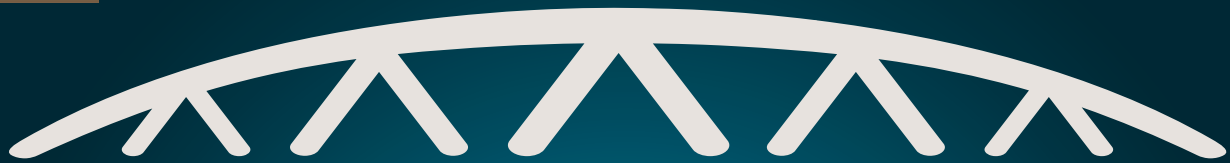


Selected papers from the 10th Trondheim Conference on
CO₂ Capture, Transport and Storage

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Trondheim CCS Conference

CO₂ Capture, Transport and Storage



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INVESTIGATION OF CORROSION-RELATED FAILURE OF REBOILER AT TECHNOLOGY CENTRE MONGSTAD

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Abstract

Technology Centre Mongstad (TCM) has experienced in 2017 a corrosion-related failure in a stripper reboiler of an amine unit, during a series of long-term test campaigns 2017-2018 for CO₂ capture using monoethanolamine (MEA). The failure affected the heat exchanger plates within the reboiler, made of 316L stainless steel.

A thorough investigation has been initiated in collaboration with Institute for Energy Technology (IFE), Equinor and Total to evaluate the key factors responsible for the damage and the mechanisms leading to the failure of heat exchanger plates. A two-phase approach was adopted. In the first phase the aim was to make a complete evaluation of all circumstances, outline the most plausible hypotheses for the cause of the damage, design and define a sensible scope for a detailed experimental laboratory study allowing to assess the factors contributing to the corrosion issues. The second phase is to encompass the experimental study to either confirm or refute the starting hypotheses.

This contribution presents a detailed account of the relevant findings, the reasoning and the outline of the planned laboratory investigations. On the other hand, the results of the experimental study itself are not part of this paper.

Keywords: *Post-Combustion Capture, MEA, Corrosion, Erosion, Oxygen Scavenger, Potassium Bisulfite*

1. Introduction

Technology Centre Mongstad (TCM) is the world's largest test facility where various technologies for CO₂ capture are assessed and qualified since its completion in 2012. The site is equipped with two distinct units, a proprietary Chilled Ammonia Process (CAP) plant and a highly flexible amine plant on which this paper focuses. The plant is currently owned by Gassnova, Equinor, Total and Shell.

In 2017, TCM started a long-term testing with MEA solvent through a series of test campaigns, to generate useful knowledge and information to be used to reduce the cost, the technical, environmental and financial risks of commercial scale deployment of post-combustion CO₂ capture. Some test activities were also conducted over longer periods in parallel to the abovementioned test series, one being material testing and corrosion monitoring for assessment of suitable materials for the post-combustion CO₂ capture process with MEA solvent. In July 2017, a corrosion-related failure occurred in a stripper reboiler of the amine plant, that affected the heat exchanger plates within the reboiler, made of 316L stainless steel. The operations of the amine plant over the experimental campaign yielded a complex scenario of varying conditions (i.a. flue gas type, solvent composition) over a long period of time, making it a difficult task to identify the key factor(s) responsible for developing the failure, since both corrosion and erosion

contributions could be plausible but without any factor appearing as an obvious and certain cause [2], [3].

In addition to the practical value of finding the causes for the damage, the importance of gaining a more fundamental understanding of the possible processes involved was recognized. It was therefore important that the failure assessment includes both a thorough study of the available information of relevance as well as validation by laboratory experiments.

A task force with TCM, Equinor and Total, as TCM owners, performed a complete evaluation of all circumstances leading to the failure, in order to identify the most plausible mechanisms and define working hypotheses to be verified experimentally.

In this context, TCM and their partners Equinor and Total have initiated a collaboration with Institute for Energy Technology (IFE) with the following objectives:

- a) To review the study performed to evaluate the circumstances of the failure
- b) To agree on the selection of the most plausible mechanisms for the failure and the working hypotheses to be verified experimentally
- c) To define a sensible scope for a detailed experimental laboratory study allowing to assess the factors contributing to the corrosion issues
- d) Perform the tests and assess whether the experimental results confirm or refute the hypotheses

A two-phase approach has been adopted, where evaluation, defining the hypotheses and scope would constitute the first phase whereas the second stage would consist of the experimental work and assessment of the results.

This paper is concerned with the first stage of this study, the experimental work will be reported separately in due course.

