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Lowering Absorption process **UNcertainty, risks and **C**osts by predicting and controlling amine degradation**

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DORA: A Novel Technology for Lowering Solvent Degradation

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Executive summary

Amine solvents used for post combustion CO₂ capture mainly degrade through three main mechanisms – oxidative, thermal and CO₂ induced degradation [1]. Oxidative degradation of amines occurs in the presence of oxygen and normally at temperatures below 100°C. This mechanism leads to the formation of a variety of undesirable products including ammonia and acidic compounds that react further to form Heat Stable Salts (HSS) [2]. Thermal degradation occurs at temperatures higher than 100°C and results in the formation of heavier degradation products [3]. In the third mechanism, primary and secondary amines react to form oxazolidones and diamines in the presence of CO₂ [1]. Solvent degradation leads to several adverse impacts on the overall capture process including loss of solvent capacity which leads to the need for solvent replenishment to maintain capture rates [4]. The solvent quality also deteriorates in terms of change in physical properties such as viscosity and density. Corrosivity of the solvent also increases leading to accumulation of metals like iron, nickel and chromium in the solvent [5], [6]. Solvent degradation, therefore, leads to an overall negative impact on the economics and environmental impact of amine based post combustion CO₂ capture.

To mitigate the impact of solvent degradation and accelerate the wide scale implementation of amine based CO₂ capture, it is critical to control degradation of solvents. This can be achieved through the use of solvent management strategies. Within LAUNCH-CCUS, several solvent management strategies such as oxygen removal through nitrogen sparging and membranes, use of activated carbon and thermal reclaiming are being investigated. This report presents the data of the laboratorial experiments done with the DORA (Dissolved Oxygen Removal Apparatus) and TNO's ODIN (Oxygen Depletion Installation) with MEA, MDEA/PZ and CESAR1. During these experiments, two membranes were used: a commercial membrane from Liqui-Cell (with MDEA/PZ) and a dense layer membrane developed by NTNU (with MEA and CESAR1).

During the MEA experiments the operating conditions of the membrane such as liquid flow rate, gas flow rate and pressure (vacuum) were optimized. The most efficient operation was done with a combination of nitrogen as a sweep gas and vacuum on the outlet of the membrane. The use of vacuum made it possible to use significant lower amount of nitrogen in the sweeping gas. This is an important improvement of the process not only in terms of efficiency of removal but also OPEX related to the flow of nitrogen used during the operation. Another significant result was the fact that DORA was able to remove oxygen even for MEA in which degradation occurs in a fast rate. The tests with MDEA/PZ also showed the decrease in oxygen while using the DORA in the system for different CO₂ loadings. For both solvents, the oxygen content during the operation with DORA dropped to almost zero (or even zero in some cases) in less than 30 minutes. For CESAR1, it was not possible to quantify the impact of the DORA since one of the membranes was not compatible with the solvent and the other one showed a high rate of leakage when operated with CESAR1.

In conclusion, DORA is potentially a technically viable solution for controlling the degradation of CO₂ capture solvents and can be applied independently or in combination with other solvent management strategies such as reclaiming. However, some further development is still needed before the technology can be commercially available. TNO, NTNU, SINTEF and RWE are working in a proposal called MeDORA (project approved in ACT4 scheme) to ensure that the DORA technology will be ready for implementation by 2026.



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1 Introduction

1.1 Solvent Degradation

Amine solvents used for post combustion CO₂ capture mainly degrade through three main mechanisms – oxidative, thermal and CO₂ induced degradation [1]. Oxidative degradation of amines occurs in the presence of oxygen and normally at temperatures below 100°C. This mechanism leads to the formation of a variety of undesirable products including ammonia and acidic compounds that react further to form Heat Stable Salts (HSS) [2]. Thermal degradation occurs at temperatures higher than 100°C and results in the formation of heavier degradation products [3]. In the third mechanism, primary and secondary amines react to form oxazolidones and diamines in the presence of CO₂ [1]. Solvent degradation leads to several adverse impacts on the overall capture process including loss of solvent capacity which leads to the need for solvent replenishment to maintain capture rates [4]. The solvent quality also deteriorates in terms of change in physical properties such as viscosity and density. Corrosivity of the solvent also increases leading to accumulation of metals like iron, nickel and chromium in the solvent [5], [6]. Solvent degradation, therefore, leads to an overall negative impact on the economics and environmental impact of amine based post combustion CO₂ capture.

To mitigate the impact of solvent degradation and accelerate the wide scale implementation of amine based CO₂ capture, it is critical to control degradation of solvents. This can be achieved through the use of solvent management strategies. Within LAUNCH-CCUS, several solvent management strategies such as oxygen removal through nitrogen sparging and membranes, use of activated carbon and thermal reclaiming are being investigated.

1.2 Dissolved Oxygen Removal Apparatus (DORA)

Oxidative degradation of amines can be controlled by removal of oxygen from the solvent. This can be done through the use of oxygen scavengers in the solvent, nitrogen sparging or membranes. TNO has developed and patented DORA (**D**issolved **O**xygen **R**emoval **A**pparatus), an online and non-invasive membrane based solvent management tool. DORA operates by using membranes that remove dissolved oxygen from amines. The driving force for this removal can be provided either by a sweeping gas in combination with vacuum or a liquid with an oxygen scavenger as shown in Figure 1 [7]. The use of DORA allows for oxygen removal without the addition of any chemicals to the plant, thereby avoiding issues such as foaming and additional undesirable reactions [8], [9].

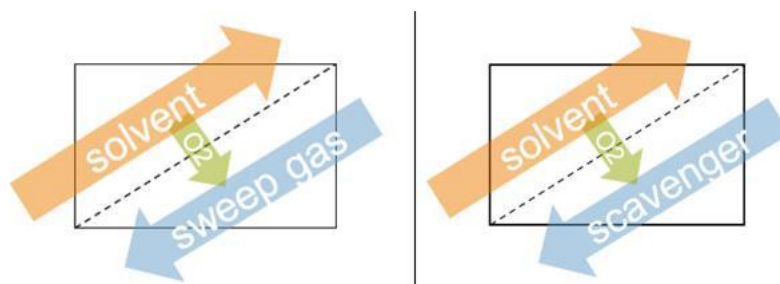


Figure 1 - Working principle of DORA in the L-G and L-L modes

So far TNO has tested a commercially available porous membrane from 3M and a coated dense layer membrane developed by NTNU [7]. The commercial porous membrane has been tested by TNO at TRL6 in combination with TNO's mobile capture unit, Miniplant using 30 wt% monoethanolamine (MEA) at an industrial facility in Rotterdam, The Netherlands. DORA was integrated in the miniplant after the absorber sump as shown in Figure 2. Further tests done as part of a Dutch National project called DECIPHER brought the technology to TRL7 by operating it at HVC (Waste to Energy plant in The Netherlands) treating the entire solvent flow (an aqueous blend of N-methyl diethanolamine, MDEA and piperazine, PZ).

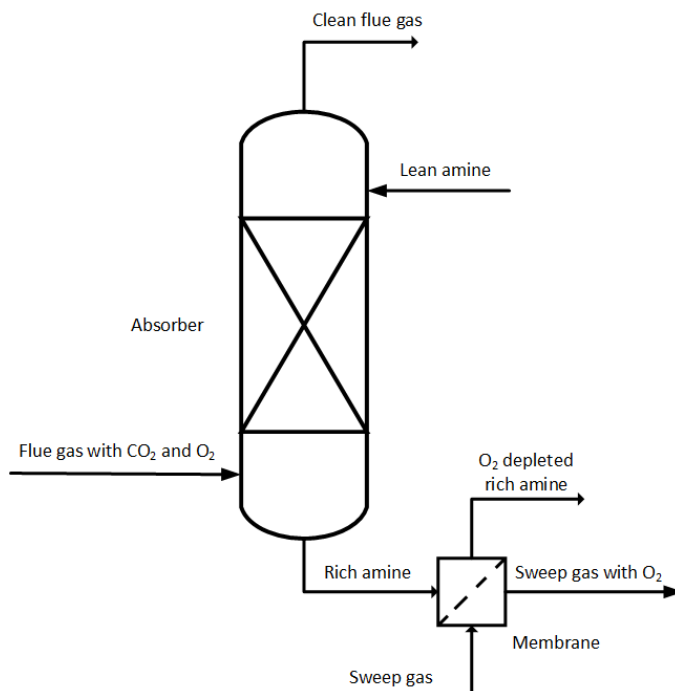


Figure 2 – Connection scheme of DORA with TNO's Miniplant and HVC's pilot

The smaller dense layer membrane has been tested by TNO at the laboratory scale. This has been done in combination with TNO's Oxygen Depletion Installation (ODIN) shown in Figure 3. The setup consists of two closed jacketed vessels of volume 0.67 liters each. The vessels are connected to a water bath, that allows for precise temperature control. The temperature of the solvent is measured with a type K thermocouple. The solvent is stirred continuously with a magnetic stirrer. The oxygen decay in the solvent is measured using an oxygen sensor from Endress+Hauser (Memosens COS81D). This sensor is capable of measuring oxygen in the range of 0.004mg/l to 30 mg/l. The sensor accuracy is 0.5 mg/l in the operating ranges used for testing DORA [10]. This sensor has been validated against two other sensors - VWR pHenomenal® OX-4100 galvanic sensor and a HI-5421 dissolved oxygen and BOD meter from Hanna Instruments. In addition to this validation, the sensor has also been validated against literature and exhibits good agreement with all three validation methods [9].

