetsrud pilot plant project (FOV FEED report, 2020).

Participant Role	Participant Role
FOV	FOV Project owner. Integration with the existing WtE plant
TechnipFMC	TechnipFMC Pilot plant supplier
Kanf Ingenuity Process AS	Subsupplier to TechnipFMC. Engineering, fabrication and supply of the pilot plant
Sl�ttland Mek. Industri AS	Pilot plant fabricator
Shell Catalysts & Technologies	Technology Licensor
University of Oslo	Flue gas analysis contractor
Ramboll Finland Oy	Analysis contractor
Eurofins Environment Testing Finland Oy	Offsite analysis laboratory
Airborne Labs International, Inc.	Offsite analysis laboratory
TCM (Technology Centre Mongstad)	Advisory services
DNV GL	Technology qualification, advisory services

Klemetsrud (Oslo, Norway) for technology qualification purposes. The pilot plant was ordered from TechnipFMC, and Table 1 provides an overview of the project participants.

The pilot plant was designed to represent the planned Klemetsrud full-scale CC plant as well as possible. Key process numbers are presented in Table 2.

The pilot plant was designed to be operated 24/7 on all possible variations of incoming flue gas within the limits of the WtE plant emission permit. A process flow diagram (PFD) of the pilot plant (with sampling points indicated) can be seen in Fig. 1. A description of the sampling methods (liquid and gaseous, both extractive and online) can be found under Chapter 3.

The pilot plant is located inside a 40 ft (12.2 m) container. Columns (absorber and stripper) penetrate the container roof and are supported by backstays. The height of the absorber column is 26 m and the height of the stripper column is 17 m. A second 20 ft (6.1 m) container next to the pilot plant is used to house the process control system, as well as sensitive on-line measurement and analysis equipment. In addition, a dedicated onsite laboratory (a so-called field laboratory) for some of the liquid sample analysis was set-up for routine liquid analysis.

The pilot plant is equipped with provisions to enable online measurement as well as sampling points for external analysis sampling. The flue gas quality, including amine emissions, was measured by PTR-TOF-MS equipment hired from and maintained by the University of Oslo (UiO).

The pilot plant can be divided into three parts with the following main equipment:

- pre-treatment: pre-scrubber.
- capture unit: absorber, stripper, water wash and demister.
- post treatment: aerosol emission mitigation device (AMD).

Fig. 2 shows a 3D representation and an actual on-site picture of the pilot plant that was built inside a 40 ft container. It should be noted that the columns (stripper, absorber and pre-scrubber) penetrate the container roof and that the post treatment unit was located outside the container.

Notable differences to the full-scale plant design were that (FOV FEED report, 2020):

- No thermal reclaimer unit (TRU) was installed;
- No mechanical vapour recompression (MVR) system was installed;
- Steam supply was provided from a separate steam generator instead of from the WtE plant;
- No caustic was injected in the pre-scrubber;
- The cooling system was designed as a once-through system, i.e. no closed loops in the cooling system;
- All piping and vessels were made from stainless steel 316 (unlike in the full-scale design which consists of various materials optimized considering area of use)

These differences are mostly linked to cost and complexity considerations and were accepted as they did not affect the relevance of the pilot plant results for the full-scale plant.

Table 2
Key process values used to design the pilot plant.

Flue gas properties	Operating values
Inlet pressure (kPa)	100
Inlet temperature (�C)	110
Mass flow (kg/h)	870
Molar weight (kmol/kg)	27.85
Circulating solvent properties	Operating values
Mass flow - lean amine	Per Design

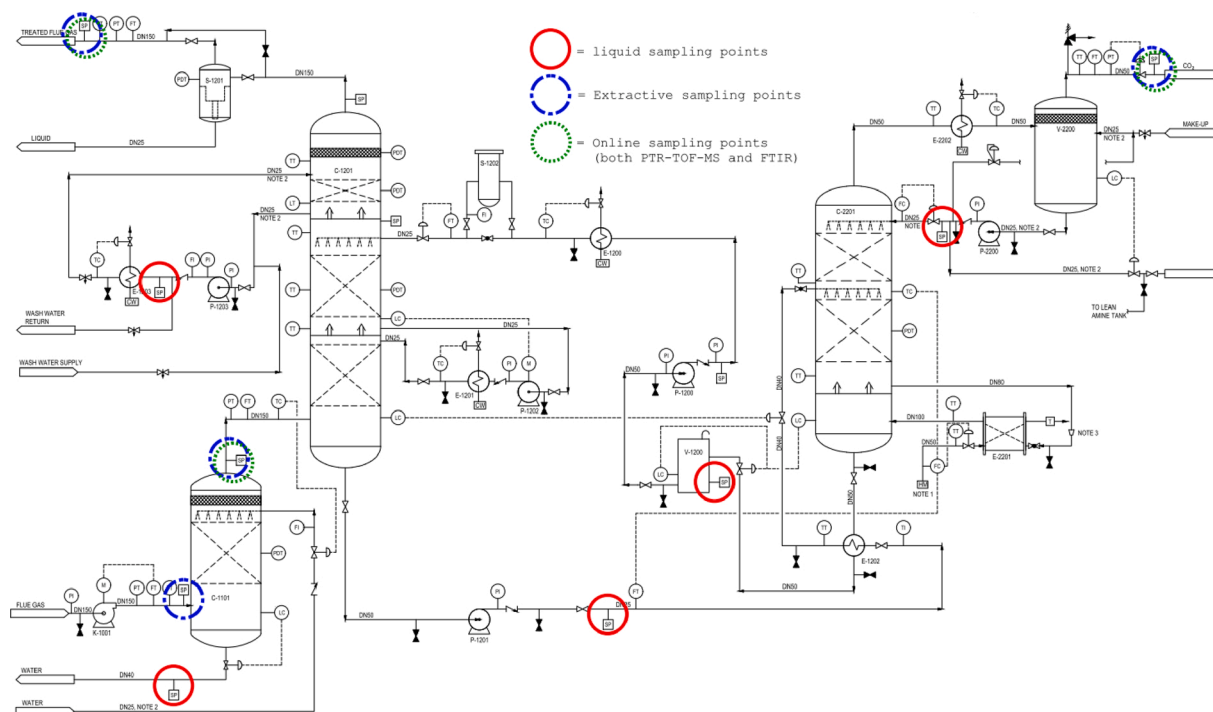


Fig. 1. Simplified PFD of the pilot plant with main measurement and sampling points indicated (FOV FEED report, 2020).

2.2. Pilot plant process description

2.2.1. Pre-scrubber

Before entering the absorber, the flue gas, a slip-stream from all three WtE plant incineration lines (see Chapter 2.3), needs to be cooled down. The pilot plant was designed for cooling down the flue gas to a temperature of $<40\text{ }^{\circ}\text{C}$ by direct contact with water (no additives) in a pre-scrubber (thus limited to the role of a direct contact cooler).

2.2.2. Absorber

From the pre-scrubber, the flue gas flows through the CO_2 absorber which consists of an absorption section based on structured packing beds and one water-wash section at the top. In the absorber, the flue gas is contacted with the solvent (DC-103) flowing down the packing beds.

The lean solvent temperature at the inlet of the absorber is controlled through cooling in the lean absorbent cooler, and further cooling to limit the temperature bulge in the absorber resulting from the exothermic absorption of CO_2 , is provided by circulating the solvent through an absorber intercooler.

After the absorption sections, the flue gas is passed through a water-wash section to minimize the solvent concentration leaving with the flue gas. The water used in the water-wash section is continuously recycled and cooled in the water-wash cooler. Water is thus condensed from the flue gas in the water wash section, and excess water is drained to the solvent to prevent solvent build-up in the water.

After the absorption column, the CO_2 lean flue gas can be directed to the stack for release to atmosphere, but as part of test campaigns a post-treatment step was also included in the design. This allowed testing the impact on emissions to air with and without post-treatment.

The CO_2 rich solvent is pumped to the CO_2 stripper via a lean/rich heat exchanger from the bottom of the absorber.