2. Evaluation of plant data

2.1. The amine plant and operational conditions

A simplified process flow diagram of the TCM amine plant is illustrated in Figure 1. The adjacent Mongstad refinery provides the flue gases for testing CO₂ capture, i.e. flue gas from the Combined Heat and Power (CHP) plant at 3.6-4 % CO₂, and flue gas originating from the Residual Fluidized Catalytic Cracker (RFCC) unit at 13-14 % CO₂. The typical gas compositions are also shown in Table 1. TCM has the flexibility to enrich the CHP flue gas by recycling captured CO₂ or to dilute the RFCC flue gas with air to adjust the CO₂ content.

Once the flue gas enters the TCM test site it is conditioned and saturated with water in a Direct Contact Cooler (DCC). The RFCC flue gas is also passed through a highly effective Brownian Diffusion (BD) filter in order to remove aerosols and catalyst particles. Conditioned

flue gas enters the bottom of the absorber where it is contacted with amine solvent as gas and solvent flow counter-currently through three sections of structured packing (12, 18 or 24 meters at TCM discretion). CO₂ in the flue gas reacts with the amine to become chemically bound in the solvent. The rich solvent is pumped through the lean/rich cross heat exchanger for pre-heating by hot lean solvent before it enters the stripper section. TCM amine plant is equipped with two strippers, one for operation with low CO₂ content (CHP stripper) or higher CO₂ content (RFCC stripper). Additional heat is supplied by steam to the stripper reboiler in order to reverse the absorption reactions and release CO₂ from the solvent. The regenerated lean solvent leaving the stripper is cooled down in the lean/rich cross heat exchanger and lean cooler, before it is recirculated back to the absorber. The depleted flue gas leaves the top of the absorber after being conditioned in one or two absorber water wash sections, while CO₂ product gas is released to the atmosphere through the stripper water wash and stripper overhead condenser system, as illustrated in Figure 1.

The feed gas type and corresponding CO₂ and O₂ concentrations for the entire test period (2017-2018) are shown in Figure 2, whereas the operating temperatures are plotted in Figure 3. Oxygen scavenger (potassium bisulfite) has been injected in the early periods of the campaign (see Figure 4).