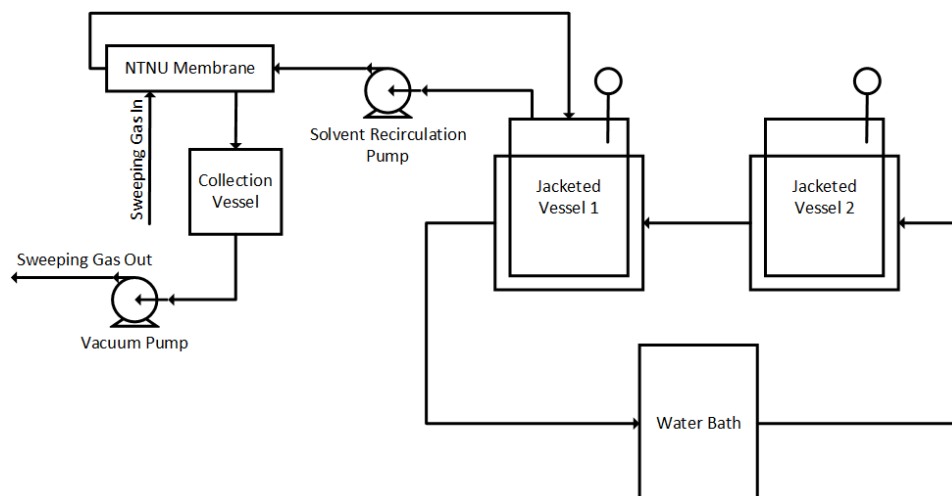


Figure 3 - Scheme for testing DORA with TNO's Oxygen Depletion Installation (ODIN)

1.3 Challenges with Porous Membranes

When DORA was tested at both TRL6 and TRL7 at industrial facilities, operation of the CO₂ capture plant was done with and without the use of the DORA to assess its impact. The effectiveness of DORA was determined through the formation of degradation products in the solvent. Samples were taken throughout the campaigns and analyzed for degradation products like formic acid. It was observed that when DORA was online, the formation of formic acid stabilized and increased significantly when DORA was offline. This impact can be seen in Figure 4 when DORA was operated at PlantOne [7]. The yellow rectangle indicates the period in which DORA was operational. It should be emphasized that the DORA operation wasn't continuous – in fact, in the period indicated, the DORA availability was ca. 37%. Nevertheless, the graphs show that the operation of DORA has a clear impact on the degradation of MEA, controlling the composition of the key oxidative degradation products. From the start of the campaign until almost 2500 h, a steady increase in the concentrations of formate, acetate and oxalate is observed. Then, for about 700 h, the concentrations remain constant. And after DORA is by-passed, the concentrations start to increase rapidly. This campaign showed that DORA is an effective solvent management strategy that can control the accumulation of degradation products in the solvent and therefore mitigate the impact of oxidative degradation of amines.

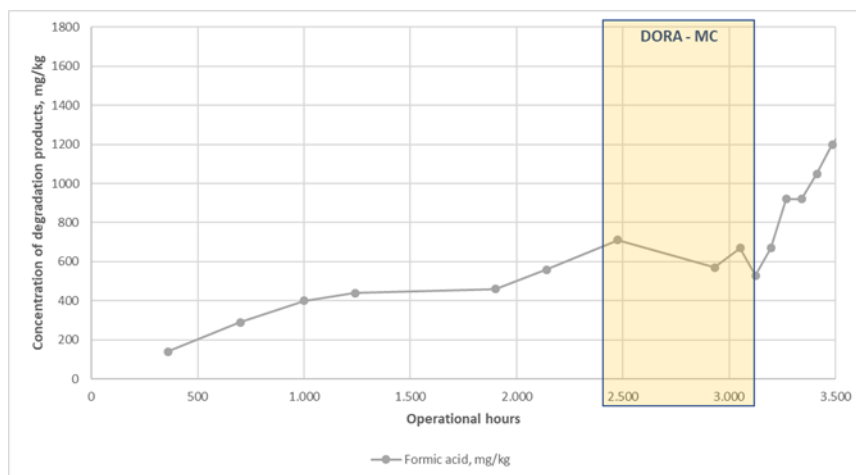


Figure 4 - Effect of DORA on formic acid formation

During the end of the aforementioned campaign, however, it was observed that the membrane efficiency reduced overtime. This was attributed to amine leakages through the membrane. The resulting pore wetting led to membrane damage and overall reduction in removal efficiency. To mitigate the impact of membrane leakages, TNO and NTNU investigated the benefits of using a dense layer membrane, initially developed by NTNU to capture CO₂ from flue gases [11]. This membrane has a porous support on which a polymeric material is coated. This coating prevents amine leakages through the membrane while still allowing oxygen to pass through. As part of the deliverable 2.1.1 *Membrane Selection for DORA*, The LAUNCH partners decided to further investigate the application of this dense layer membrane for oxygen removal from amines in a lab scale environment.

1.4 Solvents Tested

DORA has been developed as a solvent- and flue gas independent technology i.e. it can be applied as solvent management strategy in capture plants irrespective of the solvent being used and flue gas source. This feature of DORA is being illustrated and demonstrated within LAUNCH-CCUS. Oxygen removal with DORA has been tested for different solvents within this project along with determination of optimized operating conditions for each system. The solvents tested were:

- 30 wt% monoethanolamine (MEA)

- CESAR1 (27 wt% 2- Amino-2-methylpropan-1-ol (AMP) and 13 wt% piperazine (PZ))
- 42 wt% Methyl diethanolamine (MDEA) and 8 wt% piperazine (PZ)

DORA was tested for the aforementioned solvents at the laboratory scale in the ODIN (Oxygen Depletion Installation). To determine the efficacy of DORA, the rate of oxygen depletion is measured with and without DORA. Optimized operating conditions are obtained by varying liquid and sweep gas flow rates, as well as the pressure (application of vacuum on the permeate side). The tests were also carried out for several CO₂ loadings and temperatures.

The solvents chosen for these experiments are open solvents in use either at a commercial scale (MEA, 1st generation) or at pilot scale (CESAR and MDEA/PZ, 2nd generation) for post-combustion capture. According to the original LAUNCH proposal, slow bicarbonate forming solvents were supposed to be tested at RWE as part of WP5. This plan was modified so that a longer operation of CESAR1 could be possible. Accordingly, these class of solvents was also not considered in these experiments. However, as part of the Horizon Europe project REALISE, a strong bicarbonate forming solvent (HS-3, a blend of 40wt% 1-(2-Hydroxyethyl)-Pyrrolidine and 15wt% 3-Amino-1-propanol) is under demonstration. The investigations will include the application of DORA. The results are expected to be published in 2024.

1.5 Description of Membranes

The two membranes mentioned above will be described in this session. The porous membrane is a commercially available module from 3M. The membrane contactor module from Liqui-Cel™ SP Series (2.5 x 8 extra-flow model), consists of polypropylene hollow fibers with 25% porosity [12]. This membrane can be seen in Figure 5.



Figure 5 - Porous membrane for oxygen removal

On the other hand, NTNU's dense layer membrane uses a porous polypropylene (PP) support (Celgard® 2400). This support has a thickness of 25 µm and porosity of 41% [13]. Fluorinated polymers were chosen for the dense layer coating since they provide chemical stability towards various amines and also provide high CO₂ selectivity over the amines [14]. The coating was carried by dip coating and a coating thickness of 1.7 ± 0.3 µm was achieved [11]. The dense layer is placed in contact with the amine solvent flowing on the shell side of the membrane and the sweep gas flows counter currently on the tube side. The total interfacial area of this membrane is 8.1cm². The dense layer membrane can be seen in Figure 6.



Figure 6 - Dense layer membrane for oxygen removal

1.6 Experimental Methodology

To determine the extent of oxygen removal through DORA, tests with and without the membrane were performed in the ODIN, Figure 3. The solvent is either tested without CO₂ loading or it is loaded with a predetermined amount of CO₂. The CO₂ loading is measured using an ATR-FTIR. The temperature was controlled by means of a water bath, between 40°C and 60°C. Variations in CO₂ loading and temperature allows estimate reaction kinetics at relevant process conditions. The loaded solvent is charged to the vessel and allowed to reach the selected operating temperature. After reaching the desired temperature, the solvent is loaded with oxygen. When the oxygen concentration stabilizes, the vessels are filled to the top, so that no gas head space is left, and the oxygen sensor is introduced to the vessel. Special attention must be paid to potential trapping of bubbles on the sensor tip. To avoid measurement errors caused by such bubbles, the sensor is taken out and reinserted in the vessel. The vessel is sealed with a plug. The oxygen is then allowed to decay in the solvent over time. These experiments provide a baseline of oxygen consumption against which the performance of DORA can be assessed. The entire ODIN setup is placed inside a glove box purged with nitrogen to avoid any potential leak of oxygen into the vessel.

In the second set of experiments, DORA was attached to the vessel. The solvent is circulated over the membrane by means of a pump. The selected sweep gas flows counter currently on the permeate side of the membrane. In these tests, the oxygen is removed from the solvent by means of the membrane but is also consumed via the oxidative degradation reactions. Therefore, the tests without the membrane (oxygen decay only due to reaction) serve as zero tests and allow us to assess the effect of the membrane in isolation. In some tests, the gas outlet of the membrane was also connected to a vacuum pump to enhance the driving force and reduce the flow of the sweep gas. All tests were performed in duplicates to assess repeatability.

2 DORA Testing

2.1 Tests with 30wt% Monoethanolamine (MEA)

To date 30 wt% MEA is considered to be the benchmark solvent for amine-based post combustion CO₂ capture [15], [16]. However, testing campaigns at various pilots have reported losses up to 1.5 kg MEA/ton CO₂ captured [6], mostly due to oxidative degradation.

Baseline tests were carried out to measure the decay of oxygen in 30 wt% MEA at different CO₂ loadings and temperatures to reflect absorber conditions. DORA tests were then carried out at the same CO₂ loadings and temperatures to assess the extent of oxygen removal. To properly observe the decay of oxygen in MEA since the reaction is very fast, the experiments were carried out with pure oxygen. These laboratory tests were carried out with the dense layer membrane. An overview of the experimental conditions for baseline tests and DORA tests have been provided in Table 1 and Table 2, respectively.

Table 1 – Experimental matrix for baseline tests with 30 wt% MEA

Experiment ID	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
1	0	40
2	0	50
3	0	60
4	0.24	40
5	0.27	40
6	0.4	40
7	0.5	40

Table 2 - Experimental matrix for DORA tests with 30 wt% MEA

Experiment ID	Gas Flow Rate (NI/h)	Gas Outlet Pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
8	100	1.01	0	40
9	50	1.01	0	40
10	25	1.01	0	40
11	1	0.85	0	40
12	50	0.6	0.27	40
13	50	0.6	0.5	40

2.1.1 Baseline Tests without DORA (Experiments 1- 7)

To establish baseline oxygen consumption in 30 wt% MEA a series of tests with varying CO₂ loadings and temperatures were carried out. In experiments 1 to 3, oxygen decay was observed in unloaded 30 wt% MEA at different temperatures between 40°C and 60°C. It was observed that the rate of decay did not increase significantly when increasing temperature. The amount of dissolved oxygen decreased with increasing temperature, as expected. These results can be seen in Figure 7.

In the next set of experiments (4 to 7), oxygen decay was observed in MEA with CO₂ loading. These tests were carried out at different CO₂ loadings at 40°C. As shown in Figure 7, the introduction of CO₂ into the system significantly increases the rate of oxygen consumption. However, for the loading range investigated in these experiments, the rate of oxygen consumption is seen to be higher for lower loadings and decreases as the loading increases. This behaviour of loaded solutions has also been reported by Supap et al. [17]. This behaviour was attributed to salting out of oxygen when the amount of CO₂ in the solvent increases. However, Supap et al. also reported a higher rate of degradation for unloaded solvents as opposed to the trends observed in this work. In the work by Supap et al, the overall loss of MEA was measured over time as opposed to the oxygen decay. This work indicates that factors other than oxygen solubility have an impact

on the oxygen consumption rate. One could speculate that a different mechanism takes place once the solvent is loaded (MEA carbamates or protonated MEA could be more prone to degradation than molecular MEA). However, if this would be the case, one would expect the oxygen decay to increase with increasing loading, which is the opposite of what is observed. It should be reinforced that all experiments were executed – at least – in duplos, and the results presented in here are reproducible. At the moment, we cannot offer a fundamental explanation for these observations.

