SHARING ACT LAUNCH RESULTS

An overview and discussion on results and conclusions

Peter van Os, TNO, Project Coordinator

February 15th 2023

AGENDA

13h00 – 13h15 Welcome (TNO), Introduction and project overview (Peter van Os)

The agenda includes 10 minutes of presentation and 5 minutes for questions.

13h15 – 13h30 Pitch - The war on solvent degradation - Gary
13h30 – 13h45 How to qualify a solvent? - Hanna
13h45 – 14h00 Demonstration of solvent degradation Database – Andreas
14h00 – 14h15 Degradation Modelling – Juliana
14h15 – 14h30 Accelerated solvent degradation – Eirini/Akram

14h30 – 14h45 Break

14h45 – 15h00 The LAUNCH rig – Juliana
15h00 – 15h15 Controlling degradation (mitigation technologies at RWE) – Peter Moser
15h15 – 15h30 Controlling degradation (oxygen removal) – Roberta/Fred
15h30 – 15h45 Controlling degradation (cost estimation) – Angus

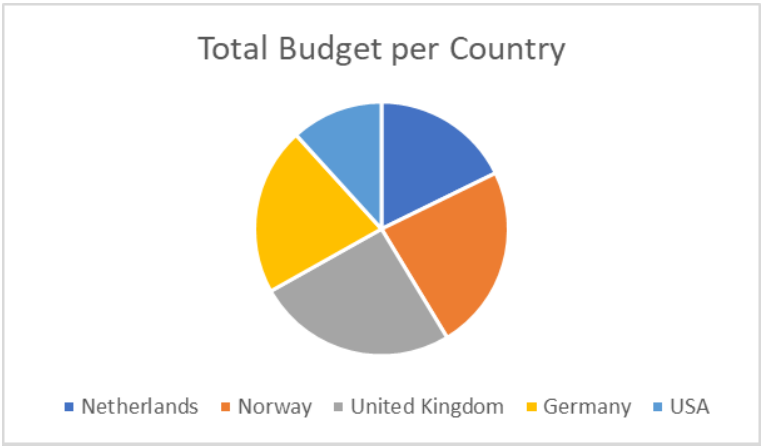
15h45 – 16h30 Discussion on remaining battles to win the war – Juliana / All