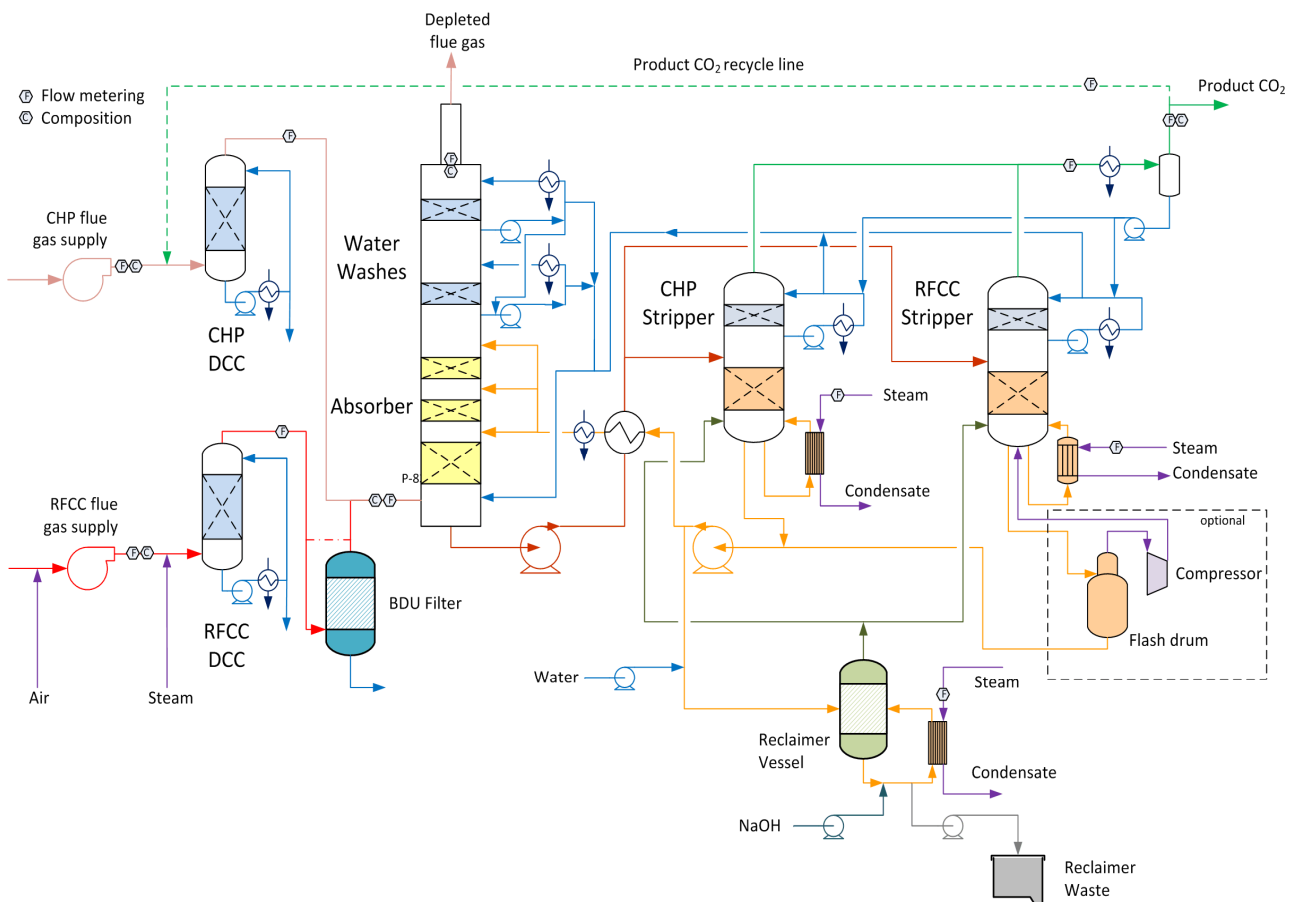


Figure 1: Simplified process diagram of the TCM amine plant (from [1])

Table 1: Typical flue gas compositions (adapted from [1])

Gas composition	Conditioned CHP gas	Conditioned RFCC gas
N ₂ (mole%)	73 - 79	73 - 79
O ₂ (mole%)	13 - 14	3 - 8
CO ₂ (mole%)	3.6 - 4.0	13.0 - 14.5
H ₂ O (mole%)	Saturated	Saturated
SO ₂ (ppmv)	<0.3	<5
NO _x (ppmv)	<5	100
NH ₃ (ppmv)	<5	<1
CO (ppmv)		<10
Particles (parts/cm ³)		0.3-0.8×10 ⁶

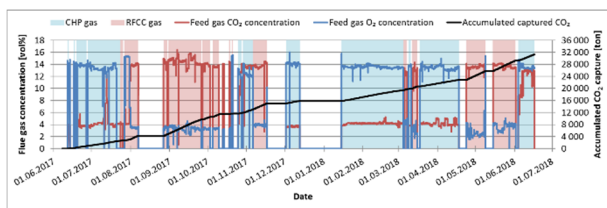
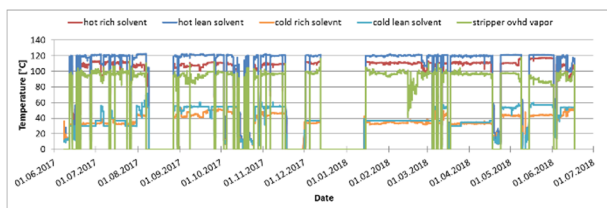
Figure 2. Flue gas sources and corresponding CO₂ and O₂ concentrations throughout the MEA test period (adapted from [1]).

Figure 3. Operating temperatures throughout the MEA test period (adapted from [1]).

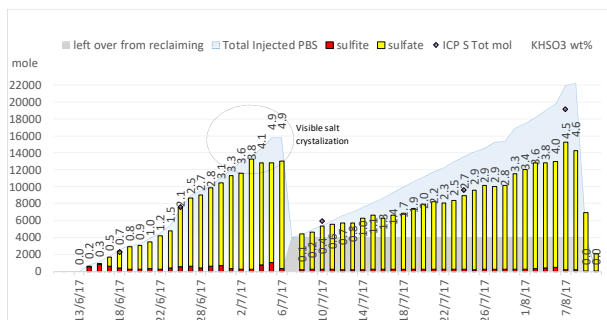
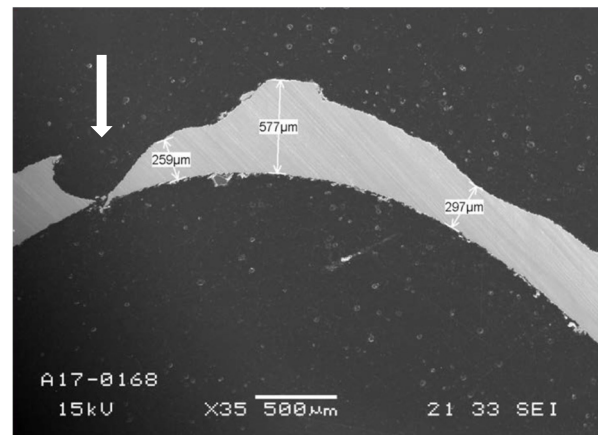


Figure 4. Accumulation of various species related to oxygen scavenger injection

2.2. Relevant findings

Examination of the reboiler after it was taken out of service revealed that the failure consisted in two holes on the heat exchanger plates built in AISI 316L stainless steel. It was observed that plate thickness in the affected area was reduced by about 200-250 μm as shown in

Figure 5; the failure might thus have resulted from a corrosion/erosion origin in the context of apparently increased general corrosion.