2.2.3. CO_2 stripper

The CO_2 stripper consist of a stripping section with a collector/chimney tray below the packing, and a reflux section on top of the column. The rich amine enters the CO_2 stripper on top of the stripping section of the column. CO_2 is removed from the amine in the stripping

section with the liquid accumulating in the bottom tray of the column and further routed to the stripper reboiler. Here the solution is heated by steam and transferred back to the bottom section of the CO_2 stripper where the flashed vapours are separated from the liquid. The vapour re-enters the stripping section through the chimney tray to strip out CO_2 from the rich amine.

The gas from the stripping section passes through the reflux section of the CO_2 stripper, and is routed to the reflux condenser. The residue steam is condensed, and liquid is separated from the gas in the reflux drum. The bulk of the liquid is returned to the top of the reflux section of the CO_2 absorber.

The lean amine exiting the bottom of the stripper is routed to lean/rich exchanger where it is cooled against a flow of rich amine before entering the lean amine tank.

2.2.4. Solvent purification

As mentioned above, the pilot plant is not equipped with a solvent reclaiming system. Starting with pure solvent, the degradation products concentration is left uncontrolled and allowed to progressively increase over the campaign. This allows to accurately estimate the degradation rate of the solvent, and also to compare performance at different concentrations of degradation products.

The only solvent purification system in place is a mechanical / activated carbon filtration system that can be brought online if and when required.

2.3. Existing waste to energy plant

During testing, the pilot plant was located next to FOV's waste to energy (WtE) plant in Klemetsrud, Oslo, Norway. The Klemetsrud WtE plant consists of three separate incineration lines, called K1, K2 and K3, respectively. All three lines will feed flue gas to the planned full-scale CC plant and it is important to note that the flue gas composition of the three incineration lines is different. Therefore, a representative flue gas sample (feed to the pilot plant) consists of flue gas from all three incineration lines in the right proportion, roughly 23/23/54 (vol%).

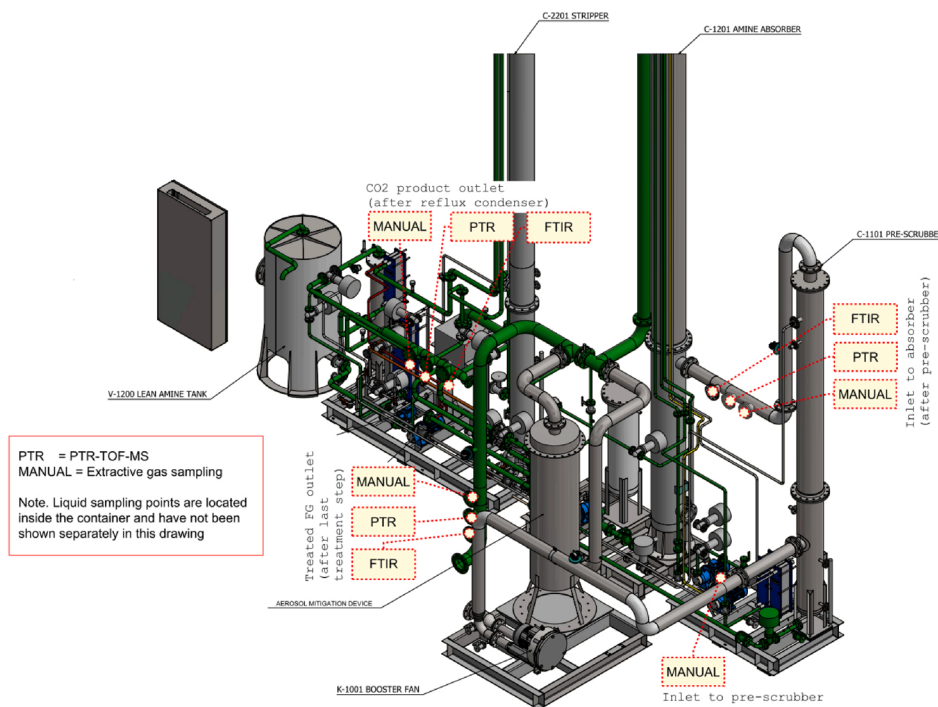


Fig. 2. 3D representation (top image (modified from FOV FEED report, 2020), container not shown) and picture (bottom image) of the FOV pilot plant for carbon capture (image courtesy of FOV).

2.3.1. Waste incineration lines K1 and K2

All three lines consist of individual grate fired boilers, i.e. waste is fed via a feed hopper to a slowly moving grate where the combustion of the waste takes place. Air for incineration is primarily provided through the grate, but additional air is also provided at other locations to ensure an excess of oxygen for complete combustion. The temperature in the furnace is typically between 850 and 1100 °C and most of the heat is recovered by the steam system, consisting of water and steam filled tubes in contact with the hot flue gases.

After primary heat recovery has taken place and the flue gases have passed through the boiler, the temperature of the flue gas has been reduced to around 200 °C. This temperature is however still too high for the following flue gas treatment steps and the temperature is reduced further by a heat exchanger (providing heat to the district heating network).

Downstream ECO 1 (economizer), the flue gas is treated with calcium hydroxide (Ca(OH)₂) and activated carbon in order to remove acidic and other harmful components. Nitrogen oxide emissions are reduced already in the boiler using a method called selective non-catalytic reduction (SNCR) that involves aqueous ammonia injection. The final flue gas cleaning step for lines 1 and 2 consists of a series of bag filters for reducing the particle amount down to the allowed emission level.

A schematic flow diagram with indicative temperatures of the flue gas treatment system is given in Fig. 3.

2.3.2. Waste incineration line K3

While the basic setup (incineration, heat recovery, flue gas treatment, disposal through stack) of the waste incineration line 3 is similar to that of K1 and K2, the flue gas treatment consists of slightly different

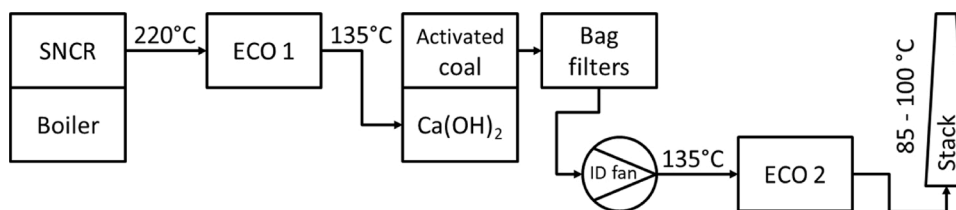


Fig. 3. Schematic flow diagram of the flue gas system for K1 and K2 (modified from FOV FEED report, 2020).

elements and arrangements. The first part of the flue gas treatment consists of an electrostatic precipitator (ESP) for particle removal, then the flue gas is passed through a 4-stage wet scrubber and finally it is treated in the selective catalytic reduction (SCR) reactor, again using aqueous ammonia.

Besides the differences in flue gas treatment it should be noted that K1 and K2 have different heat recovery systems as well, both for heat and for power. A schematic flow diagram of K3 with indicative temperatures is given in Fig. 4.

2.4. Flue gas composition

The flue gas composition to the pilot plant (into the CC plant pre-scrubber) is the same as that leaving the existing WtE plant. The tie-in points for the pilot plant for all lines are located after the final treatment steps which also represents the case for the full-scale design. Table 3 provides average values of the combined flue gas (all three lines mixed) as measured between 2017 and 2018.

The most common mixture of flue gases (~80 % of the time) entering the full-scale CC plant would be 23 vol% from K1, 23 vol% from K2, and 54 vol% from K3. The contribution of K1, K2 and K3 to the pilot plant feed gas was kept as close to this ratio as possible.

The flue gas supply lines from the WtE plant to the pilot plant are heat traced and thermally insulated in order to ensure that the flue gas entering the pilot plant is identical to the flue gas in the WtE plant stacks.

2.5. Pilot plant key parameters

Table 4 represents the key parameters needing to be controlled for successful pilot plant operation (i.e. representative of the full-scale plant operation). However, excursions (outside the typical range) for testing purposes have been made.