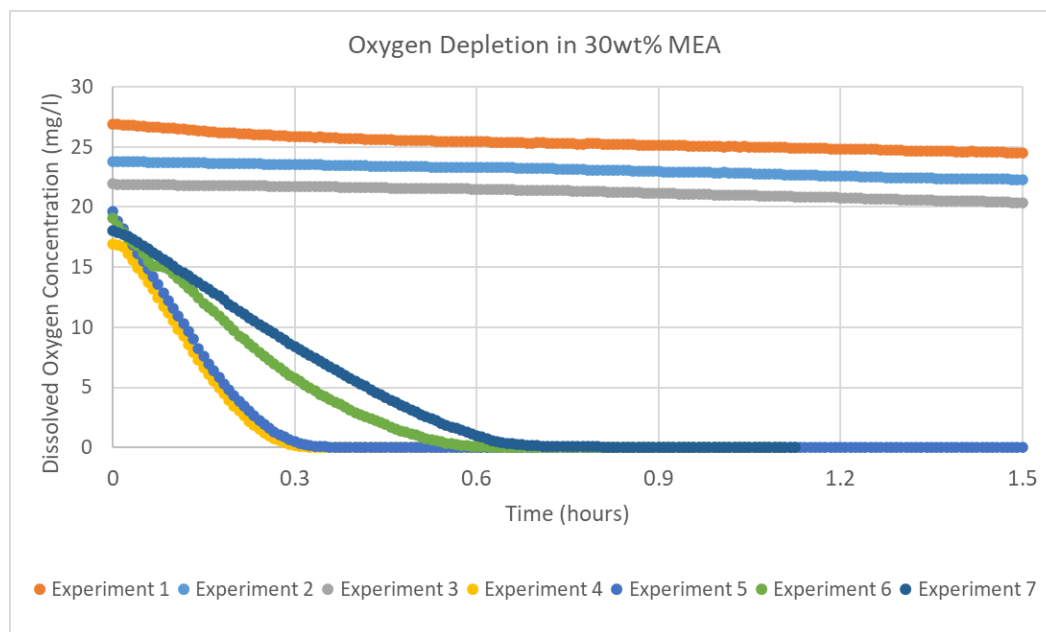


Figure 7 - Oxygen decay in 30 wt% MEA without DORA

2.1.2 DORA Tests with N₂ as Sweeping Gas (Experiments 8 to 10)

After baseline oxygen consumption was established for unloaded and loaded 30 wt% MEA, DORA tests were carried out with nitrogen as a sweeping gas. The membrane was tested previously to establish optimum operating conditions of liquid flow rate. The liquid flow rate was fixed at 3 l/h as determined by previous work done at TNO [7]. 30wt% MEA with no CO₂ loading was circulated over the membrane on the shell side. Nitrogen was used as a sweep gas and flowed counter-currently on the tube side. The nitrogen flow was varied from 25 NI/h to 100 NI/h. As shown in Figure 8, the gas flow rate does not have a significant impact on the amount or rate of oxygen decay. An overview of the operating conditions for these experiments is provided in Table 3.

Table 3 - Overview of operating conditions for Figure 8

Experiment ID	Gas Flow Rate (NI/h)	Gas Outlet Pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
1	-	-	0	40
8	100	1.01	0	40
9	50	1.01	0	40
10	25	1.01	0	40

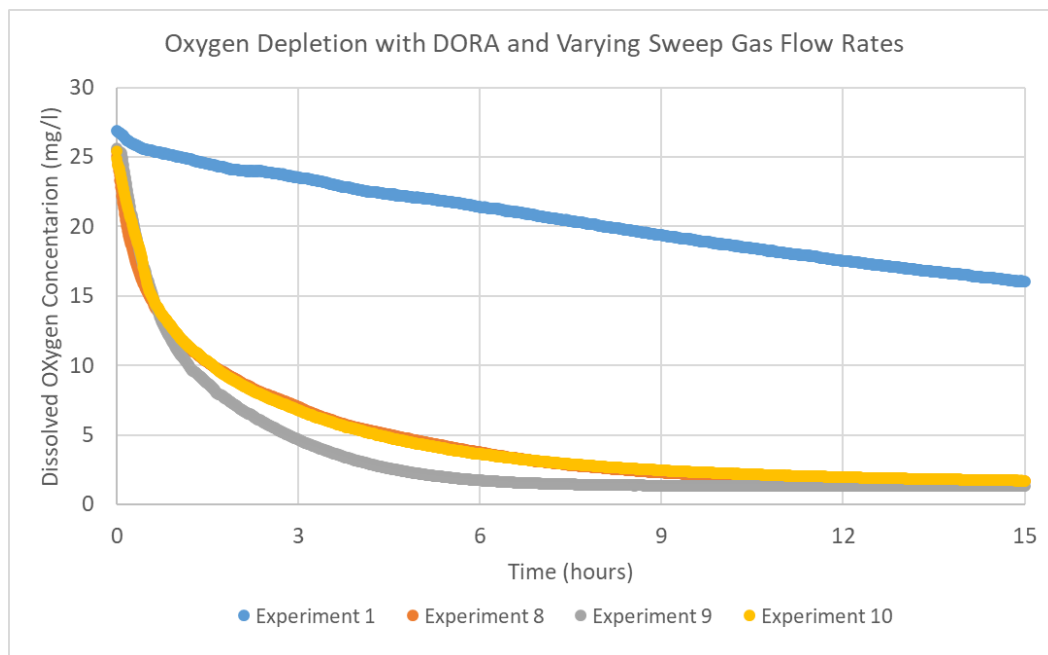


Figure 8 – Oxygen decay with DORA and varying sweep gas flow rates

2.1.3 DORA tests with Vacuum (Experiment 11)

Driving force for oxygen removal is provided by a sweeping gas. The amount of sweeping gas can be a limiting factor for the large scale implementation of DORA due to high costs attributed to nitrogen use. Reducing the amount of sweeping gas used while maintain driving force can be achieved by the use of vacuum on the gas outlet of the membrane. The application of vacuum was observed to reduce the amount of sweep gas needed by a factor up to 25 as shown in Figure 9. A vacuum of 850 mbara was maintained at the gas outlet (experiment 11) with sweep gas flow rate of 1 NL/h and the same rate of oxygen decay was observed as with 100 NL/h of N₂ at atmospheric pressure. An overview of the operating conditions has been provided in Table 4. Based on these experiments, all subsequent tests were performed with a combination of sweep gas and vacuum.

Table 4 - Overview of operating conditions for Figure 9

Experiment ID	Gas Flow Rate (NL/h)	Gas Outlet Pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
1	-	-	0	40
10	25	1.01	0	40
11	1	0.85	0	40

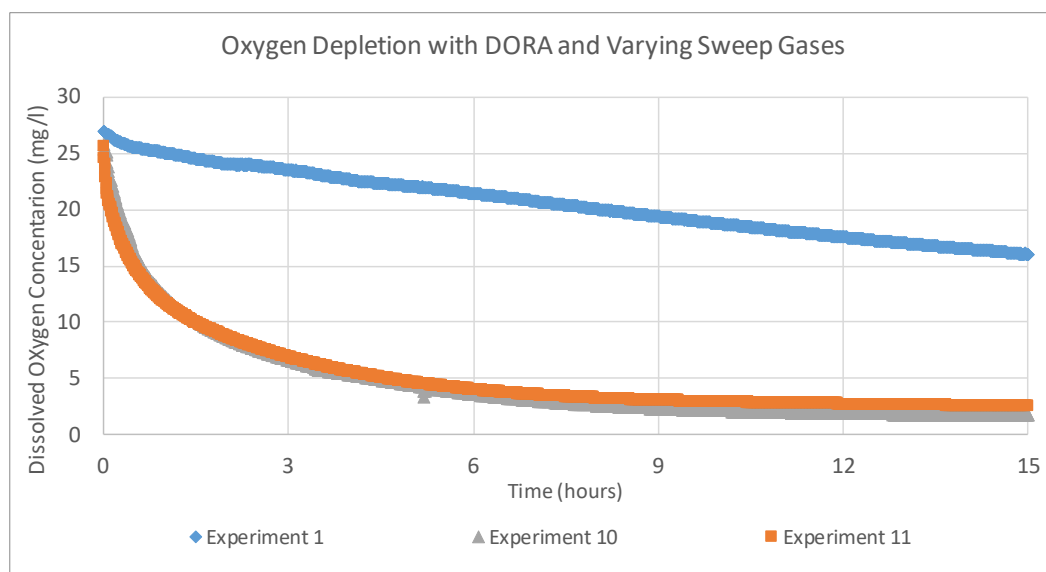


Figure 9 - Oxygen decay with DORA and vacuum

2.1.4 DORA tests with Loaded Solvent (Experiments 12 and 13)

As shown in Figure 7, the rate of oxygen decay increases significantly when the solvent is loaded with CO₂. A series of tests were carried out to check if the dense layer membrane can remove dissolved oxygen from loaded solvents. The operating conditions for these tests are presented in Table 5. Figure 10 shows that the rate of oxygen decay increases when DORA is used (experiment 12) when compared to experiment 5 where no membrane is used for solvent with loading of 0.27 mol CO₂/mol MEA. This increase in oxygen decay is attributed to the oxygen removed by the membrane. Thus, the dense layer membrane was proven to be capable of removing oxygen from loaded solvents.

Table 5 - Overview of operating conditions for Figures 10 and 11

Experiment ID	Gas Flow Rate (NI/h)	Gas Outlet Pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
5	-	-	0.27	40
7	-	-	0.5	40
12	50	0.6	0.27	40
13	50	0.6	0.5	40

The same test was also repeated for solvent with loading of 0.5 mol CO₂/mol MEA. Figure 11 shows that for this loading the oxygen removal rate is the highest at the beginning of the test and then drops significantly around 8 mg/l (experiment 13). This suggests that there is room for improvement of separation which can be achieved by application of deeper vacuum.