Figure 5. Cross-section SEM image of the reboiler plate region around the leak indicated by the arrow; original thickness 600 μm (adapted from [1]).

Plant inspections did not reveal any sign of corrosion elsewhere in the plant. However, a red/brown layer believed to be corrosion product deposits originating from the CHP reboiler was observed on surfaces exposed to the solvent, e.g. solvent piping and material test racks, process valves, CHP stripper and internals and CHP reboiler.

The period of oxygen scavenger injection (see Figure 4) appears to be closely related to the corrosion issues leading to the failure of the reboiler. The evolution of the chemical composition of the solvent within the scavenger injection period is highly relevant in this context. Rapid changes in metallic cation concentration (iron, nickel, chromium and molybdenum see Figure 6) were clear indications of on-going corrosion at that time, as well as likely precipitation of iron-based products, as observations previously suggested.

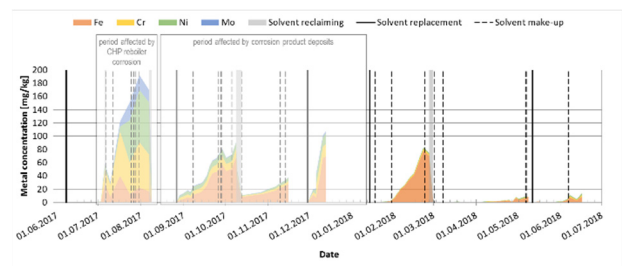


Figure 6. Metal cation concentrations during the MEA test campaigns (adapted from [1]).

Precipitation of solids (sulfates) has also been observed in the period of scavenger injection, this is suspected to have caused local flow restrictions that may have been a relevant factor supporting a flow enhanced damage, possibly in combination with the corrugated shape of the plates.

As part of a material selection assessment [1] corrosion coupons of different metals (including 316L stainless steel, bent) were placed in key locations within the solvent circuit. In each location there were two coupon racks positioned towards the top (upper) and bottom (lower) of the pipe. Analysis of these coupons also provided additional useful feed-back on the possible origins of the corrosion failure.

Among all corrosion coupons locations (hot and cold lean solvent, hot and cold rich solvent, stripper overhead) the 316L stainless steel specimens only showed signs of corrosion in the hot rich solvent (see Table 2). No corrosion was observed on stainless steel coupons exposed to the hot lean solvent. The carbon steel specimens were completely corroded in several locations, indicating generally high intrinsic corrosivity of the solvent.

Table 2. Corrosion rate of carbon steel (CS235) and stainless steel (SS 316L) coupons based on 7420 hours exposure to solvent (from [1])

Location	Steel type	Corrosion rate (mm/y)	
		upper	lower
Hot lean solvent	CS235	>5.91	>5.95
	SS 316L	0.00	0.00
Hot rich solvent	CS235	>6.46	>6.33
	SS 316L	0.02	0.08
Cold lean solvent	CS235	1.62	1.56
	SS 316L	0.00	0.00
Cold rich solvent	CS235	>3.24	>3.25
	SS 316L	0.00	0.00
Stripper overhead	CS235	0.00	0.00
	SS 316L	0.00	0.00

The solvent composition and temperature conditions in this location have similarities with the region where the corrosion damage occurred. This suggests that elevated temperature and the solvent composition (either or both) may be related to increased general corrosion rate. The conditions in the hot rich solvent coupon rack location alone, however, did not cause corrosion of unacceptable severity. Other factors must have contributed to the corrosion of the reboiler plates. Flashing of CO₂, expected to occur in the reboiler, is likely such a factor.

It is notable that within the same specimen location (hot rich solvent) the bent stainless steel coupons had quite different corrosion behavior and appearance depending on their position (see Table 2, Figure 7; for more details see Flø et al [1]):

- the lower bent coupon had higher corrosion rate
- stainless steel coupons in the lower position showed a red coloration (identified as iron oxides)
- not all coupons from the same rack (or position) displayed the colored layer (bent stainless steel in the upper position did not, while other coupons in the upper position did; photos not shown)

These observations may suggest that:

- bent shape may have played a role (strain or flow related)
- the red-brown deposit layer may not have played a significant role (some coupons with deposits did not corrode)

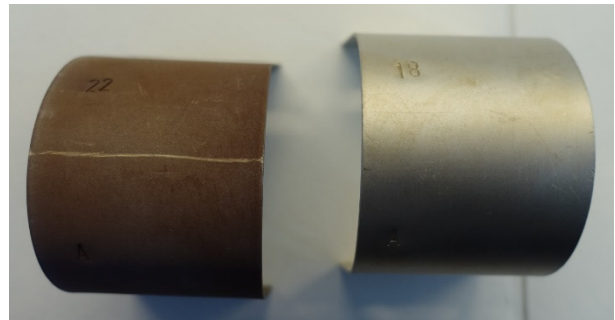


Figure 7. Appearance of bent stainless steel coupons after exposure in the hot rich solvent location. Coupon from the lower position on the left (adapted from [1]).