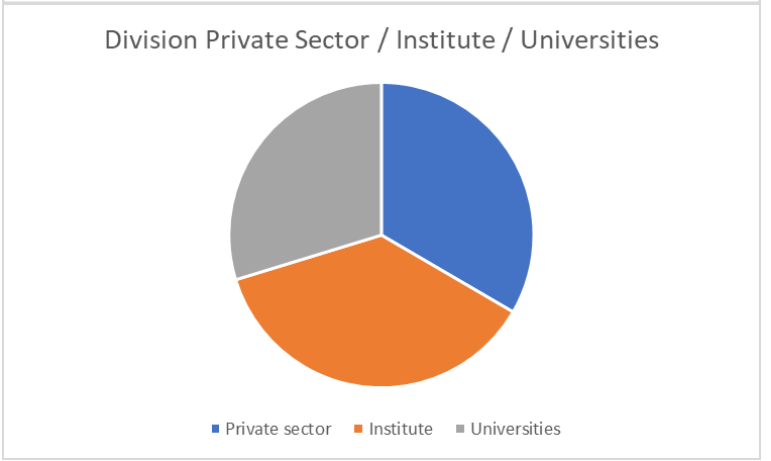
16h30 – End of the meeting

The LAUNCH Project

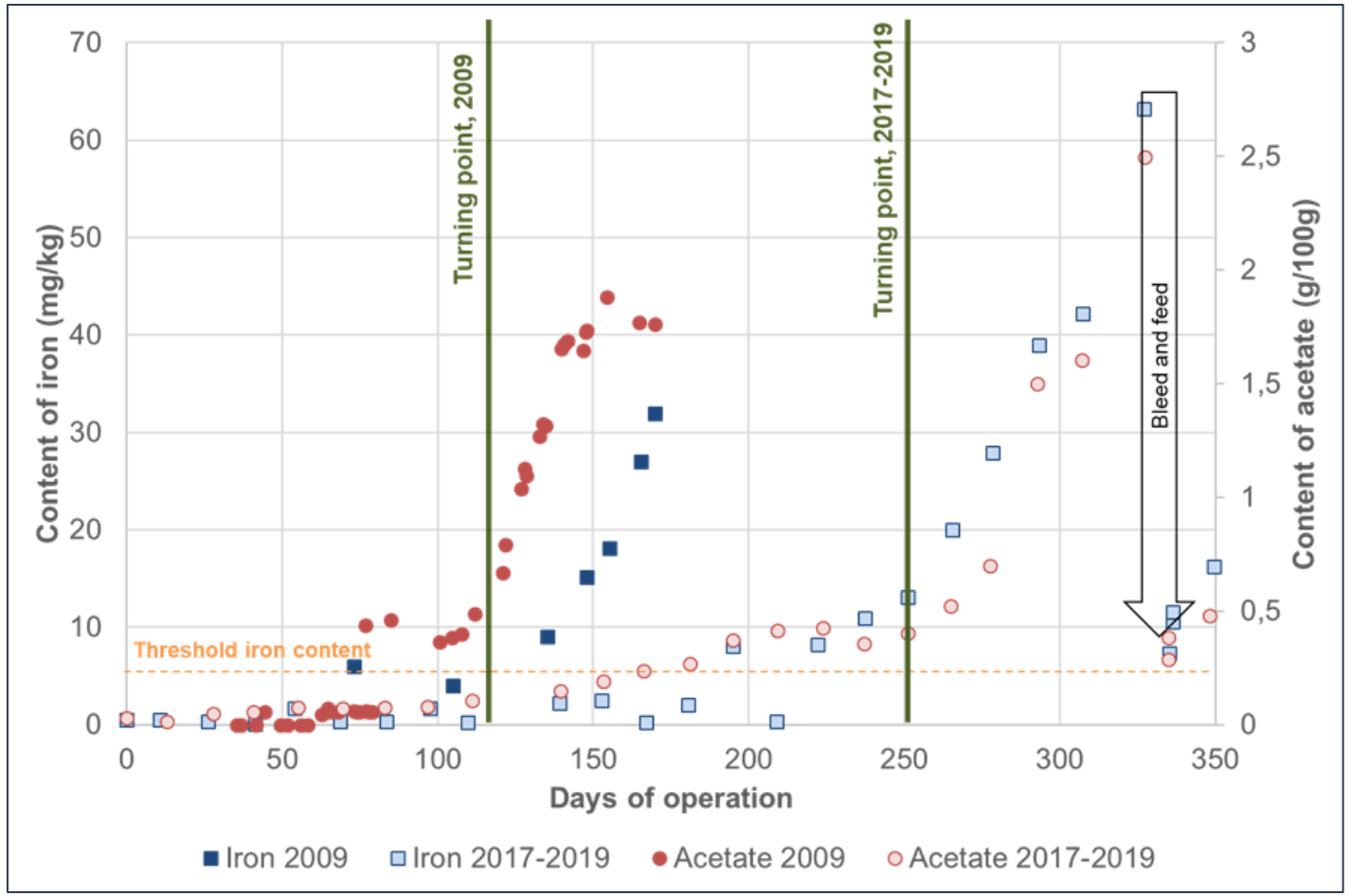
Lowering Absorption process UNcertainty, risks and Costs by predicting and controlling amine degradation

- 11 partners from NL, UK, DE, NO, USA
- Total budget: € 7.248.625
- Total funding: € 5.090.849