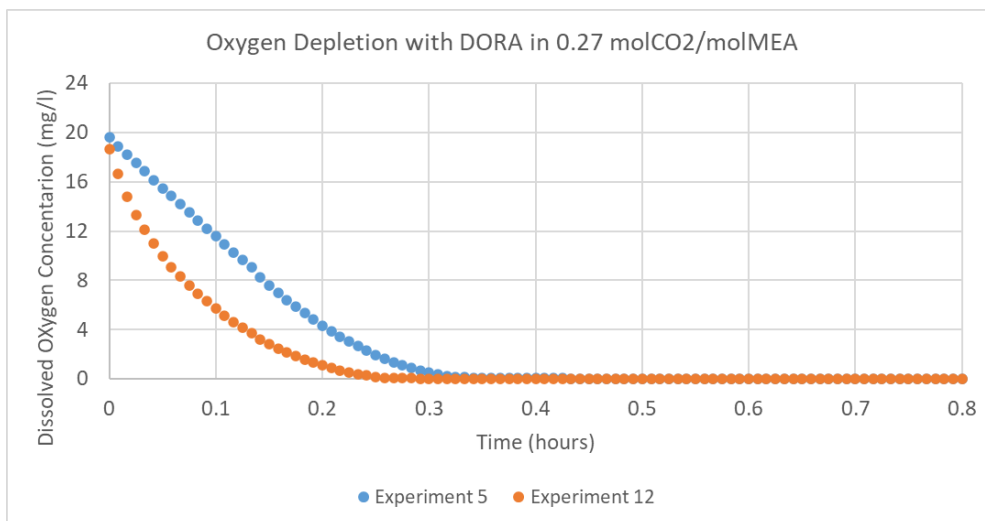


Figure 10 - Oxygen decay in loaded 30 wt% MEA (0.27 mol CO₂/mol MEA) with and without DORA

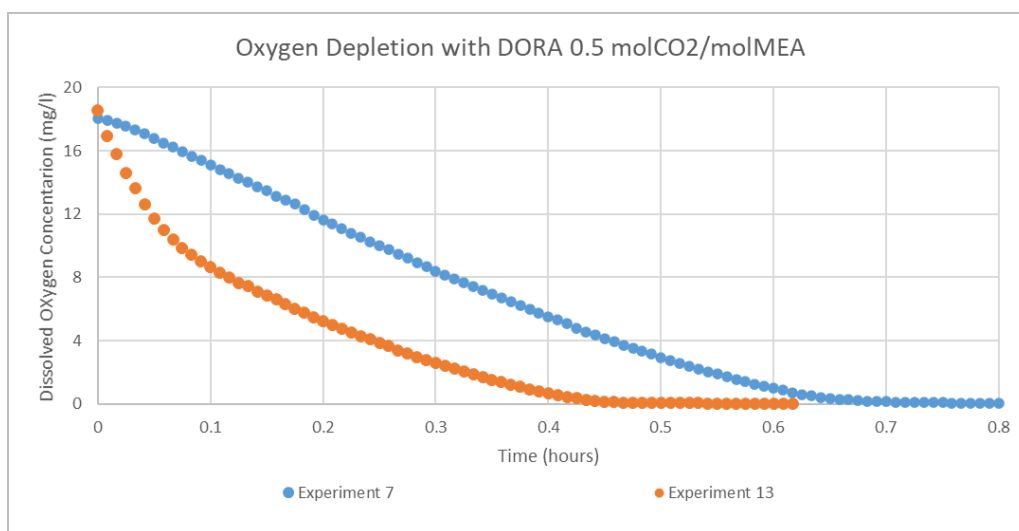


Figure 11 - Oxygen decay in loaded 30 wt% MEA (0.5 mol CO₂/mol MEA) with and without DORA

2.2 Tests with MDEA/PZ

As mentioned before MEA is a first generation CO₂ capture solvent. MEA is prone to degradation leading to solvent losses, along with adverse economic and environmental impacts. MDEA/PZ is a blended solvent that can better resist to oxidative and thermal degradation [18]. DORA experiments were also carried out with a blend of 42 wt% MDEA and 8 wt% PZ. The same approach as the previous solvents was followed i.e. first baseline experiments without DORA were carried out followed by experiments with DORA. Since MDEA/PZ degrades slower than MEA, these experiments were carried out with air instead of pure oxygen. Additionally, since it was found that application of vacuum was effective in reducing the amount of sweep gas needed while maintaining driving force, it was decided to apply as much vacuum as possible. It was possible to achieve 300 mbara of vacuum at the gas outlet.

Initially experiments were carried out with the dense layer membrane. However, these experiments were unsuccessful since the solvent was not compatible with the glue used in the membrane and it was observed that crystals (potentially PZ) were formed on the joints in the membrane module. This can be seen in Figure 12. Due to this it was not possible to maintain a constant sweep gas flow over the membrane. As a result, it was decided to perform the MDEA/PZ tests with the commercial porous membrane from 3M instead [12].



Figure 12 - Crystals observed on the gas outlet of the dense layer membrane

An overview of the experimental conditions used for the tests with MDEA/PZ have been presented in Table 6 and Table 7.

Table 6 – Experimental matrix for baseline tests with MDEA/PZ

Experiment ID	CO ₂ Loading (molCO ₂ /molMDEA-PZ)	Temperature (°C)
14	0	40
15	0	50
16	0	60
17	0.1	50
18	0.3	50
19	0.4	50

Table 7 - Experimental matrix for DORA tests with MDEA/PZ

Experiment ID	Liquid flow rate (ml/min)	Gas Flow Rate (NI/h)	Gas Outlet pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
20	25	10	0.3	0	50
21	50	10	0.3	0	50
22	100	10	0.3	0	50
23	100	10	0.3	0	50
24	100	2	0.3	0	50
25	100	10	0.3	0.1	50
26	100	10	0.3	0.3	50
27	100	10	0.3	0.4	50

2.2.1 Baseline Tests Without DORA (Experiments 14 to 20)

Baseline tests were carried out without a membrane with MDEA/PZ. The oxygen decay was observed at different temperatures and CO₂ loadings according to the matrix presented in Table 6. **Error! Reference source not found.** shows the oxygen decay in unloaded MDEA/PZ. As temperature increases the rate of oxygen consumption also increases. As expected, the solubility of oxygen reduces at higher temperatures.

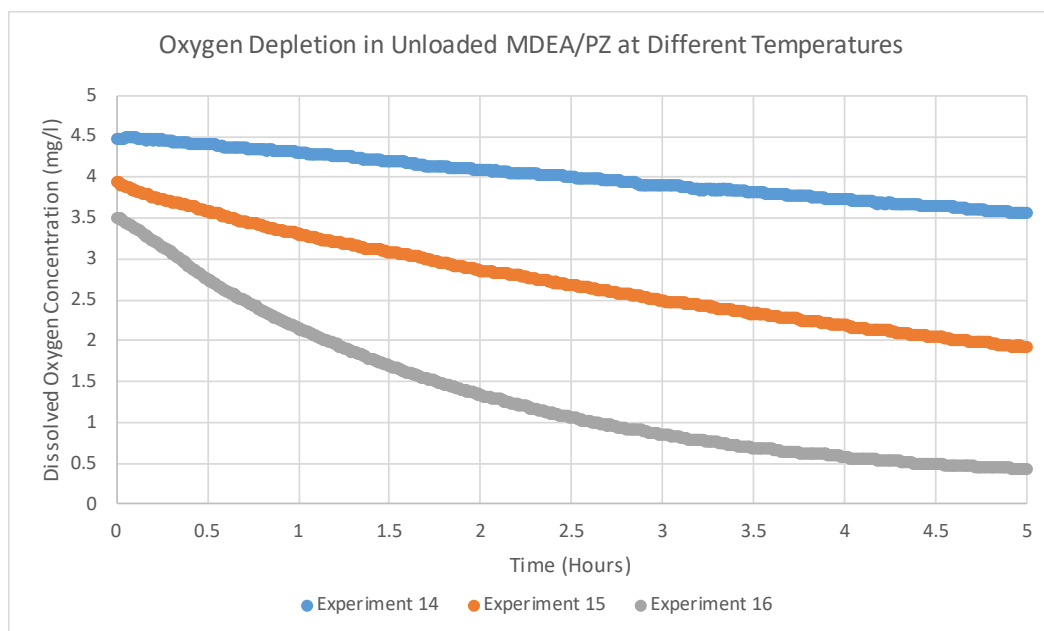


Figure 13 – Oxygen decay in unloaded MDEA/PZ at different temperatures

Baseline tests were also carried out for loaded solvents. Figure 14 shows that like MEA, the rate of oxygen decay increases upon introduction of CO₂ in the solvent. However, in this case no clear trend has been observed in terms of the rate of oxygen decay and level of loading. According to these experiments, the rate of oxygen decay is fastest for solvent with loading of 0.4 mol CO₂/mol amine (experiment 19), followed by solvent with loading of 0.1 mol CO₂/mol amine (experiment 17) and finally slowest in solvent loaded with 0.3 mol CO₂/mol amine (experiment 18). At this moment it is unclear why there is no clear trend in oxygen decay and also why the behavior differs from that of MEA. However, all these experiments have been performed in duplicates and are reproducible.

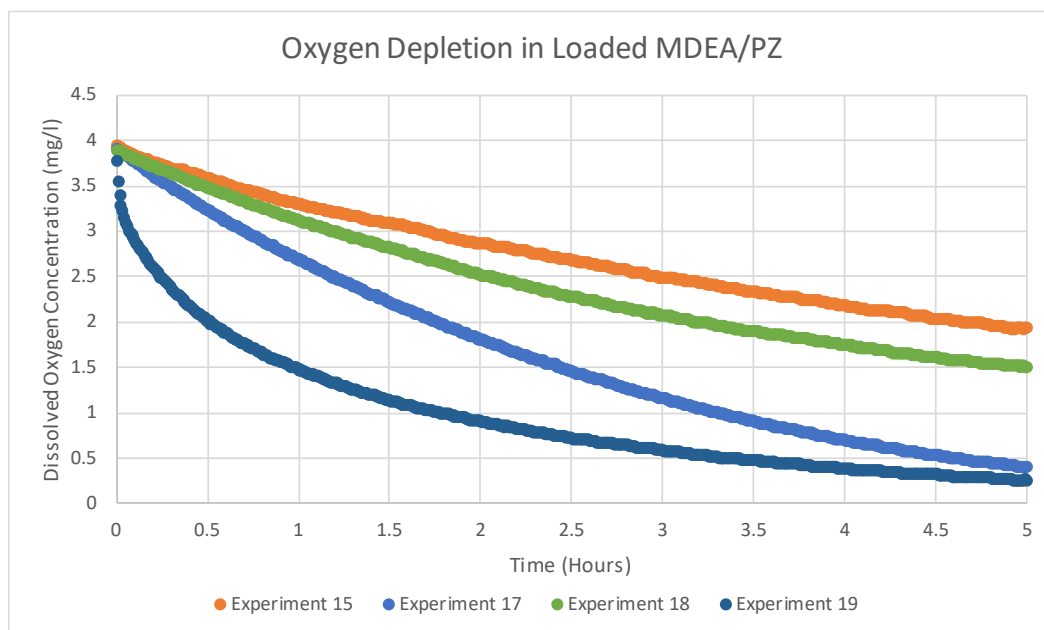


Figure 14 - Oxygen decay in loaded MDEA/PZ without DORA

2.2.2 DORA Tests with Varying Liquid Flow Rates (Experiments 20 to 22)

Since the dense layer membrane was not suitable for oxygen removal from solvents containing PZ, the commercial porous membrane was used. As a result new experiments were carried out to determine the optimum liquid and sweep gas flow rates. These results are shown in Figure 15. The oxygen decay increases significantly at higher liquid flow rates. The experimental conditions for these experiments are presented in Table 8. As a result, the subsequent tests were carried out with liquid flow rates of 100 mL/min which is the limiting flow for the membrane.