Solids precipitating (sulfates) in the period of scavenger injection did not appear to affect the corrosion coupons considerably. Deposition within the reboiler plates causing local flow restrictions, however, may be a relevant factor.

An important detail of the reboiler plates is their corrugated shape. The flow regime is expected to be different in the space between the plates and this shape may provide locations for impingement to occur. This may be a relevant factor both in the presence of abrasive particles (erosion) and in their absence.

2.3. Main hypotheses and scope of experimental work

From a detailed consideration of the collected data, two distinct scenarios and a combination of the two have been explicitly considered when forming the main hypotheses to guide the planning of the experimental testing.

1. Erosion. This hypothesis considers that erosion is both necessary and sufficient to cause the failure, the corrosivity aspect being irrelevant for this scenario. Erosion can remove the passive film from the stainless steel surface and even the regular operation conditions may have been corrosive enough to attack the unprotected stainless steel. In principle erosion could be severe enough to abrade the steel as well, not only the passive film.

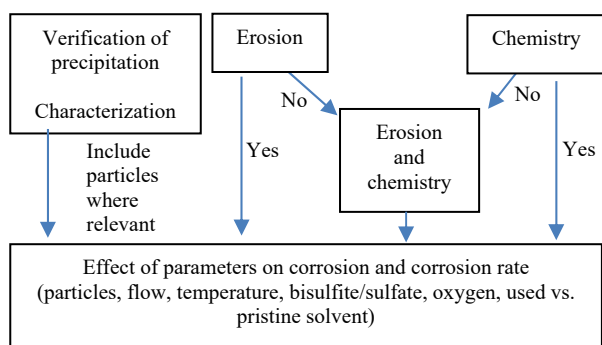
2. Enhanced corrosivity. According to this hypothesis the specific chemistry in the scavenger period can cause depassivation and sustain a considerably high corrosion rate, probably in combination with some depassivating effect of the amine or its degradation products. The erosion aspect would be in practice irrelevant in the context of this scenario.

3. Erosion and enhanced corrosivity. Erosion could remove passivity, but regular conditions would not be enough to prevent rapid re-passivation to sustain corrosion. The specific chemistry in the scavenger period could sustain corrosion in the absence of a passive film but would not be able to de-passivate the surface. Enhanced corrosivity could de-passivate and maintain a certain corrosion rate, but not high enough to lead to rapid failure.

The main working hypotheses delineated above form the basis for defining the scope of the experimental validation work, as outlined in the diagram below.

Precipitation of solids has been observed, yet there is a lack of information about its nature and properties. Verifying the precipitation and characterization of the solids are therefore considered an important part of the scope.

More specific aspects are discussed within the Experimental approach section.



5. Experimental approach

5.1. Apparatus

It is important at the outset to distinguish between two possible modes of operation when it comes to experimental design. The real-life conditions in the region of the reboiler where the failure has occurred are thought to be represented by a transition between partly stripped rich solvent and lean solvent. It is a dynamic scenario where the system may not reach thermodynamic equilibrium within the residence time of the solvent flowing through. Studying such a system experimentally can be done in two ways:

- dynamic (flowing type) experiments mimicking closely the plant process (including continuous feeding and removal of solvent); this type of experiments may be a better representation of the conditions in the reboiler, but those specific conditions would at the same time be difficult to assess due to continuous transitions.
- static (batch type) experimental series where one set of conditions is implemented, variations are studied by parametric studies (test matrix); for this approach thermodynamic equilibrium can be reached, the conditions are known with higher level of certainty, making correlations between parameters and results

more robust. The static mode is preferred for most of the planned experiments.

The experimental setup is to be built around a closed autoclave that can be operated under the desired pressure and temperature conditions. The setup is designed to be modular, allowing for both flowing and batch type of experiments.

A key consideration of the test setup was that it should be flexible enough to allow specimens to be exposed to a combination of the key factors (temperature, pressure, flow, action of suspended solids, specific chemical environments) and ideally allow in situ electrochemical measurements to be carried out. A glass reactor allows visual observation of precipitation and efficient suspension of solid particles.

Figure 8 presents a graphical representation of the envisioned test autoclave setup, where two types of specimens can be exposed, possibly in all experiments:

- impeller blade specimen: mounted in special holders that allow rectangular specimens to be affixed to the end of a rotator shaft. The specimen will act as impeller blade which will provide enhanced flow, shear, entrainment of suspended solids and possibly entrainment of bubbles. The specimen will be electrically isolated from any metallic parts (e.g. the shaft itself) to avoid galvanic coupling
- stationary specimens placed to receive impinging flow from the impeller; outfitted with electrical connections for electrochemical measurements



Figure 8. Sketch of the reactor with specimens placed as both impeller blades and stationary

5.2. Test conditions

The “history” of the solvent may render its chemical composition greatly different from pristine solvent. Comparison of the stainless steel behavior in clean and

used solvent from TCM may reveal whether these differences have a detrimental effect on the passivity of the steel or may affect other aspects (e.g. solubility and precipitation). It is therefore envisioned that experiments will include both pristine and used solvent.