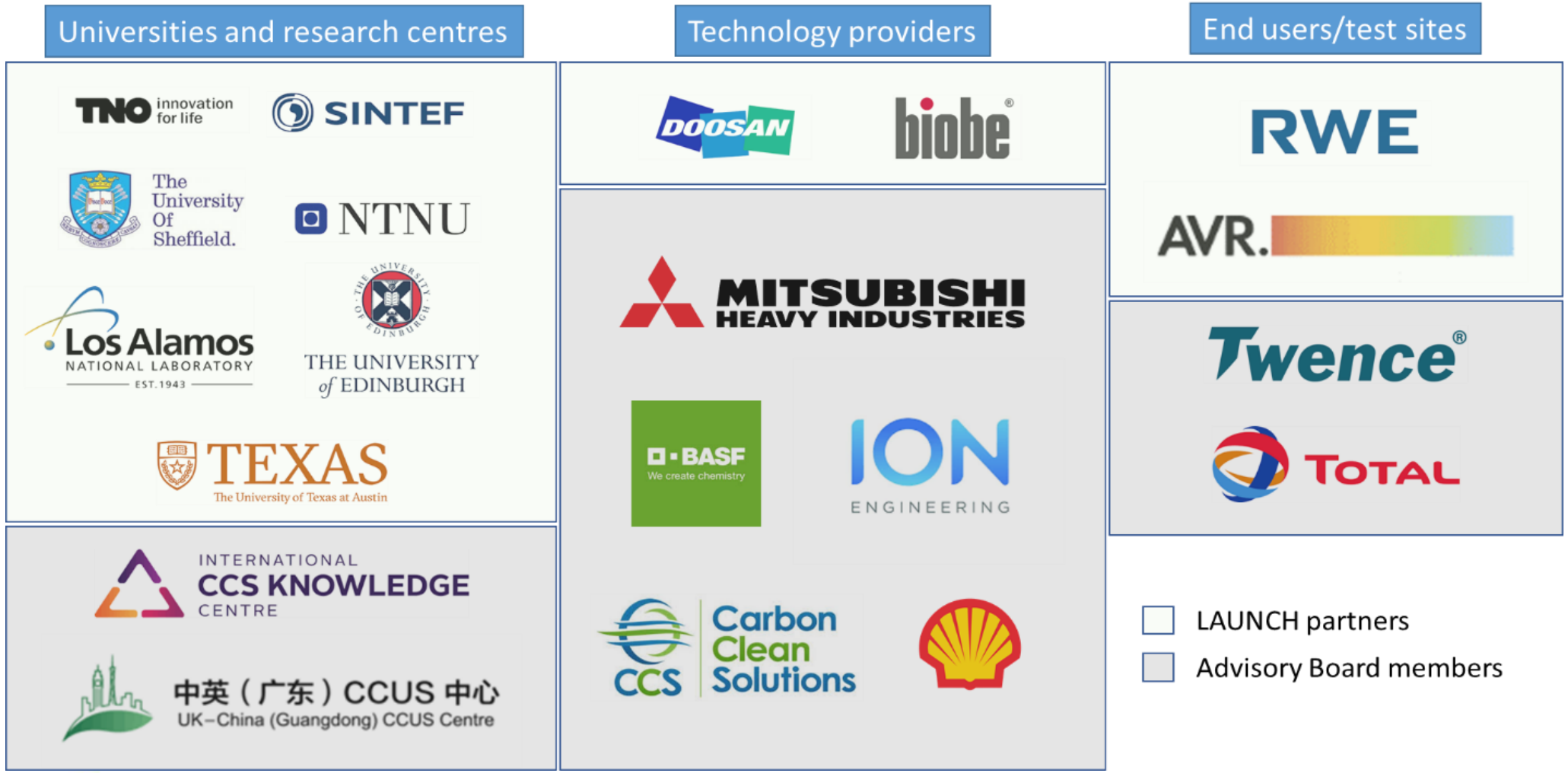


The ISSUE LAUNCH want to tackle



The results of the MEA campaign at the RWE pilot plant in Niederaussem within the ALIGN project. The "turning point" of the **first campaign was 100 days of operation in contrast to 250 days in the second campaign**.

The LAUNCH Consortium



The LAUNCH GOAL

Development of **The LAUNCH Solvent Development Protocol**
→ This protocol will be made **public**.