3. Test program overview

The operations of the pilot plant were divided into three campaigns, the first of which is called the 2000 h test campaign. The primary reason for setting the duration of the first test campaign to 2000 h were related to the fact that it takes time (estimated to around 1000–1500 hours with DC-103 at Klemetsrud) for the initially pure solvent to degrade sufficiently. A representative degradation concentration for full-scale CC plant operation is around 1–2 wt% degradation products concentration (on a dry basis) in the solvent. In addition, the time for familiarization and troubleshooting should not be neglected when operating a new-built pilot plant for the first time and sufficient time should be reserved for

Table 3

Combined, all three lines, average (yearly) flue gas data at Klemetsrud WtE plant.

Component	vol%	Component	mg/Nm ^{3*}
H ₂ O	13.9	NO	43.9
CO ₂ (dry)	11.3	CO	23.1
O ₂ (dry)	7.3	SO ₂	7.0
N ₂ , Ar	balance	NH ₃	2.0
		NO ₂	1.2
		TOC	1.2
		HCl	0.8
		PM	0.7
		PM count**	19,000
		H ₂ S	0.3
		Trace elements	0.1

* 101.3 kPa, 0 °C, dry, 11 % O₂.

** Nuclei / cm³.

Table 4

Key process parameters for the pilot plant.

Variable	Unit	Target	Typical range	
Inlet absorber temperature	°C	40	30	– 50
Online instrument				
CO₂ removal efficiency	wt%	90+	80	– 100
Online calculation				
Amine concentration	wt%	50	45	– 55
Offline lab result				
Reboiler pressure	bar(g)	1.0	0.9	– 1.1
Online instrument				
Reboiler temperature	°C	122	120	– 125
Online instrument				
FG flow (absorber inlet)	kg/h	937	750	– 1124
Online instrument				

this as well.

Following the initial 2000 h, the pilot testing entered a so-called extended phase with two more campaigns executed between June and August 2019, and September to December 2019. It should be noted that no make-up solvent was added during the entire pilot plant operation.

During the over 5000 h of pilot plant operation, various tests were planned and performed, but the primary purpose, besides verifying the overall performance of the pilot plant, was always to measure and monitor the emissions to the air as well as to follow-up on solvent degradation over time.

Thus, this article focuses on results related to overall performance (such as CO₂ capture efficiency, CO₂ product purity, steam

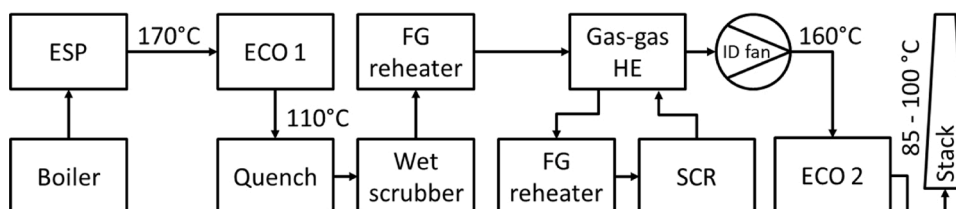


Fig. 4. Schematic flow diagram of the flue gas system for K3 (modified from FOV FEED report, 2020).

consumption), total amine emissions to air and solvent degradation over time.

3.1. Liquid sampling and analysis

There are five different liquid streams (as indicated in Fig. 1) on the pilot plant that have been focused on. These are, lean amine entering the absorber, rich amine exiting the absorber, pre-scrubber effluent, water wash circulation fluid and reflux circulation fluid.

The sampling points are provided with Dopak® sampling systems and all sampling points are located inside the main container.

The purpose of the liquid analysis was to analyse liquid samples for DC-103 solvent amines and degradation products. For the liquid analysis two different approaches were considered, i.e. so called “routine” analyses and “non-routine” analyses.

The routine analyses represented by CO₂ loading and amine concentration by titration was performed at Klemetsrud “field-laboratory”. While the non-routine analyses, such as liquid chromatography - mass spectrometry (LCMS) was shipped to labs with the appropriate equipment.

3.1.1. Solvent degradation

CO₂ was captured using DC-103 solvent and one of the main causes for costs (also environmentally) of operation is its degradation and subsequent requirement of make-up solvent. The full-scale plant is designed to operate with a specific degradation product concentration, in this case 1–2 wt%. In terms of actual solvent degradation, this would represent around 2–4 % due to the solvent water amount.

By frequently taking liquid samples (using the Dopak® system) of the circulating solvent and analysing its composition, the degradation rate over time was obtained. It should be noted that no make-up solvent was added during the entire pilot plant operation.

The degradation rate was obtained from liquid chromatography mass spectrometry (LCMS) results. The amine concentration determined by LCMS is the sum of the amines in the solvent, referred to as Am1, Am2, and Am3, and main degradation products, referred to as Deg1, Deg2 and Deg3.

3.2. Gas sampling and analysis

Gas analysis involved both extractive (manual) gas sampling and analysis and online gas measurements as detailed below.

Extractive gas sampling (using an impinger train, see supplementary material: S1) was done on four process gas streams (as indicated in Fig. 1). These streams were the inlet flue gas before and after the pre-scrubber, the treated flue gas after the absorber (and after AMD if in service), and the CO₂ product after the reflux condenser.

There were two types of main online gas analysers, FTIR and PTR-TOF-MS. In addition, O₂, SO₂, CO₂ and NO_x (mainly NO₂) were measured using separate instruments. The suitability of the PTR-TOF-MS instrument has been demonstrated at the CO₂ Technology Centre Mongstad (TCM) (Zhu et al., 2013; Morken et al., 2014).

The three process gas streams of importance that were monitored with on-line analysers (as indicated in Fig. 1) during the piloting were the inlet flue gas after the pre-scrubber (i.e. the absorber inlet), the treated flue gas outlet and the CO₂ product after the condenser. Both the FTIR and PTR-TOF-MS instruments had the possibility to measure at all three locations. This was made possible by manual switching between sampling locations.

All gas measurements were done iso-kinetically to ensure representative samples (with the exception of the CO₂ product outlet). The pilot plant has a number of 4" (DN100) connections for setting up isokinetic sample lines between the sample location and online analysis instruments. The length of the sample lines was minimized, and the sample lines were heated a suitable amount above the dew point of the gas to avoid condensation and other possible reactions.

3.2.1. Total amine emissions to air

As part of the Klemetsrud CC plant full-scale design, amine dispersion and formation simulations were performed, and the maximum allowed total amine emissions to the air was set at 0.4 ppmv. This number was obtained using flue gas dispersion analysis based on the NIPH recommendations of maximum concentrations of nitrosamines and nitramines in air (0.3 ng/m³) and drinking water (4 ng/l) sources around the future full-scale Klemetsrud CC plant. To ensure that all amine emissions have been considered a full-scan PTR-TOF-MS analysis was made. Any components detected (> 5 ppbv acetone equivalent) were quantified and included in the sum of amines to air, i.e. total amine emissions. In practice, due to the very low concentration of other amine species in the flue gas, the total amine emissions reported here are only due to the (most volatile) amine called Am1. It should be noted that the speciation of the solvent and degradation components in question cannot be disclosed for proprietary reasons.

3.3. Particle measurements and analysis

As part of the designed full-scale CC plant at Klemetsrud flue gas characterization has become increasingly important. Although the particulate matter, referred to as dust, content of the flue gas is typically very low on all three lines (see Table 3), the size distribution of the particles has been subject to additional investigations. The reason for this is mainly related to the apparent correlation between sub-micron sized particle number concentration and amine (mist) emissions (Mertens et al., 2014; Lombardo et al., 2017).

ELPI™ and ELPI+™ methods have been used for the particle number concentration and particle size characterization (see e.g. Lombardo et al., 2017) both before and during the pilot plant campaign. ELPI stands for electrical low-pressure impactor and is a real-time particle spectrometer which provides the total particle number concentration and the particle size distribution (PSD) of aerosols/particulate matter present in a gas stream.