The majority of the experiments will fall within a parametric study performed in batch mode. The test system will reach equilibrium within the timeframe of the experiments, yet can initially undergo transitions, e.g. when starting with rich solvent and driving the conditions toward lean or an intermediate state of the solvent. An advantage implied is that the thermodynamic (and, to a certain extent redox) behavior of the solvent system can be predicted, helping technical planning and execution of the tests as well as interpretation of the results.

The key parameters considered/studied to be included within the scope of the experimental work are listed below:

Temperature: in the range relevant to rich-lean transition (around 120 °C), extensions to lower and higher values for sensitivity.

Solvent and CO₂ loading: both pristine and used solvents to be included, both rich and lean may be relevant starting solutions. Aspects of chemical composition related to solvent history (i.a. degradation products) are implicit.

Oxygen and scavenger: these species are of special relevance and the impact of their reaction product (sulfate) as well as the excess of one species over the other is to be included

To gain a better understanding of how environmental parameters will affect the studied system of equilibria, a series of scenarios have been preliminarily modelled using the OLI Studio software where the composition and speciation as a function of temperature at a given total pressure were predicted.

CO₂ loading of lean solvent

It is envisioned that initial loading of pristine lean solvent shall be performed by continuous purging with pure CO₂ at low temperature and ambient pressure. Model calculations presented here are based on 30 mass% MEA and the reaction below. Dissolution of CO₂ into the aqueous phase is also accounted for. Reaction kinetics are not considered. The case presented here as an example models the CO₂ loading process at 40 °C.



To be noted that used lean solvent will likely not be completely stripped of CO₂. It is expected, however, that reloading by this procedure would yield the same final conditions as loading of pristine lean solvent.

Figure 9 shows the distribution of species during the loading process modelled at 40 °C.

Up to an uptake of ca. 0.4 mol CO₂ / mol MEA all added CO₂ is predicted to be bound by the solvent (see Figure 9). As more CO₂ gas is purged through the solvent, the

bound fraction continues to increase while some of the CO₂ is being retained as dissolved CO₂ (see lower panel in the same figure). After ca. 0.6 mol CO₂ / mol MEA has been added, the dissolved CO₂ will reach saturation and all further addition will escape into the gas phase while the speciation appears to remain constant. This gives a predictable and reproducible state with a constant chemical composition. Performing the loading process at lower temperatures will ensure that the composition will not be significantly altered by e.g. evaporation, resulting in good consistency.

Should it be considered necessary to perform experiments starting with a particular loading level, it can be achieved by mixing the loaded stock with pristine lean solvent in the required proportion.

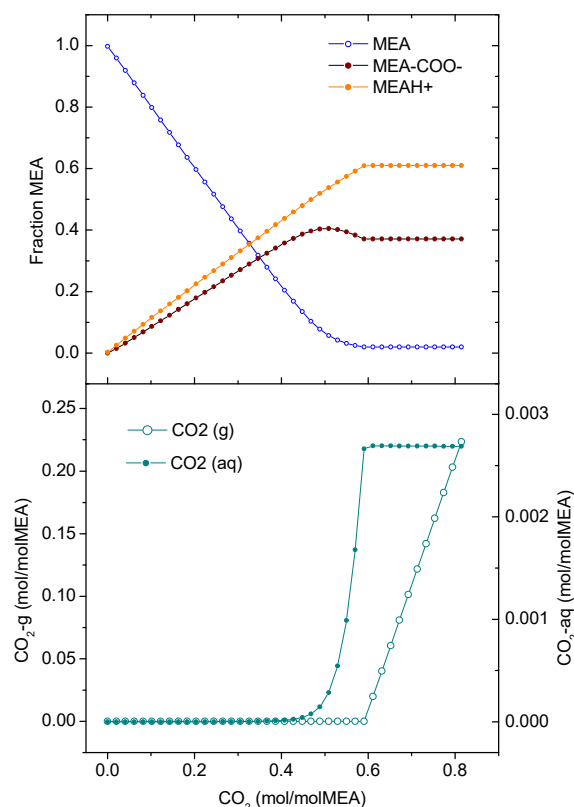


Figure 9. Plot of the evolution of selected species during loading of MEA by purging with pure CO₂ at 40 °C and ambient pressure

Isobaric heating of rich test solutions

It is expected that during temperature ramping the solution composition will show a transient, reaching the new equilibrium after some time at the target temperature depending on the kinetics of the processes involved. This implies that testing in a closed reactor at fixed conditions (batch testing) will give results where equilibrium can be assumed to have been established within the timeframe of the experiment. In a continuous flow system, in contrast, a continuous transition may be envisioned (rich solvent continuously fed, lean solvent removed, flashing of CO₂).