This protocol will include guidelines for:

LAUNCH-developed solvent degradation database

LAUNCH-developed degradation network model

Solvent testing and the drawings of a generic LAUNCH test rig

Work packages in LAUNCH

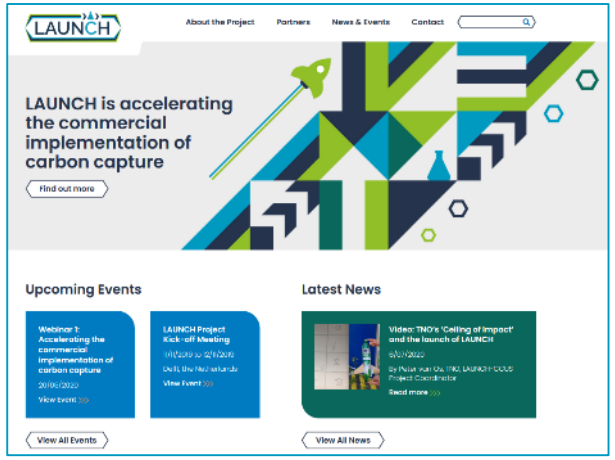
	Name	Participants (**Leader**)
WP1	Predicting degradation Andreas Grimstvedt	**SINTEF IND**, NTNU, TNO, RWE, UnivShef
WP2	Controlling degradation Roberta Figueiredo	**TNO**, LANL/UT, RWE, Biobe, NTNU
WP3	Closing degradation knowledge gaps Hanna Knuutila	**NTNU**, SINTEF IND, LANL/UT
WP4	Development of LAUNCH solvent qualification program Jon Gibbins	**UnivShef**, TNO, SINTEF IND, NTNU, LANL/UT, DOOSAN, UEDIN
WP5	Demonstration of LAUNCH solvent qualification program Peter Moser	**RWE**, AVR, LANL/UT, TNO, NTNU, UnivShef
WP6	Techno-economic evaluation Jonathan Slater	**DOOSAN**, TNO, RWE, BIOBE
WP0	Management, dissemination and exploitation Peter van Os / Juliana Monteiro / Richard Stevenson	**TNO**, SINTEF IND, NTNU, UnivShef, UEDIN, RWE, DOOSAN

Dissemination

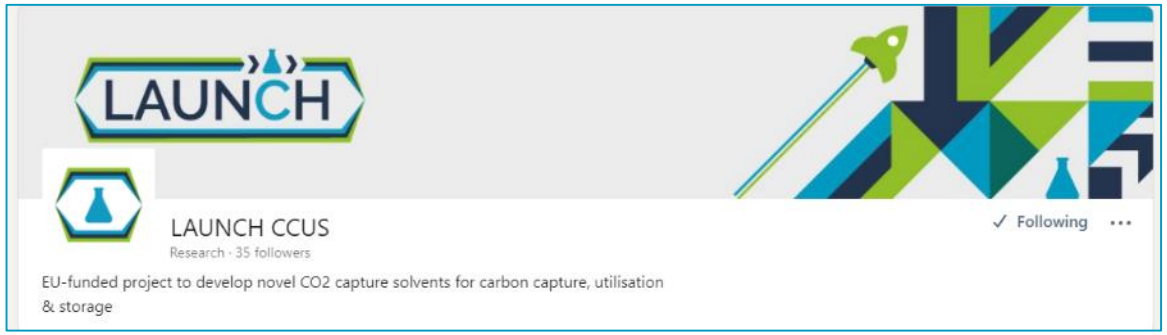
Project website: www.launchccus.eu

Twitter: @launchccus

LinkedIn: https://www.linkedin.com/company/launch-ccus/



Website



LinkedIn

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Acknowledgements

The LAUNCH project is funded through the ACT programme (Accelerating CCS Technologies, Horizon2020 Project No 299662).

Financial contributions have been made by Ministry of Economic Affairs and Climate Policy, the Netherlands; The Federal Ministry for Economic Affairs and Energy, Germany; Gassnova of Norway through the CLIMIT program; and the Department for Business, Energy & Industrial Strategy, UK, with extra funding from the US Department of Energy. All funders are gratefully acknowledged.

Now let’s move on into technology!