4. Results

4.1. General performance

The pilot plant campaigns demonstrated the ability to sustain operation at the targeted high capture efficiency (90–95 %) while keeping the amine emissions to a minimum (less than 0.4 ppmv). And although the purpose of the pilot plant was not to demonstrate and optimise energy performance (already well documented, but cannot be accurately disclosed), some evaluation of the impact of various parameters on the desorber (stripper) steam consumption has been performed, thereby establishing operational ranges for optimal performance in terms of GJ (steam)/t CO₂ captured.

Furthermore, the long enough test period (see Fig. 5) allowed for various upset conditions (both intentional and unintentional) to be studied strengthening the conclusions of the pilot plant study.

From Fig. 5, it is possible to evaluate the performance of the pilot plant in terms of operational hours. It can be seen, that during the first 2000 h, a high priority was to achieve the required cumulative operational hours in a certain time-frame (resulting in low downtime). After that, the staffing of the pilot plant was reduced, and any disturbances/trips could not immediately be rectified leading to longer downtimes. The significant downtime periods have been numbered in Fig. 5 and below are brief explanations of the reasons why:

- 1 Trip due to high-high level in stripper followed by blower fan being stuck and requiring maintenance.
- 2 Unstable incineration plant (resulting in e.g. loss of “permissive to start” signal)
- 3 Water wash pump failed and was replaced.

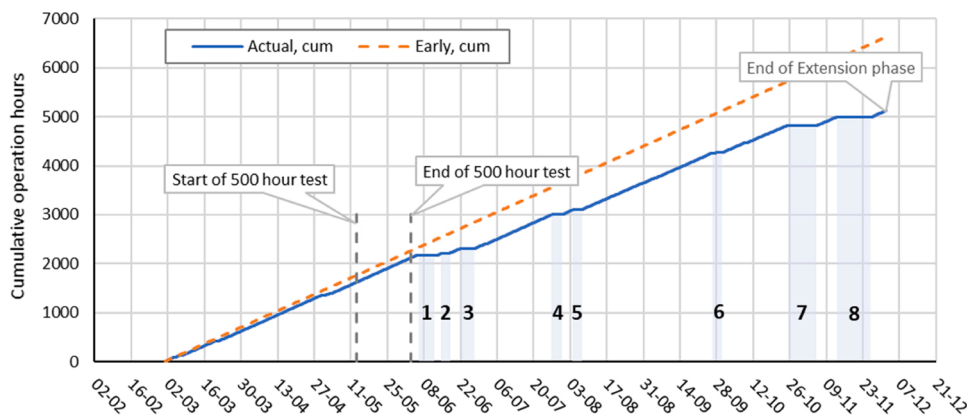


Fig. 5. Cumulative operational hours for the pilot plant (including an initial 24-h test run). The “Early, cum” represents how many hours could have been achieved without any downtime (significant downtime periods have been indicated by a number).

- 4 Plant tripped (due to flashing and low level in the stripper) followed by blower fan being stuck and requiring maintenance
- 5 Plant tripped (possibly due to lightning strike) followed by blower fan being stuck and requiring maintenance
- 6 Planned shut down due to service at WtE plant drainage system.
- 7 Trip due to high level in reflux drum followed by blower fan being stuck and requiring maintenance.
- 8 Fan motor short circuited. Further inspection revealed water in the connector lid to the motor, extensive service required. (Plant tripped December 1st caused by the same fan motor problem as before. Extensive service required and therefore no time for further pilot plant operation.)

Due to the multiple blower overhauls, it was decided to modify the control system to keep the blower fan running when the system trips (to avoid excessive downtime), in so called safe mode operation. This work was successfully completed (in September) and prevented the blower from being stuck during minor trips (not visible in Fig. 5).

It should be noted that the blower selected for the pilot plant (side channel type) is very different from the fan/blower type selected for the full-scale plant, i.e. similar problems are not to be expected.

4.1.1. CO₂ capture efficiency

Fig. 6 shows the capture efficiency as a function of time for the period June to August. The data for CO₂ capture has been based on 10-minute averages.

The continuously (assuming similar pressure and temperature conditions) estimated capture efficiency (η_{CO_2}) is based on the following equation (ref Method 1 in Faramarzi et al., 2017):

$$\eta_{CO_2} = \frac{CO_2 \text{ in product}}{CO_2 \text{ in supply}} = \frac{\dot{m}_{CO_2 \text{ product}} \times x_{CO_2 \text{ product}}(T_{sat})}{\dot{m}_{CO_2 \text{ supply}} \times x_{CO_2 \text{ supply}}}$$

where,

$\dot{m}_{CO_2 \text{ product}}$ = CO₂ mass flow after CO₂ reflux condenser

$x_{CO_2 \text{ product}}(T_{sat})$ = CO₂ concentration, assumed H₂O saturated based on temperature

$\dot{m}_{CO_2 \text{ supply}}$ = flue gas mass flow into absorber

$x_{CO_2 \text{ supply}}$ = CO₂ concentration into absorber

During the extended testing phase, the concentration of CO₂ was not continuously measured at the outlet of the absorber, instead occasional manual (spot value) checks were done by shifting the CO₂ measurement device to the outlet of the absorber ($Efficiency = 1 - [CO_2, \text{ treated FG} / CO_2, \text{ supply FG}]$, ref Method 3 in Faramarzi et al., 2017). The results of both the CO₂ measurement estimates and the spot checks have been presented in Fig. 6.

For testing purposes, the CO₂ capture efficiency has been allowed to vary. The CO₂ mass balance (closure), based on performed spot checks and flow meters located at the inlet of the absorber and the CO₂ product outlet, has been below 2 %-points. The overall mass-balance has been less accurate at times (depending mainly on incoming flue gas variations, such as moisture content) as can be seen from Fig. 7.

The reason for the fluctuating flue gas mass flow through the pilot plant (see Fig. 7) is related to a number of things, mainly the capacity of the FG blower (operating close to max capacity at all times), availability of FG from all three WtE incineration lines to the capture unit and testing purposes.

4.1.2. Steam consumption

Although the specific steam consumption (per ton CO₂) measured at

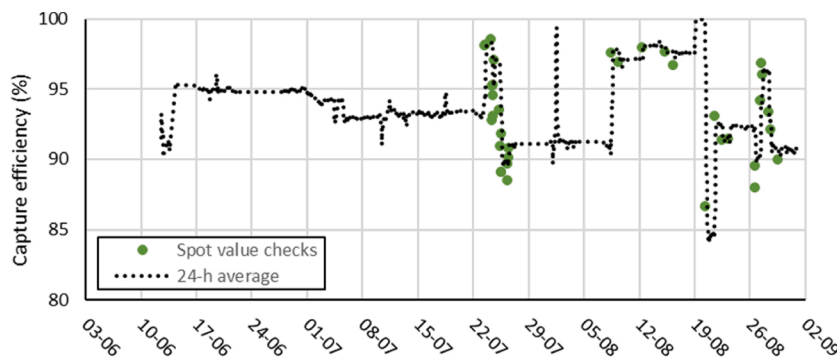


Fig. 6. CO₂ capture efficiency based on absorber balance for June to August. The dotted line represents 24-h running averages.

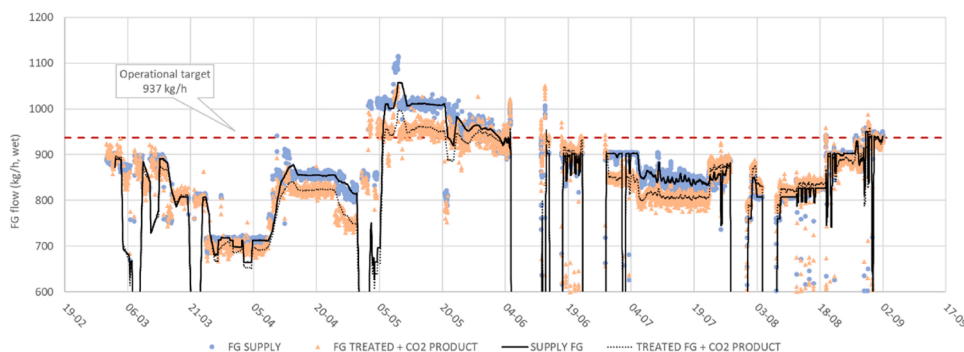


Fig. 7. Flue gas mass flow to the CC plant as a function of time. Mass balance closure estimated by plotting the sum of treated flue gas and CO₂ product as one line. The (trend)lines represent moving averages of 50 h.

the pilot plant is very different to that designed for the full-scale plant, a good comparison with simulated results was obtained.