Table 8 - Overview of operating conditions for Figure 15

Experiment ID	Liquid flow rate (ml/min)	Gas Flow Rate (NI/h)	Gas Outlet pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
15	-	-	-	0	50
20	25	10	0.3	0	50
21	50	10	0.3	0	50
22	100	10	0.3	0	50

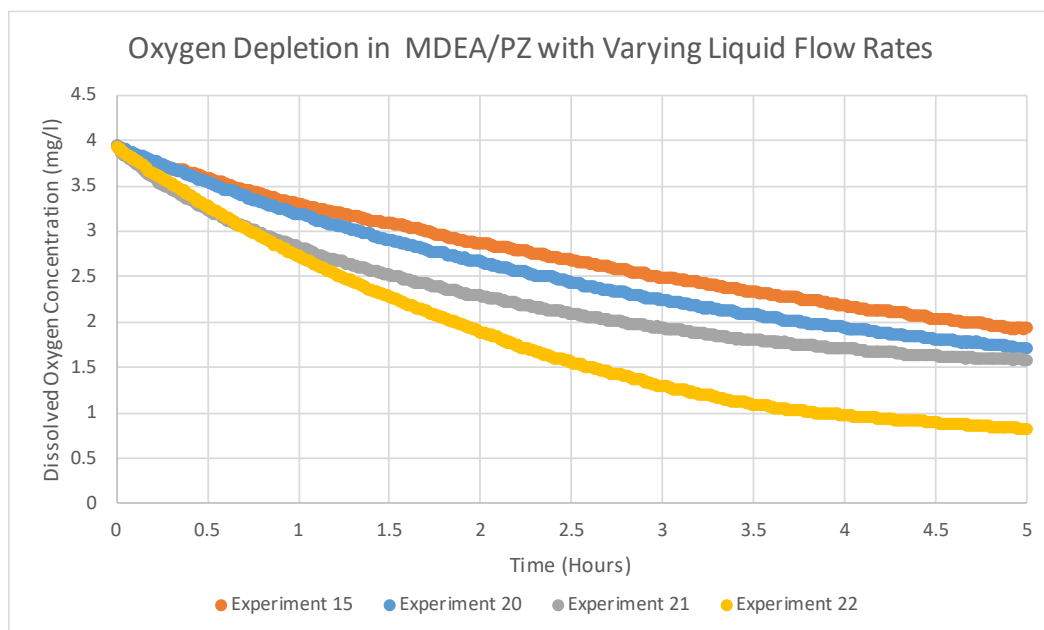


Figure 15 - Oxygen decay in unloaded MDEA/PZ with DORA and varying liquid flow rates

2.2.3 DORA Tests with Varying Gas Flow Rates (Experiment 23 to 24)

Tests were carried out to determine the optimum gas flow rate for oxygen removal. The operating conditions for these tests have been presented in Table 9. As shown in Figure 16, no significant difference was observed at flow rates tested. Marginally better separation was observed at 10 NL/h of nitrogen flow. Thus, all successive experiments were carried out with this flow rate.

Table 9 - Overview of operating conditions for Figure 16

Experiment ID	Liquid flow rate (ml/min)	Gas Flow Rate (NL/h)	Gas Outlet pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
15	-	-	-	0	50
23	100	10	0.3	0	50
24	100	2	0.3	0	50

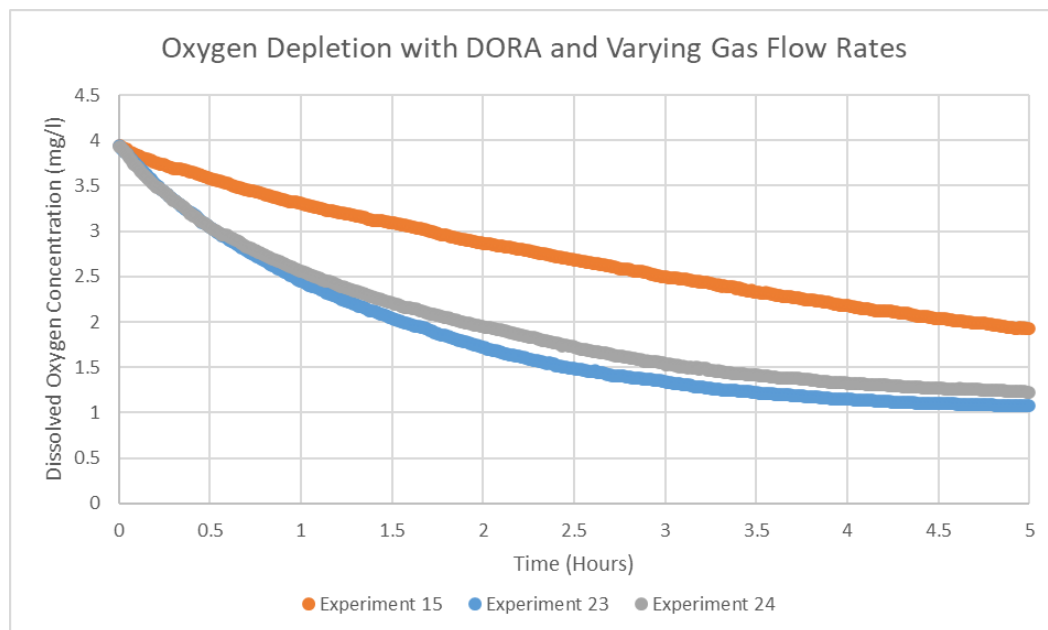


Figure 16 - Oxygen decay in unloaded MDEA/PZ with DORA and varying liquid flow rates

2.2.4 DORA Tests with Loaded MDEA/PZ (Experiments 25 to 27)

The aforementioned tests led to the determination of the optimum liquid and sweep gas flow rates. Using a liquid flow rate of 100 mL/min and gas flow rate of 10 NL/h with vacuum of 0.3 bara, DORA was tested for loaded solvents. The operating conditions for these tests have been provided in Table 10.

Table 10 - Overview of operating conditions for Figures 17, 18, and 19

Experiment ID	Liquid flow rate (ml/min)	Gas Flow Rate (NL/h)	Gas Outlet pressure (bara)	CO ₂ Loading (molCO ₂ /molMEA)	Temperature (°C)
17	-	-	-	0.1	50
18	-	-	-	0.3	50
19	-	-	-	0.4	50
25	100	10	0.3	0.1	50
26	100	10	0.3	0.3	50
27	100	10	0.3	0.4	50

Figure 17, Figure 18 and Figure 19 show the oxygen depletion in loaded solvents with and without DORA. For all loadings investigated here the membrane was able to separate more than 90% dissolved oxygen from the solvent. No significant difference was observed with the use of different CO₂ loadings.

No material compatibility issues were observed between MDEA/PZ and the commercial porous membrane. A point of attention is that when using a porous membrane there are solvent losses through the membrane. This increases the chances of efficiency loss in time due to pore wetting. However, membranes can be regenerated and that is an option that will be explored in the future demonstrations of DORA at TRL7 operations.

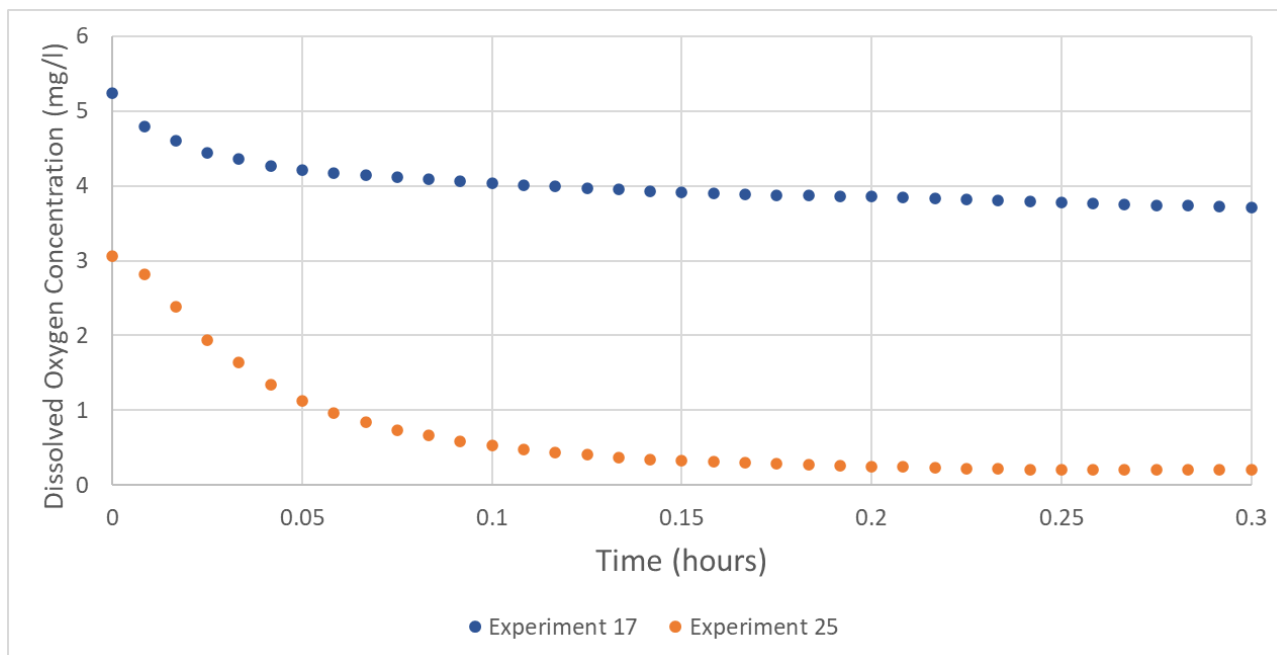


Figure 17 - Oxygen decay in loaded MDEA/PZ (0.1 mol CO₂/mol amine) with and without DORA