To understand the behavior of the test solutions upon heating to a high target temperature, the process was modelled using the OLI Studio software starting with a fully loaded rich solvent (generated as described in the previous section and ramping the temperature from 40 °C to 120 °C while maintaining the reactor vessel pressure at 2 bar.

As shown in Figure 10, increasing the temperature leads to the gradual reversal of the loading process, releasing more and more CO₂. It is to be noted that for each given temperature a new equilibrium state can be reached characterized by a distinct distribution of species. Evidently, care must be taken to release the gas-phase CO₂ to avoid excessive pressure buildup in the vessel. To illustrate this, the bubble point of three solutions was plotted in Figure 11 as the temperature was raised to 120 °C as follows:

- Solvent loaded with CO₂ at 40 °C and ambient pressure (as described in section 0)
- Solvent at equilibrium at 80 °C and 2 bar
- Solvent at equilibrium at 110 °C and 2 bar

While it is not expected that the pressure would rise to the bubble point value (since there is always a gas pocket present in the reactor), considerable pressure can build up if the released CO₂ is not vented.

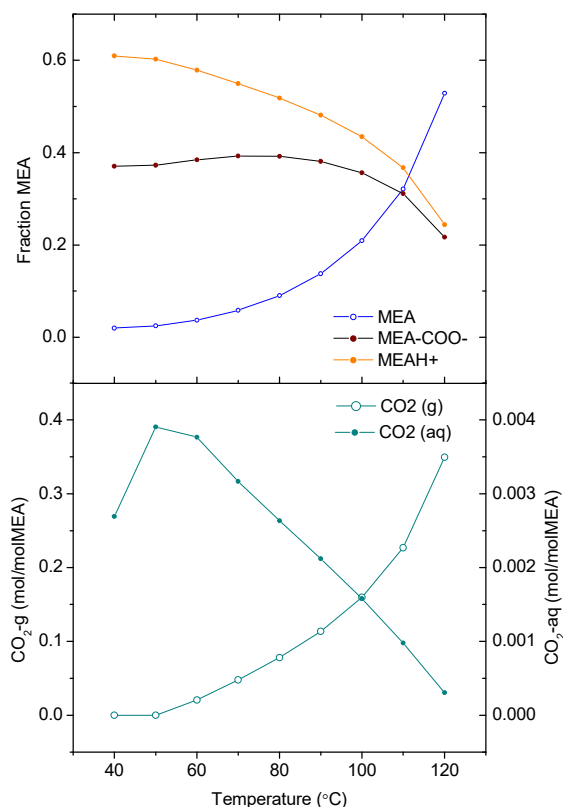


Figure 10. Plot of the evolution of selected species as a result of increasing the temperature of a solution prepared as described in the text

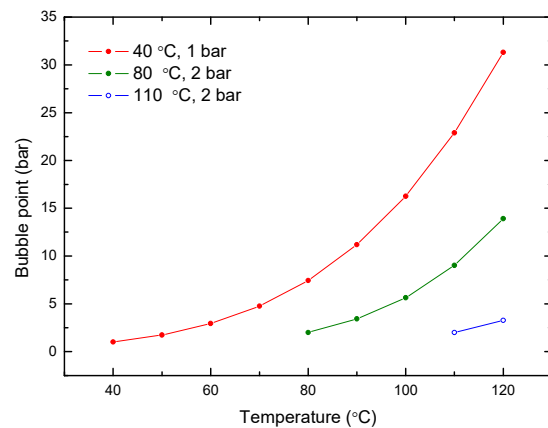


Figure 11. Plots of the bubble point as a function of increasing temperature for three different starting solutions

Addition of oxygen and oxygen scavenger

The modelling results indicate that addition of KHSO₃ as oxygen scavenger is expected to affect the speciation in the solution as it will lower the pH. Shown in Figure 12 are results of model calculations for various concentrations of added potassium bisulphite. The reaction with dissolved O₂ was taken into account in these calculations. It is predicted that the equilibrium composition of the test solutions will change as a function of the added scavenger concentration for any given temperature. In other words, for a given test temperature and pressure the composition will be different in the presence and the absence of the oxygen scavenger.

Starting solutions will be prepared by first adding the prescribed quantity of KHSO₃ to the lean solvent, followed by purging the solution with air until the scavenger-oxygen reaction is complete, to yield the desired level of sulphate in solution. This will be performed at low temperature to avoid excessive evaporation and have an increased O₂ solubility. The solvent will then be loaded with CO₂; this process is expected to effectively purge out excess dissolved O₂.

For experiments with excess bisulphite it can be dosed as desired to the resulting enriched solvent.