During the extended testing campaign (calendar weeks 38–40), tests were carried out to optimise the specific steam consumption (GJ/t CO₂). In practice this was done by changing the lean amine and steam flow to predetermined values, while maintaining other parameters as stable as possible.

Fig. 8 shows the capture efficiency obtained for different lean amine to gas flow ratio (L/G) and stripping factor (SF) conditions. The SF values were calculated based on the latent heat of vaporisation for pure water at the inlet steam temperature divided by the mass of CO₂ product out. Specific SF were in line with expectations/modelling results. To eliminate the impact of pilot plant size on steam consumption, Fig. 8 shows SF values relative to the SF value for operating conditions representative of the full-scale plant. This indicates the range of efficiency improvement available using this method. All points represent average values during representative/ comparative time periods of various durations (~6 h). The lines represent 2nd order polynomial fits to the available data points. Note that for the 93 % capture efficiency, there are only two periods of representative data, i.e. only two data points, thus the straight line.

4.1.3. CO₂ product purity

On two occasions, the PTR-TOF-MS instrument was switched to measure the composition of the CO₂ product. The purpose was to verify that the CO₂ product purity is in line with expectations and to evaluate the impact of solvent degradation on this purity.

During the first PTR-TOF-MS CO₂ product measurement campaign in May, only the main solvent component (Am1) and acetaldehyde was reported/detected. During the second campaign in October, the results showed a wider range of detectable components, represented by formamide, acetone and three unidentified organic compounds (oxygenated and nitrogenated hydrocarbons) (OC's) referred to as OC1, OC2 and

OC3 in Table 5.

In addition to PTR-TOF-MS measurements, the CO₂ product was also evaluated by taking extractive samples to be analysed in a lab (see supplementary material: S2). The main results from the extractive CO₂ sampling taken in June 2019 are:

- Acetaldehyde measured at 0.5 ppmv, fairly consistent with average value of 0.6 and 0.8 ppmv as measured previously by PTR-TOF-MS (see Table 5)
- Confirmation that all components are within CO₂ product specification (see supplementary material: S4), including O₂ (assuming a normal oxygen removal step, as designed for the full-scale plant).
- No formaldehyde (LOQ: 0.1 ppmv) or ammonia (LOQ: 0.5 ppmv) were detected

4.1.4. Activated carbon filter operation and foaming tests

Although there were no issues with foaming during the pilot plant operation, an activated carbon filter was included in the design to be used if needed. The use of activated carbon to reduce foaming has been demonstrated at Boundary Dam (ICCS presentation, 2017).

The activated carbon filter (ACF) ended up being tested without flue gas from the WtE plant, having only the solvent circulation pumps turned on. Solvent samples were taken both before and after the ACF test to be analysed for foaming tendency. Both samples showed reduced tendency towards foaming compared to earlier samples and as expected, the active carbon treatment made the solvent solution more transparent.

4.2. Amine emissions to air

The amine emissions have been monitored continuously by PTR-TOF-MS (excluding periods when the instrument was used to analyse the CO₂ product gas stream). During the first 2000 h, the primary purpose of the pilot plant testing was to demonstrate low amine emissions during various normal (stable) operational conditions. Having successfully demonstrated low amine emissions during normal operation, the post 2000 h testing period focused in turn of testing the emission handling capacity of the pilot plant during various upset conditions. Therefore, the focus of the results is also on the latter, post 2000 h testing period.

4.2.1. First 2000 h of testing

After a period of familiarization / troubleshooting (first weeks of testing), the amine emissions stabilized below the target of 0.4 ppmv on average. During the 500 h test the amine emissions were on average of 0.16 ppmv (based on PTR-TOF-MS data reported on a minute basis), and only exceeded the target of 0.4 ppmv during an upset condition of the ESP (note, that the AMD was being bypassed when the upset condition happened).

Table 6 summarizes the average emissions during the 500 h test

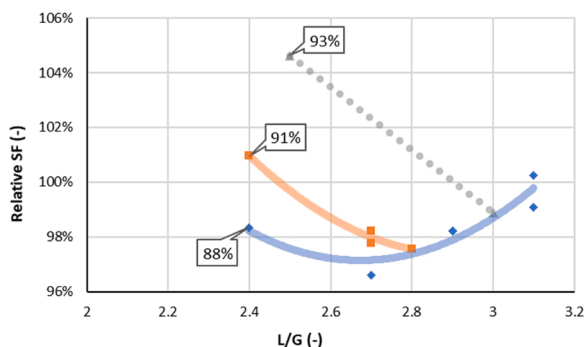


Fig. 8. Specific steam consumption / stripping factor (values not disclosed) as a function of L/G for different capture efficiencies (88, 91 and 93 %).

Table 5

Indicative (PTR-TOF-MS not calibrated for pure CO₂ matrix) results of trace components in CO₂ product as reported/measured by PTR-TOF-MS in October. Where available, first campaign results from May shown in parenthesis.

	Acetaldehyde (ppbv)	Formamide (ppbv)	Acetone (ppbv)	OC1 (ppbv)	OC2 (ppbv)	OC3 (ppbv)	Am1 (ppbv)
Max	718 (2400)	76	24	28	11	25	3 (83)
Avg.	587 (800)	59	21	24	7	15	1 (3)

Table 6

Amine emissions during the final 500 h of the first pilot plant test campaign (modified from FOV FEED report, 2020).

	units	value
Target	ppmv	< 0.4
500 h test period	ppmv	0.16
500 h test period excluding ESP malfunction	ppmv	0.04

period.

4.2.2. Post 2000 h testing - extended phase

Following the end of the first 2000 h of operation, the pilot plant testing entered a so-called extended phase. During this period, the amine emissions continued to be low, with a median value as low as 0.01 ppmv (right image of Fig. 9). Fig. 9 shows the distribution of average hourly emission values for the entire extended phase (left image) and e.g. that 80 % of the time, the amine emissions have been below 0.05 ppmv. The average for the entire period was 0.12 ppmv (target <0.4 ppmv), including the intentionally induced amine emission tests and the high amine emissions experienced during weeks 42–48. It has been possible to provide explanations for all significant emission peaks / spikes and excursions, most noteworthy those correlating with elevated dust concentrations in flue gas from K3.

The concentration of other (than the main solvent) amine in the treated flue gas stream have not been significant during the entire extended test period (June to December). However, a new degradation product, referred to as "Deg4", was detected in the emissions to air around August 28 and 29 coinciding with an extreme absorber temperature test of 60 °C (representing absorber inlet and outlet FG temperatures). This new degradation product was not detectable before August and the concentration of Deg4 in the gas is normally low (below 5 ppbv) and does not impact the 0.4 ppmv emission target. Since August, Deg4 has been reported/detected reaching up to 60 ppbv (hourly average), in general coinciding with the amine (Am1) emission peaks visible in Figs. 10 and 11.

The results presented here consist of PTR-TOF-MS measurements that account for all amine emissions above 5 ppbv (reporting limit). It should be noted that including Deg4 to Figs. 10 and 11 does not change the appearance of the Figures significantly nor the conclusions and therefore the reported total amine emissions consist of only one type of amine, referred to as Amine 1 (Am1). The amount of the other known amines (Am2 and Am3) in the solvent and known degradation products

(Deg2 and Deg3) were typically below the 5 ppbv reporting threshold and therefore not considered for the reported total amine emissions.

Figs. 10 and 11 have been created using 1 -h average data. Fig. 10 emphasises the fact that the 24 -h average data (dotted line) has stayed below the 0.4 ppmv target concentration for the entire period, except for the period of FG from K3 (ESP upset condition) with unusually high dust concentration. Fig. 11 highlights the emission peaks that have been detected (mainly related to pilot plant trips or testing purposes). Items number 9, 10 and 11 in Fig. 11 are discussed in more detail in Chapter 4.2.3.