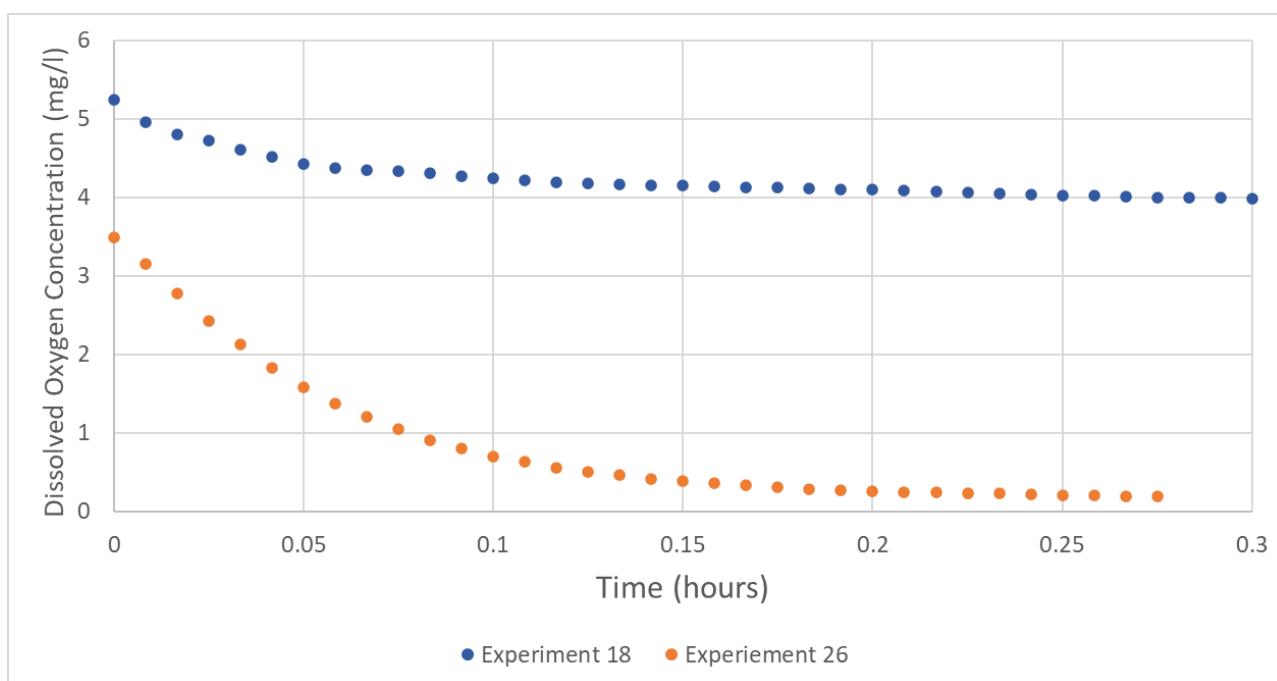


Figure 18 - Oxygen decay in loaded MDEA/PZ (0.3 mol CO₂/mol amine) with and without DORA

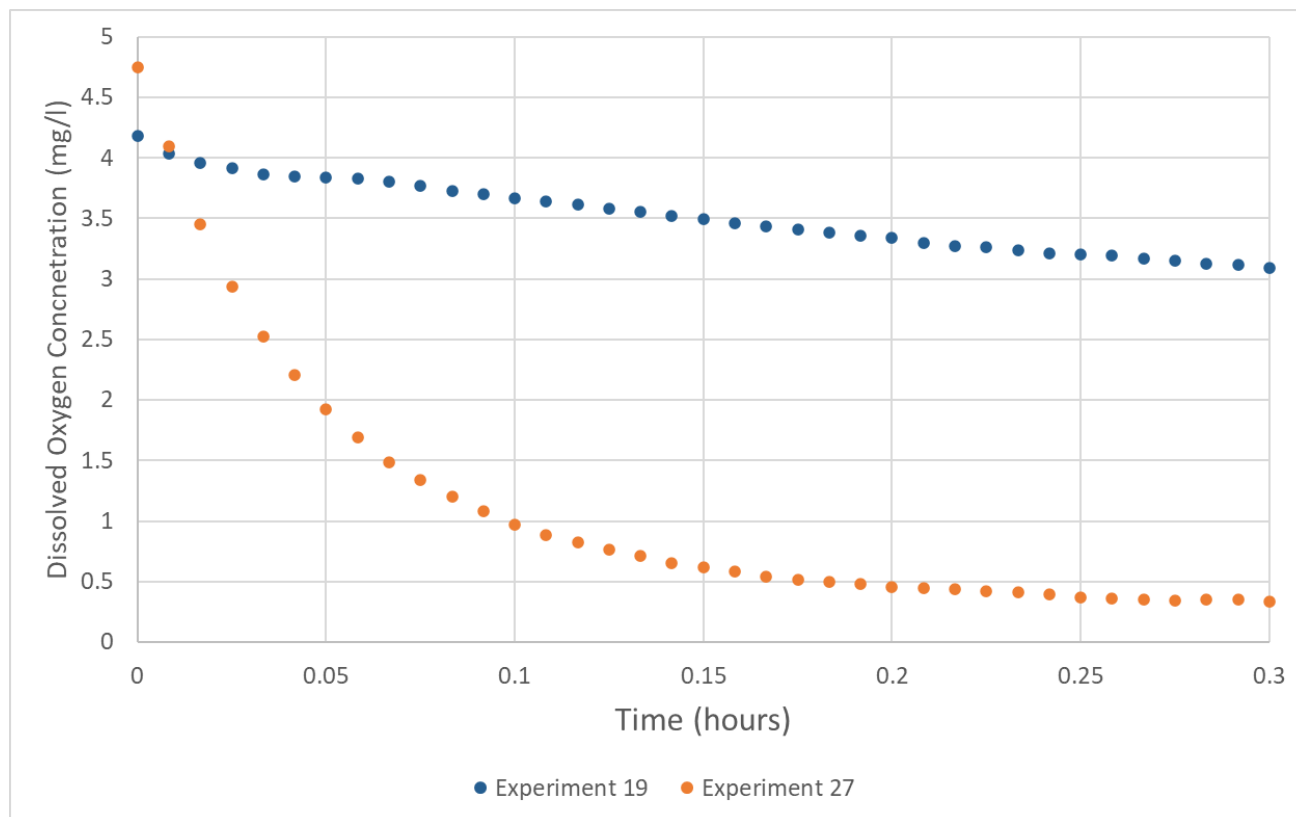


Figure 19 - Oxygen decay in loaded MDEA/PZ (0.4 mol CO₂/mol amine) with and without DORA

2.2.5 DORA efficiency for loaded MDEA/PZ

To better understand the observations in Figure 17 to Figure 19, some data treatment is needed. For this, the data at 50°C and 0.4 loading will be used, as it is the most representative of rich solvent conditions in the absorber sump. From experiment 19, a kinetic constant can be derived for the oxygen consumption rate. The data is well represented by a 0.5 order function, which leads to a kinetic constant of $1.93 \text{ mg}^{0.5} \cdot \text{L}^{-0.5} \cdot \text{h}^{-1}$. The predictions of the kinetic model can be observed in Figure 20. The highest deviation is 5% (0.18 mg/L), and the average deviation is 3%. These deviations are within the expected experimental error, and the model is therefore considered to be representative of the data.

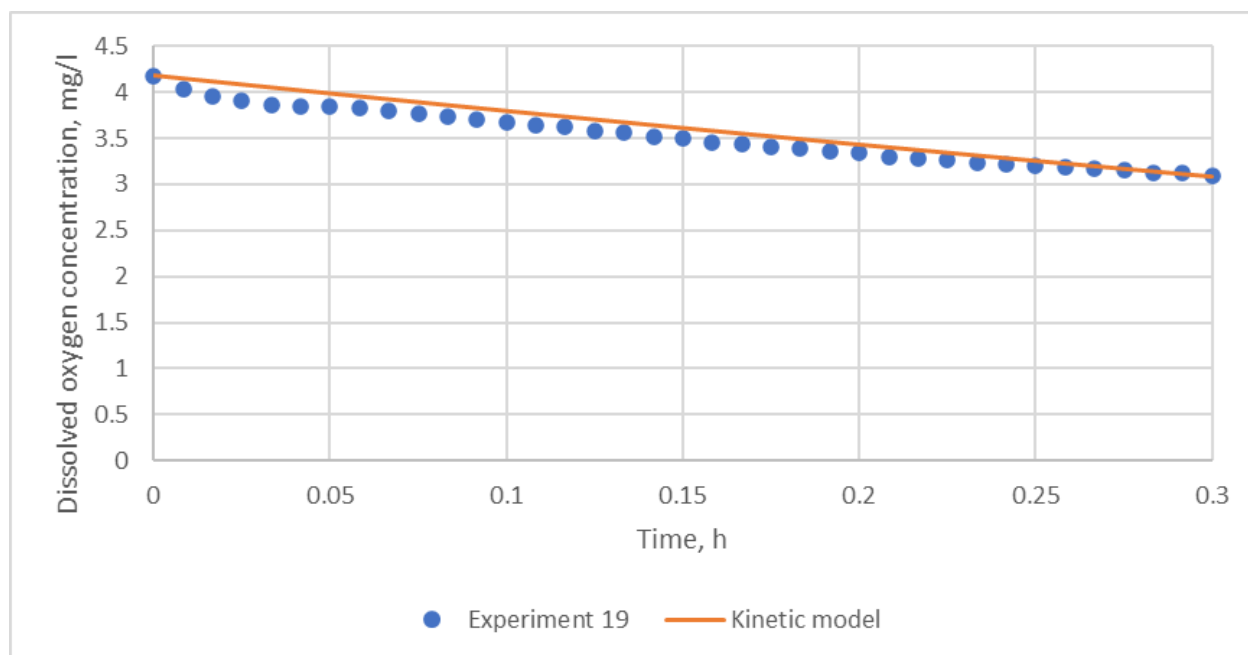


Figure 20 – Kinetic model and experimental data for oxygen reaction in MDEA/PZ with 0.4 loading at 50°C

With this, the data from experiment 27 can be interpreted by dividing the observed total rate of oxygen decay into 2 terms: Reaction and DORA. The reaction term can be calculated using the kinetic model, and the DORA term is the difference between the model and the total measured value. These results are given in Figure 21.