For experiments with excess O₂ the solution can be purged with air or a gas mixture at low temperature to yield the desired dissolved O₂ concentration at equilibrium while avoiding removal of bound CO₂. Dissolved oxygen will not remain constant during testing; flashing of CO₂ upon heating may remove dissolved O₂. If it is desirable to maintain O₂ in solution purging with air or gas mixture can be applied during tests as well.

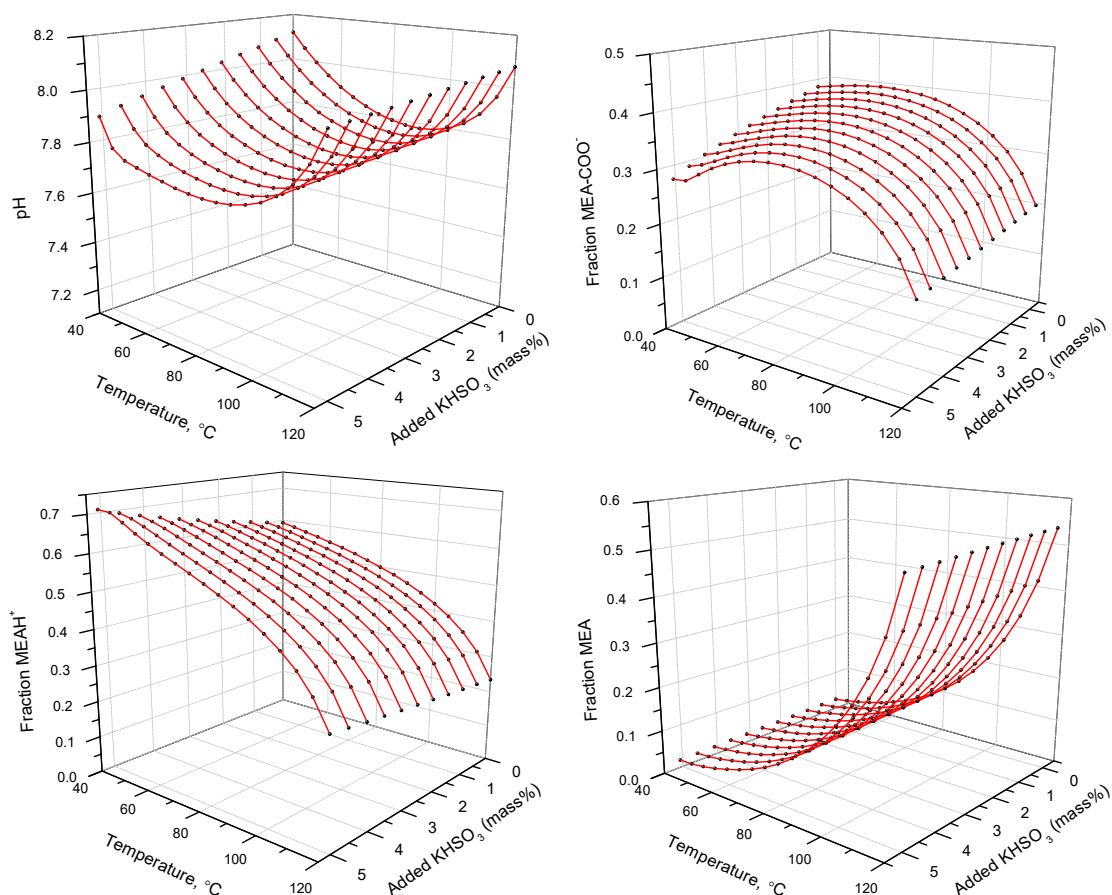


Figure 12. Plots of the pH and concentration of selected species at equilibrium as a function of temperature and added oxygen scavenger

5.3. Abrasive solids

Should precipitation studies result in a reproducible way to generate solids in situ, it shall be implemented for the tests involving an erosion aspect. If in situ generation of particles is not successful, erosion-corrosion tests can be performed using inert solids, e.g. silica (sand) particles. These may be expected to be harder and more abrasive than precipitating particles.

6. Way forward

The outlined experimental work (Phase 2 of this collaborative effort) will be carried out as a systematic parametric study at the IFE laboratories closely coordinated with TCM, Total and Equinor. The effects of erosion and corrosion (as well as synergies thereof) will be distinguished by means of judicious testing of the main hypotheses, i.e. within the scopes they define, as described in Section 2.3; experiments targeting a given hypothesis will be carried out with focus on the particularities related to that hypothesis (erosion – suspended solids, enhanced corrosion – chemical composition, etc.). It is expected that the majority of experiments will be carried out in batch mode, with selected conditions tested in flowing mode as well. The actual test matrices are to be adapted constantly as needed in view of the obtained results. The results,

interpretations and conclusions regarding the working hypotheses will be reported in a separate publication.

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