In Fig. 11, the most significant emission peaks have been indicated and below is an explanation for each of the instances:

- 1 Multiple pilot plant trips and subsequent restarts
- 2 Absorber inlet and outlet temperature testing
- 3 Plant restart after trip
- 4 Absorber temperature test (60 °C) combined with rapid changes in flue gas flow
- 5 Rapid changes in FG flow amount
- 6 Speculated (missing data for K3) FG compositional change
- 7 Sudden drop in FG flow (and negative delta T over water wash section)
- 8 Sudden drop, followed by sudden increase in CO₂ conc. (Smaller peak under #8 related to starting and stopping the pilot plant)
- 9 Induced emissions testing (with and without AMD) coinciding with unusual dust concentrations from K3. See Chapter 4.2.3 for more information.
- 10 Induced emissions testing (with and without AMD) coinciding with unusual dust concentrations from K3. See Chapter 4.2.3 for more information.
- 11 Induced emissions testing (with and without AMD) coinciding with unusual dust concentrations from K3. See Chapter 4.2.3 for more information.

The notably long period during July and August shows that the emissions are low during normal operation. However, it should be noted that the flue gas flow rate was slightly lower than design (see Fig. 7). The maximum achievable flue gas flow was reduced around July/August due to K3 maintenance period. Thus, the results indicate that the system is robust with regards to amine emissions when flue gas flow and CO₂ loading (K1 and K2 have lower CO₂ content than K3 on average) in the system is somewhat reduced compared to design capacity.

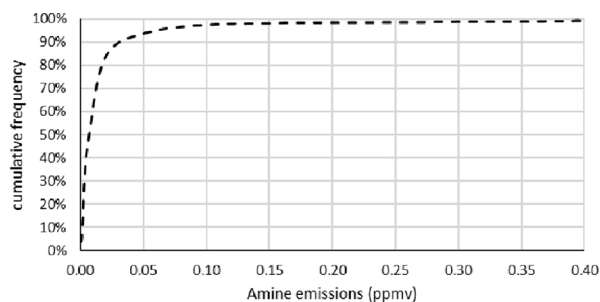
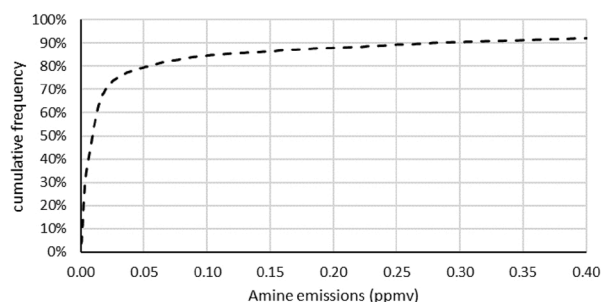


Fig. 9. Cumulative frequency distribution of hourly amine emissions during the entire extension phase (left image, representing around 3100 h of operation) and during the extension phase, but excluding weeks 42 onwards (right image, representing around 2500 h of operation).

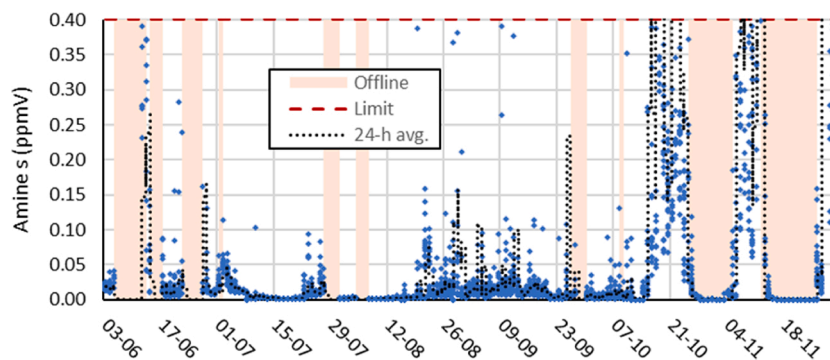


Fig. 10. Total amine (i.e. Am1) emissions measured in the period between June and August based on hourly averages. The dotted line represents 24 -h averages and the dashed line represents the long-term average emission limit.

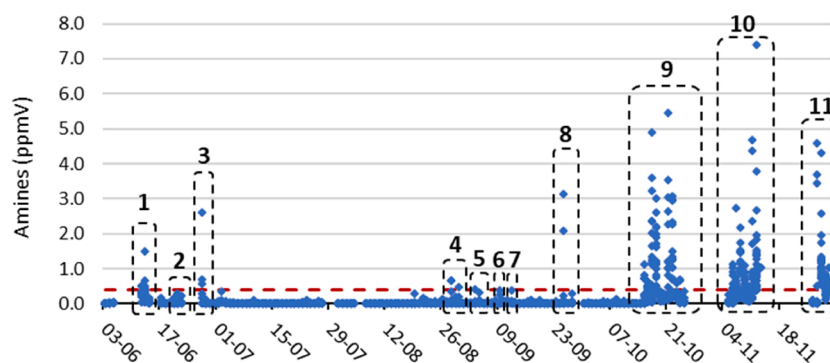


Fig. 11. Total amine (i.e. Am1) emissions measured in the period between June and August based on hourly averages (same data, but different scale than Fig. 10). The most significant emission peaks have been indicated with numbers and the dashed line represents the long-term emission limit.

4.2.3. Emissions in week 42 and 48

Week 42–48 represented weeks of unusually high amine emissions at the pilot plant as can be seen from Fig. 11. The high amine emissions correlate well with unusually high dust concentrations measured at K3 (at the WtE plant) as can be seen in Fig. 12. This also underlines the importance of testing the CC technology with real flue gas as pointed out by others (e.g. Moser et al., 2017). Unfortunately, there is no size distribution data for the dust in question and it can only be suspected that sub-micron sized particles and/or aerosols have been present causing the emission peaks.

Normally the concentration of aerosols / sub-micron sized particles measured at Klemetsrud WtE plant have been low, particularly for K1 and K2 where the flue gas is being cleaned using bag filters. Also, K3 has shown low concentrations of aerosols during testing (using ELPI+) performed prior to the pilot plant campaigns, see Table 3.

The reasons for why K3 has periods with more dust being emitted is related to the operational logic of the electrostatic precipitator (ESP). It is possible that the ESP outlet flue gas dust concentration and voltage across the ESP are correlating. A sometimes-lower voltage is related to the flue gas composition, moisture, dust concentration and composition etc., which in turn affect the breakdown voltage (or sparkover). When sparkover occurs, the high voltage load is rapidly turned off and on again until a new sparkover has occurred at a new level of voltage. This is considered as an upset condition and will most likely not occur frequently (based on historic data, it did not take place at Klemetsrud during the entire year 2018).

Note that periodic (every 4 h) peaking dust measurements have been filtered out from the data seen in Fig. 12. These (not shown) peaks are related to the cleaning of the ESP equipment and do not correlate with emissions at the CC plant. In addition, note that the pilot plant was not in operation during weeks 44 and 47 and therefore no graphs for these weeks can be presented.

4.2.4. AMD operation

The pilot plant was equipped with a commercially available flue gas post-treatment unit, called an aerosol mitigation device (AMD). In principal the unit consisted of special filter candles for sub-micron mist elimination. The reason for testing/including this unit to the pilot plant was to ensure that low amine emissions to air could be maintained at all times, i.e. even during unusual / upset process conditions.

While the AMD was operated continuously for a prolonged (3.5 weeks) period in July, it did not become clear how much the AMD was reducing/impacting the amine emissions. Therefore, a number of subsequent testing weeks (week 41 onwards) were committed to quantifying the amine emissions with and without AMD in operation.

During normal operation, the amine emissions are very low both with and without the AMD in operation (see Fig. 13) and therefore the target was to simulate (by rapidly changing the FG flow) upset conditions in order to quantify the effect of the AMD on amine emissions.