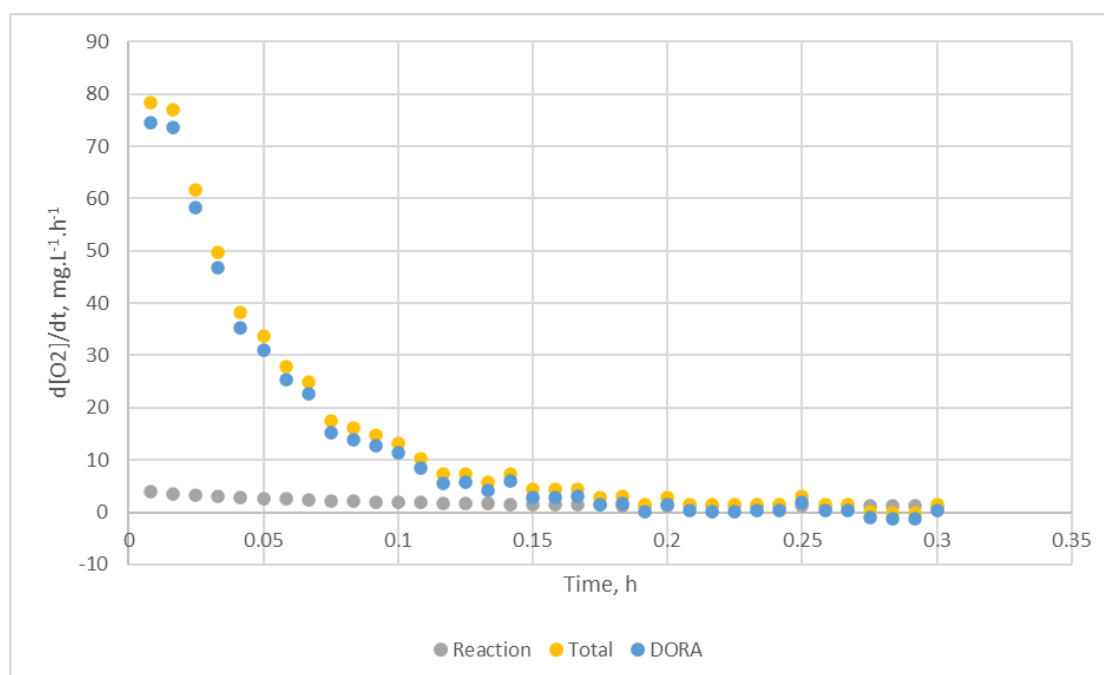


Figure 21 – Oxygen decay rate split into Reaction and DORA terms

It can be seen that, for the initial period of the experiment, DORA is the dominant factor. From 0.1 h onwards, the effects of DORA and reaction are similar. This is better understood when the reaction and DORA terms are plotted as a function of the dissolved oxygen concentration, as in Figure 22. This clearly indicates that the DORA removal rate is a much stronger function of the DO concentration than the reaction term. As the DO concentration drops, the driving force drops and so does the DORA efficiency.

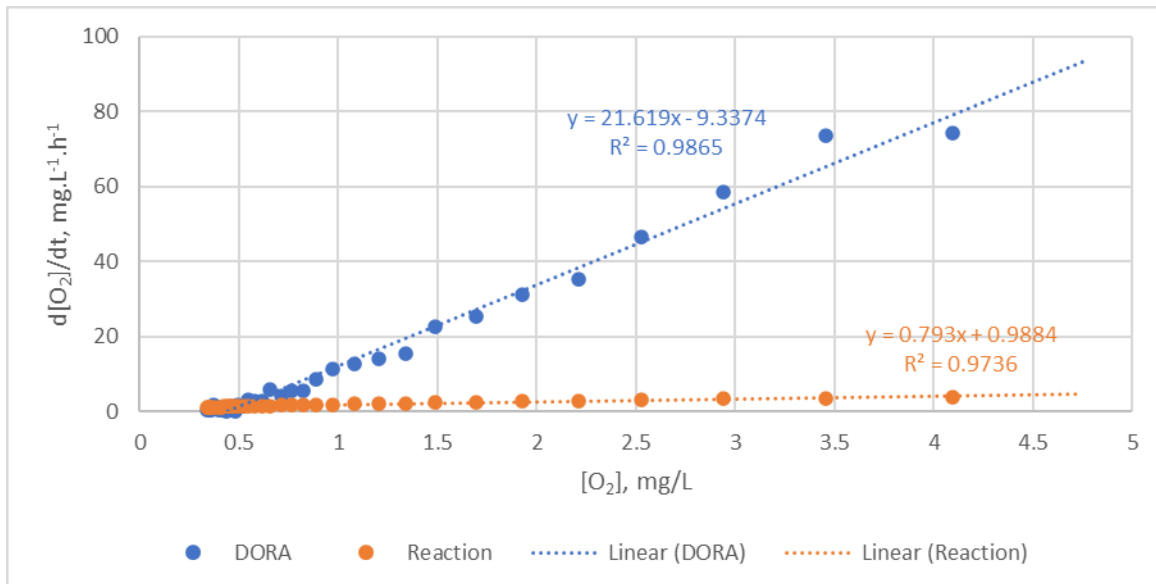


Figure 22 – DORA and Reaction terms as function of oxygen concentration

2.3 Tests with CESAR1

Following the tests performed with MDEA/PZ, tests were done using CESAR1 a blend of 26.7% AMP and 12.9% PZ. The same approach as the previous solvents was followed i.e. first baseline experiments without DORA were carried out followed by experiments with DORA. The same as for MDEA/PZ, since CESAR1 degrades slower than MEA, these experiments were carried out with air instead of pure oxygen. To increase the efficiency of the removal, vacuum was used on the outlet of the gas side of the membrane. A minimum value achieved was 300mbara.

An overview of the experimental conditions used for the tests with CESAR1 have been presented in Table 11 and Table 12.

Table 11 - Experimental matrix for baseline tests with CESAR1

Experiment ID	CO ₂ Loading (molCO ₂ /mol CESAR1)	Temperature (°C)
28	0	40
29	0	50
30	0	60
31	0	70
32	0	80
33	0.16	40
34	0.26	40
35	0.49	40

Table 12 - Experimental matrix for DORA tests with CESAR1

Experiment ID	Liquid flow rate (L/h)	Gas Flow Rate (NL/h)	Gas Outlet pressure (bara)	CO ₂ Loading (molCO ₂ /molCESAR1)	Temperature (°C)
36	3	50	0.6	0,32	40
37	3	50	0.6	0,41	40

2.3.1 Baseline Tests Without DORA (Experiments 28 to 34)

Baseline tests were carried out without a membrane with CESAR1. The first tests were done with a broader temperature range (from 40°C to 80°C) to check the influence of higher temperatures in the oxygen depletion without any addition of CO₂. For the loaded samples, a fixed temperature was chosen (40°C).

The effect of temperature observed in Figure 23 shows that for lower temperatures (between 40°C and 50°C), CESAR1 is very stable. When the temperature used is higher than 60°C, the oxygen decay is more pronounced. At the same time, the tests at higher temperatures were difficult to control since the equipment used is not constructed to be operated at higher pressures. Therefore, it was decided to keep the maximum temperature used at 60°C to ensure stability and reproducibility of the results.

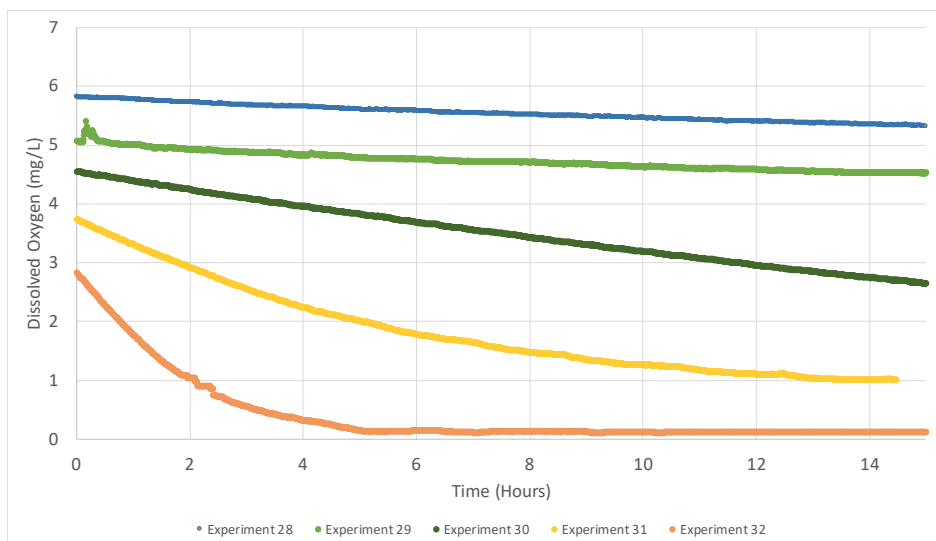


Figure 23 - Oxygen decay in unloaded CESAR1 at different temperatures

Baseline tests were also carried out for loaded solvents. Figure 24 shows that like MEA and MDEA/PZ, the rate of oxygen decay increases upon introduction of CO₂ in the solvent. However, the decay is less pronounced for higher loadings than observed in the MDEA/PZ tests, for example. When comparing both solvents with similar loadings (0.4 molCO₂/mol amine – 0.49molCO₂/mol amine), the values of oxygen in MDEA/PZ went to almost zero after 5 hours, while the minimum value observed for CESAR1 stayed above 4mg/L for all tests. This shows the higher stability of CESAR1 in terms of oxidative degradation when compared to both MEA and MDEA/PZ.

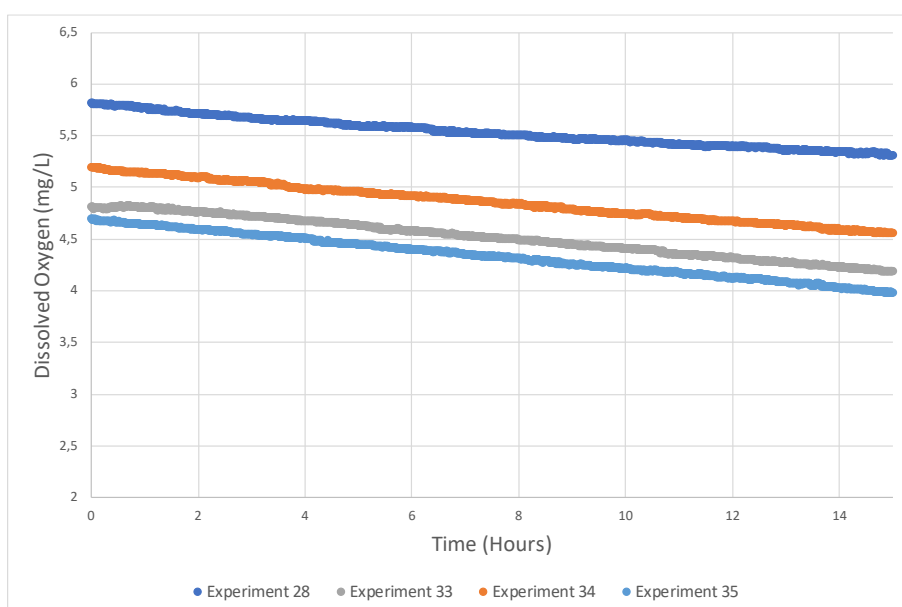


Figure 24 - Oxygen decay in loaded CESAR1 at 40°C

2.3.2 DORA Tests with CO₂ loading (Experiments 36 and 37)

The experiments with the DORA and CESAR1 were done using the dense layer membrane. This decision was taken for two main reasons: the first reason was to verify material compatibility and check if the issues observed when operating with MDEA/PZ would also be observed with CESAR1, another solvent containing

piperazine; the second is related to the volatility of AMP in combination with a porous membrane. As described in deliverable *D5.1.1 LAUNCH rig validations in head-to-head tests* from WP5, the porous membrane was first operated in combination with CESAR1 as part of the Head-to-Head comparison of RWE's plant and LAUNCH rig #2. During the operation, two main issues were encountered: clogging of the membrane leading to less efficiency and leakages of the solvent to the gas side during operation. Due to those reasons, it was decided to not go forward with CESAR1 tests using the porous membrane and to focus on the dense layer membrane to develop this technology further.