4.2.4.1. Stable operation. Fig. 13 shows an example of a graph with representative data obtained during stable operation. Each data point represents average values over significant periods of time and while comparative enough for the purposes here, it does contain varying operating conditions (capture efficiency, CO₂ concentration, L/G etc.). All of these have been plotted in the same graph, essentially showing that during normal operation the difference between operating with and without AMD is negligible in terms of amine emissions.

While the pilot plant operation was not very stable during dusty conditions from K3, it has been possible to obtain periods of time (of at least 1 h in duration) with fairly stable operation even during elevated dust conditions as those plotted in the right graph of Fig. 13. These have been further plotted against the average dust content measured during that time in Fig. 14.

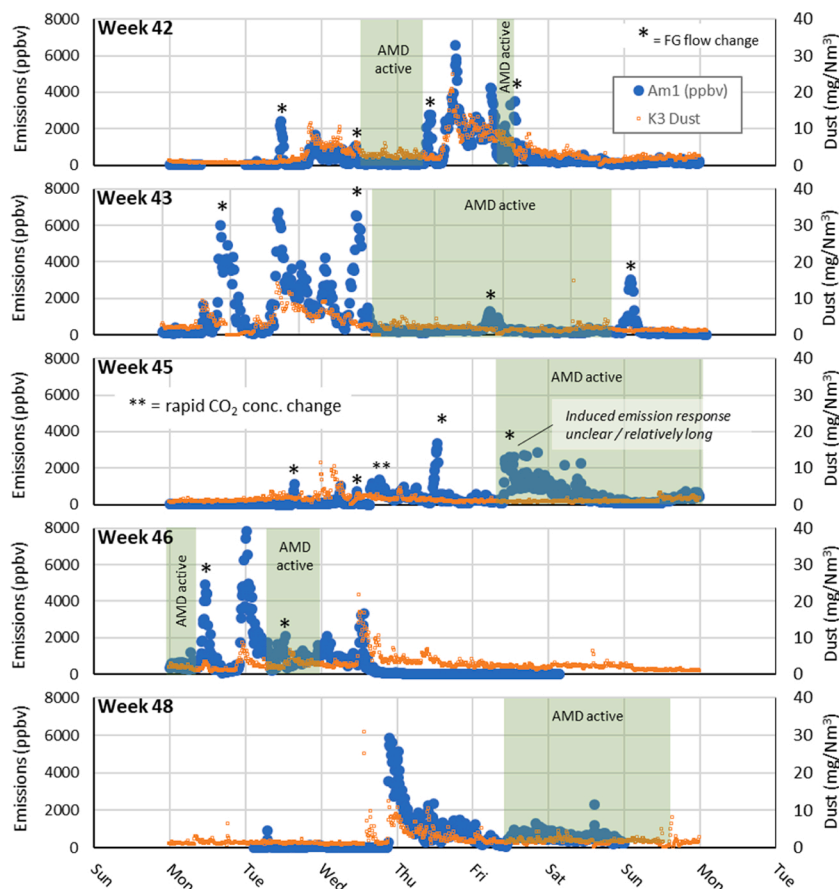


Fig. 12. Amine emissions, during weeks 42 to 48, together with dust measurements of K3 flue gas. AMD in operation has been indicated, as well as instances of induced process upsets / FG flow change and in one case rapid CO₂ concentration change (week 45).

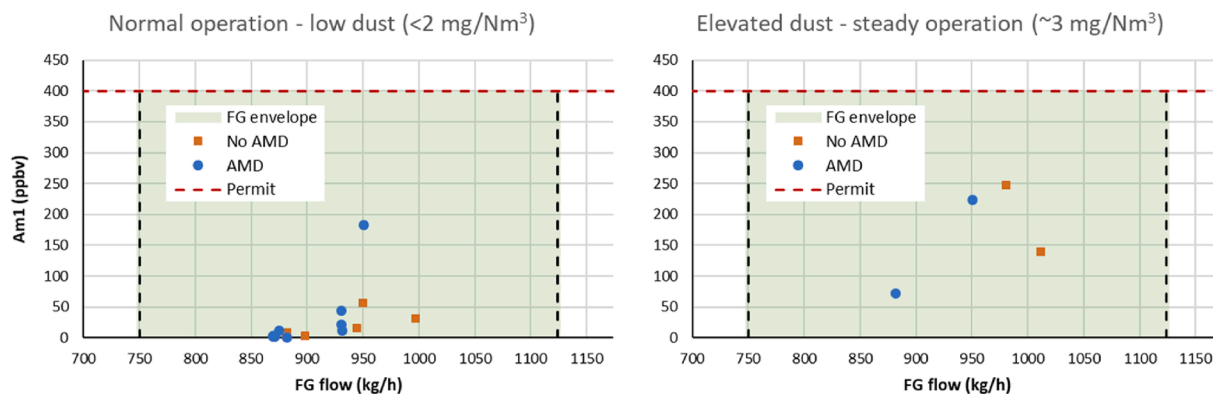


Fig. 13. Average amine emissions both with and without AMD in operation during normal (left figure) and elevated FG dust conditions (right figure). The highlighted area (FG envelope) represents the acceptable operational range for the pilot plant in terms of FG flow and amine emissions.

4.2.4.2. *Upset conditions.* Upset conditions were generated by rapidly decreasing the FG flow through the absorber both with and without the AMD in operation. Representative/comparative data from these experiments have been presented in Fig. 15. Because, at the time of testing, the FG contained unusually high amounts of dust (originating from K3), the data has been plotted against K3 dust concentration.

From Fig. 15 it can be seen that the AMD reduces both the intensity (right-hand Figure) and the duration (left hand Figure) of the emission peaks. Ideally, all other parameters except for AMD in operation or not, would be exactly the same allowing for a clean comparison. However, since this has not been the case, especially with regards to FG composition (dust content), quantifying the emission reduction capacity of the

AMD is not straightforward. However, an attempt to quantify the emission reduction capacity has been made based on the data in Fig. 15 by estimating the difference in the linear slopes. In case of both average and peak amine emissions (peak emission intensity), the reduction factor was around 5.

4.3. Other emissions to air

Acetaldehyde, formaldehyde, and ammonia are highly volatile compounds (in the CO₂ capture plant operating conditions), and will be stripped from the absorbent, either in the absorber or in the stripper.

Measurements showed low concentrations of both aldehydes (using

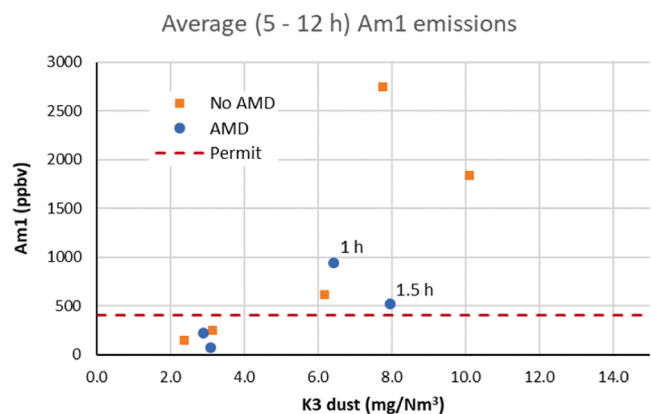


Fig. 14. Prolonged (5 to 12 h) amine emission concentrations with and without AMD in operation during unusually high dust concentrations from K3. Each data point, except when marked otherwise, represent average values between 5 and 12 h of duration.

PTR-TOF-MS) and ammonia (using FTIR and extractive sampling) throughout the test campaign. Formaldehyde was on average below the 5 ppbv reporting threshold, while acetaldehyde (using PTR-TOF-MS) remained below 0.1 ppmv on average. Ammonia was below 1 ppmv (detection limit for FTIR) during normal operation (during the first 2000 h of testing). It is notable that the emissions of aldehyde and ammonia to air did not increase with increasing solvent degradation.

Furthermore, it should be noted that the concentration of ammonia is typically higher in the WtE plant flue gas (see Table 3) compared to that leaving the CC plant.

4.4. Solvent degradation

Figs. 16 and 17 show the progression of solvent degradation products over time (as measured by LCMS). Fig. 16 shows degradation as a function of the cumulative operational hours from start of the pilot plant until end of November, while Fig. 17 shows the degradation as a function of when the sample was taken (i.e. irrespective of pilot plant up/downtime).

The total amount of degraded products (measured as received, i.e. on a wet basis) is based on the sum of three known degradation products called Deg1, Deg2 and Deg3. Work was performed to validate if Deg4 (or any other significant amine-based degradation components) could be detected in the solvent. However, no Deg4 compound was found, the reason for this being either the fact that the compound was not present in the sample or the concentration of the compound was too low to be detected.

From Fig. 16, it can be seen that the typical design range for degradation products in the solvent has been exceeded after around 3000 operational hours and that there is a notable upwards trend in the otherwise fairly linear curve of total degradation products once the concentration reaches around 4 wt%, possibly even at slightly lower concentrations. Additional operational hours would be needed to show if the build-up rate of degradation products in the solvent is indeed accelerating. It should be noted that this apparent acceleration also coincides with the elevated dust conditions experienced during the last phase of testing.

Plotting the data against time (i.e. including both operational and non-operational hours), shows a more linear trendline, indicating that degradation could have continued during downtime. Normally this not the case as most of the solvent stays cold and lean, but in case of repeated

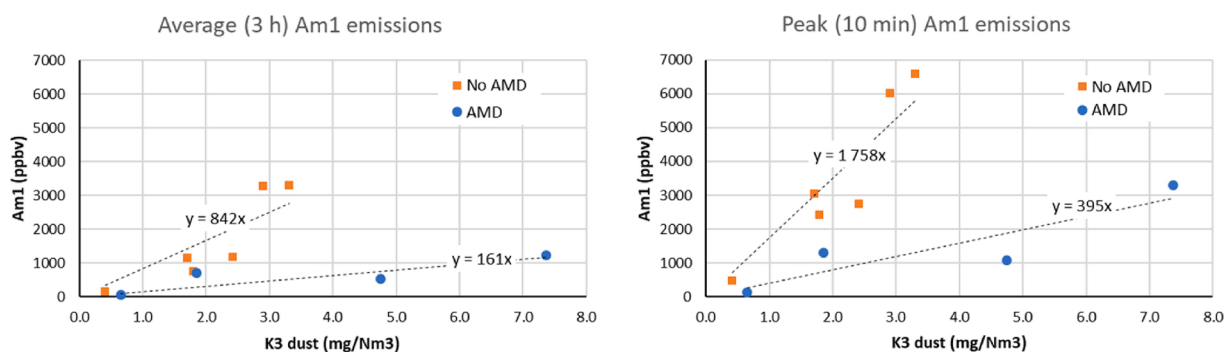


Fig. 15. Amine emissions as a function of K3 dust content both with and without AMD in operation. The left figure represents 3-h average amine emissions, while the right figure represents peak/maximum 10-minute amine emissions around the time of the simulated upset conditions. The dotted lines indicate the slope for a linear fit of the data.

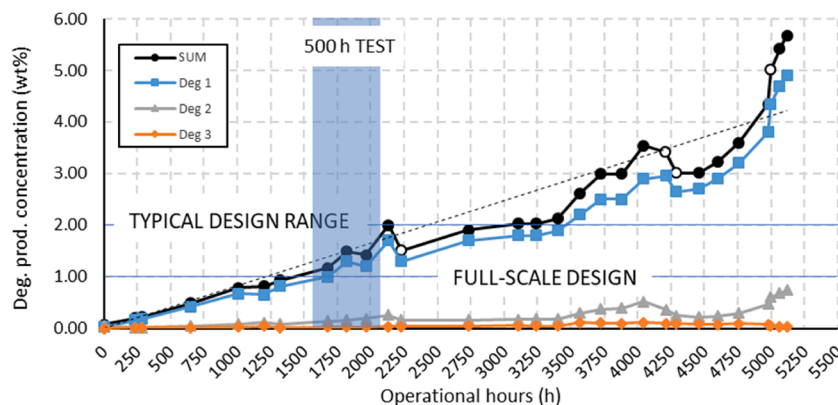


Fig. 16. Solvent degradation as measured by LCMS as a function of operational hours. The none-filled dots represent average values of samples that have been analysed twice.

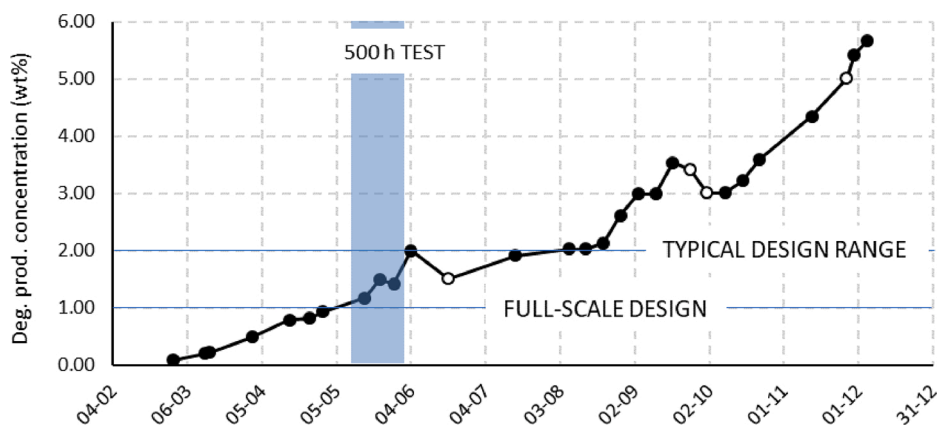


Fig. 17. Solvent degradation as measured by LCMS as a function of time regardless of operational hours. The none-filled dots represent average values of samples that have been analysed twice.

trips where rich amine in the stripper just falls to the bottom, where it remains hot for some time, this may have happened.

4.4.1. IC and ICP-MS analysis results

A number of lean amine samples have been analysed by both IC (anions) and ICP-MS (cations). This information is useful to determine the requirements for TRU operation as the anions would also need to be removed using the TRU.

With regards to the organic acids, the results were as expected, the accumulation rates of these compounds are orders of magnitude below the main degradation products accumulation rate.

Similarly, the ICP-MS results show low ingress of contaminants, but it appears as if the high dust loads from K3 are also visible in the final results.

For more information, see supplementary material: S3.

5. Conclusions

The CC pilot plant was in operation between March and December 2019 and the total number of successful pilot plant operational hours at Klemetsrud reached about 5100. The purpose of running the pilot plant beyond the original 2000 h was to obtain additional knowledge of operating the plant at higher concentrations of solvent degradation and various upset conditions.

The average amine emissions to air remained well below the emission target concentration (0.4 ppmv) during the whole campaign. However, excursions were observed during upsets, intentional or unplanned, during which the pilot was pushed in abnormal and transient operating conditions. It has been shown that an amine emissions mitigation device helps to reduce amine emissions to air during these upset conditions (experienced between weeks 42 and 48), but that its effect during normal operation appears less pronounced due to very low aerosol and amine emissions.

At the end of the campaign, the degradation product concentration in the solvent exceeded 5 wt% (i.e. around 10 % degraded solvent) without using a reclaimer. This is above the full-scale plant design envelope (1–2 wt% degradation products), where a thermal reclaiming unit (TRU) will maintain and control the degraded product concentration. The impact of a higher than design degradation product concentration has been negligible and indicates an opportunity to improve/ optimise the TRU/ full-scale CC plant design. An indication of a possible acceleration of degradation product build-up rate was only observed at the most elevated concentrations at the end of the campaign but would have needed continued operation to be confirmed.

Finally, the CO₂ product purity is subject to stringent specifications (for transport and storage purposes, see supplementary material: S4), and results of on-line (PTR-TOF-MS) analysis and (limited) extractive

sampling and analysis show that the produced CO₂ meets the required specification. This is accounting for a typical oxygen removal / dehydration step in the conditioning step which was not part of the CC pilot plant.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.ijggc.2020.103242>.

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