Since the issues with the dense layer membrane were due to the glue used, two new modules were fabricated by NTNU using a different type of glue and shipped to TNO so they could be used in the CESAR1 tests. Figure 25 shows the results of both tests done with the DORA with loaded CESAR1. From the graph it is already possible to conclude that almost no removal was achieved. The tests were done in duplicate, using also two different loadings and two new membranes, and the same result was observed. When opening the membranes, both had issues with precipitation of CESAR1 and rupture of the glue layer. Therefore, it was decided to terminate the tests with both membranes.

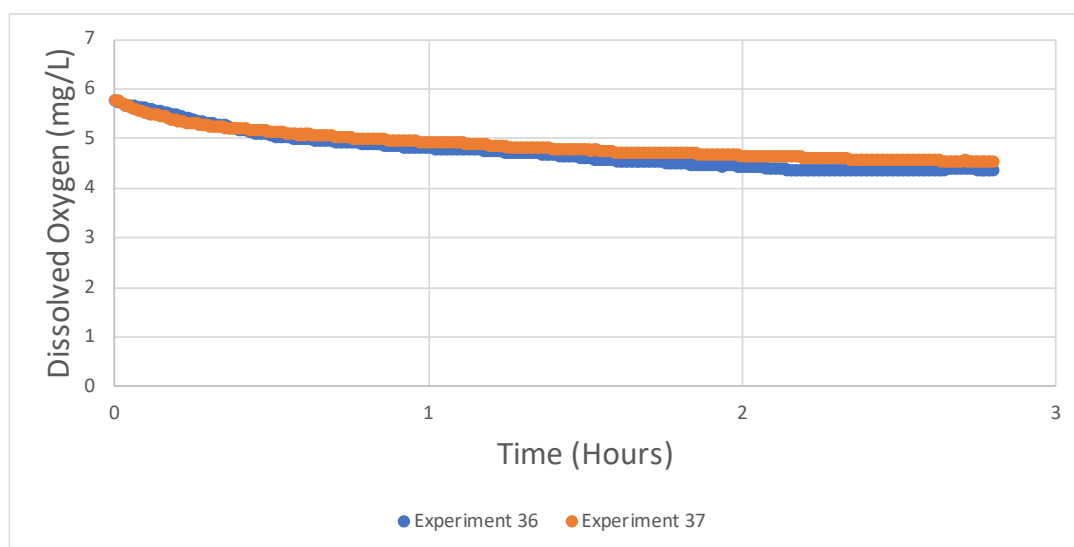


Figure 25 - Oxygen decay in loaded CESAR1 (0.31 and 0.41 mol CO₂/mol amine) with DORA

It is important to notice that even though the tests of the membrane were not conclusive and that CESAR1 is a solvent that is more stable in terms of oxidative degradation, the oxygen removal is still necessary for the process not only to slow down the effects of degradation but also to improve the quality of the CO₂ produced. While some oxygen is consumed by degradation of amines, the rest is carried over into the captured CO₂ product. To achieve pipeline transport specifications, captured CO₂ is normally compressed to about 100 bara. Presence of oxygen in the CO₂ product would increase the possibility of two-phase flow and as a result higher pressures would be needed to avoid this regime, thereby increasing the cost of compression. Additionally, oxygen may also contribute to corrosion of pipes. Along with that, captured CO₂ can be stored in underground reservoirs, however, the presence of oxygen can cause precipitation reactions leading to reduced permeability of the reservoir. Oxygen can also lead to microbial growth that can reduce the CO₂ injection efficiency due to blocking of pores [23]. In CCU applications catalysts for the hydrogenation of CO₂ can be deactivated by oxygen.

The O₂ content in the CO₂ produced at the stripper is unclear. Few literature sources report this, and at TCM, operating with MEA, it has been measured with online instrumentation as 1.8-2.0 ppmv. Manual sampling reports oxygen and argon combined concentrations between 17 and 49 ppmv [19]. Unfortunately, data for other solvents is not available. The maximum concentration of O₂ in the CO₂ product, calculated based on average O₂ solubility in loaded amines and not considering consumption due to degradation, is ca. 80 ppmv. The actual content will be higher for slow degrading solvents (e.g., CESAR1) than for fast



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degrading ones (e.g., MEA). This knowledge gap still needs to be closed for both CESAR1 and MDEA/PZ and further steps to accomplish this will be discussed in "Chapter 4 – Way Forward".

3 Impact of DORA

In order to estimate the impact of DORA, the extent of oxygen consumption due to oxidative degradation was estimated with a degradation network model (DNM). This model was developed to quantify the consumption of oxygen under different operation conditions. A first version of the model was developed by TNO during ALIGN CCUS [25], and within LAUNCH WP1 work, the DNM has been modified, updated with new ODIN data and extended to include CESAR1 (this is reported in D1.3.4).

With that model, and considering a hypothetical CO₂ capture plant with capacity of 200,000 tonnes of CO₂ per year, the MEA and CESAR1 losses were estimated as shown in Figure 26. For MEA, most of the degradation is expected to happen in the absorber packing. DORA is expected to have a higher impact for solvents that are more resistant to degradation, for which degradation happens mostly in or downstream the absorber sump, like CESAR1. In this case, considering that DORA would be able to separate 90% of the oxygen, a configuration with DORA upstream the sump would lead to 80% reduction in CESAR1 losses (from 150 to 30 g/tonCO₂).

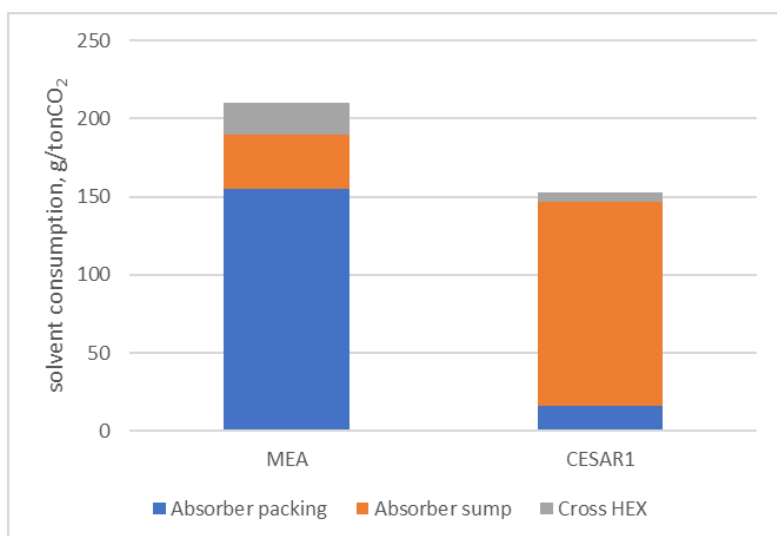


Figure 26. MEA and CESAR1 losses per equipment

A comprehensive techno-economic assessment of degradation mitigation technologies, including DORA, is performed in WP6, and presented in D6.1.1.

4 Conclusions and Way Forward

This work describes the operation of TNO's Oxygen Depletion Installation with MEA, MDEA/PZ and CESAR1 with and without the DORA. During these experiments, two membranes were used: a commercial membrane from Liqui-Cell and a dense layer membrane developed by NTNU. Even though the second membrane was chosen in deliverable D2.1.1 *Membrane selection for DORA* as the most suited for the experiments to be performed, in this report we show that the solvents containing piperazine were not compatible with the glue used for sealing the dense layer membrane module. At the same time, results from deliverable D5.1.1 *LAUNCH rig validations in head-to-head tests* showed that CESAR1 did not perform well with the porous membrane, most likely due to pore wetting. This may be related to relatively solvent losses because of the AMP volatility. Therefore, the dense layer membrane was only used for the MEA experiments while the porous membrane was used for the MDEA/PZ system. No suitable membrane was available for CESAR1 tests.

During the MEA experiments the operating conditions of the membrane such as liquid flow rate, gas flow rate and pressure (vacuum) were optimized. The most efficient operation was done with a combination of nitrogen as a sweep gas and vacuum on the outlet of the membrane. The use of vacuum made it possible to use significant lower amount of nitrogen in the sweeping gas. This is an important improvement of the process not only in terms of efficiency of removal but also OPEX related to the flow of nitrogen used during the operation. Another significant result was the fact that DORA was able to remove oxygen even for MEA in which degradation occurs in a fast rate. The tests with MDEA/PZ also showed the decrease in oxygen while using the DORA in the system for different CO₂ loadings. For both solvents, the oxygen content during the operation with DORA dropped to almost zero (or even zero in some cases) in less than 30 minutes. For CESAR1, it was not possible to quantify the impact of the DORA since one of the membranes was not compatible with the solvent and the other one showed a high rate of leakage when operated with CESAR1.

In conclusion, DORA is potentially a technically viable solution for controlling the degradation of CO₂ capture solvents and can be applied independently or in combination with other solvent management strategies such as reclaiming. However, some further development is still needed before the technology can be commercially available. TNO, NTNU, SINTEF and RWE are working in a proposal called MeDORA (project approved in ACT4 scheme) to ensure that the DORA technology will be ready for implementation by 2026. The following challenges and propositions are identified to be taken further as part of MeDORA:

1. Porous membrane wetting and fouling may lead to lower O₂ removal capacity over time, posing a stability challenge. In MeDORA we will demonstrate long-term operation at pilot scale including cycles to regenerate the membranes, retrieving the initial removal rate. Moreover, commercial non-porous asymmetric membranes will be investigated. These membranes can potentially avoid wetting, ensuring improved long-term stability. The two paths will be compared, and the best solution will be adopted for full scale.
2. Adding DORA to a CO₂ capture plant will pose engineering challenges. The optimal integration strategy will maximize oxygen removal while having minimal impact on plant dynamics. The MeDORA consortium will collaborate in reviewing the design of the DORA skid for establishing a final, commercial-ready product to be integrated in CO₂ capture plants (new-built and retrofit).
3. Side-effects of removing oxygen from the solvent may include weakening of the passivation layer on the steel surface, leading to more corrosion, and accumulation of non-oxidative degradation products that would otherwise be oxidized. Both possible issues will be closely monitored during MeDORA.

The step between the tests done as part of LAUNCH and bringing the technology to the market is of major importance to ensure a smooth transition of the DORA from laboratory and demonstration to real implementation in different industrial facilities.

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