@launchccus

www.launchccus.eu

The war on amine oxidation

Why and What
Recent results by TxCMP
Future needs and plans

Gary T. Rochelle

Texas Carbon Management Program

The University of Texas at Austin

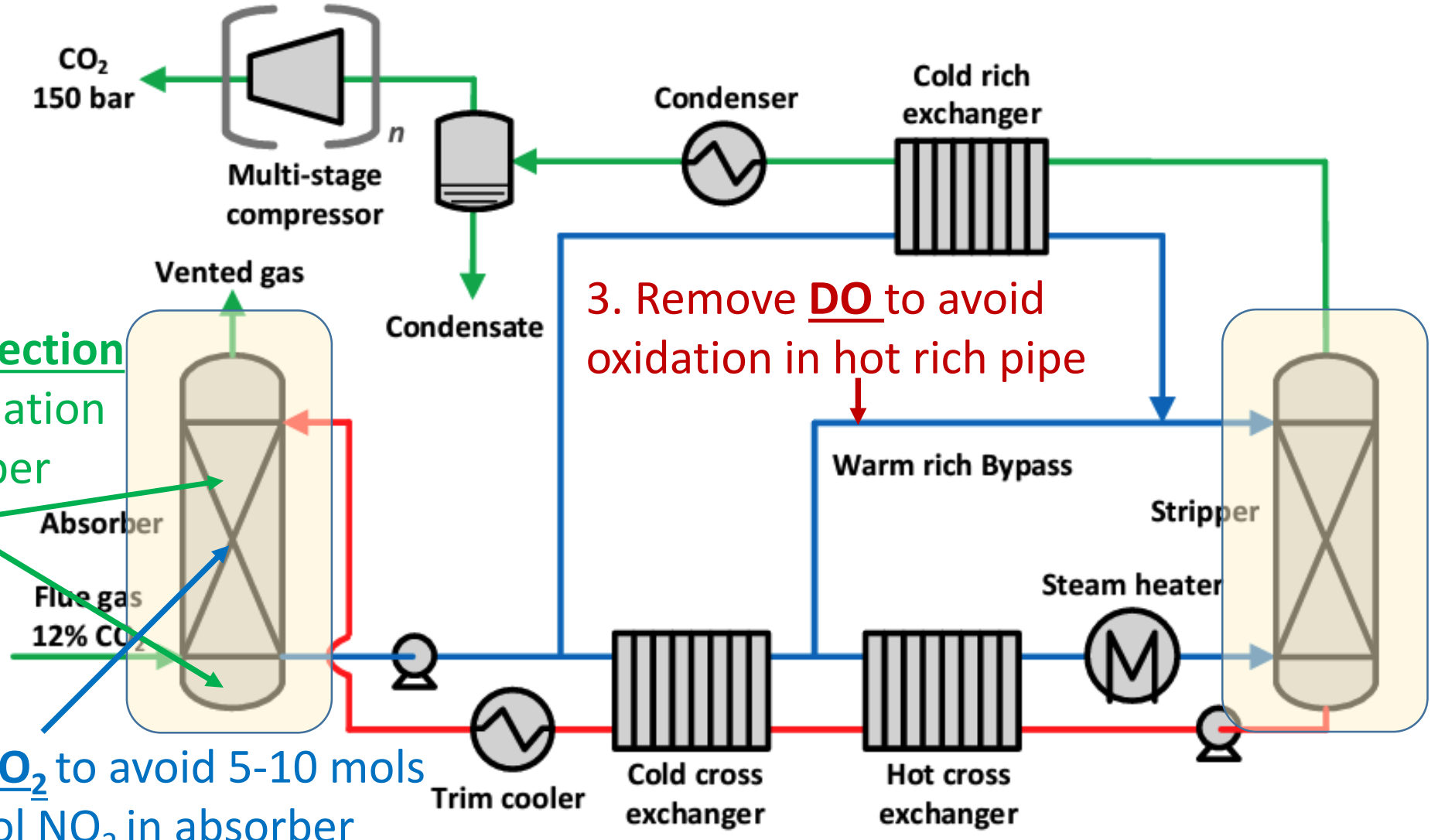
LAUNCH final meeting

2/15/2023

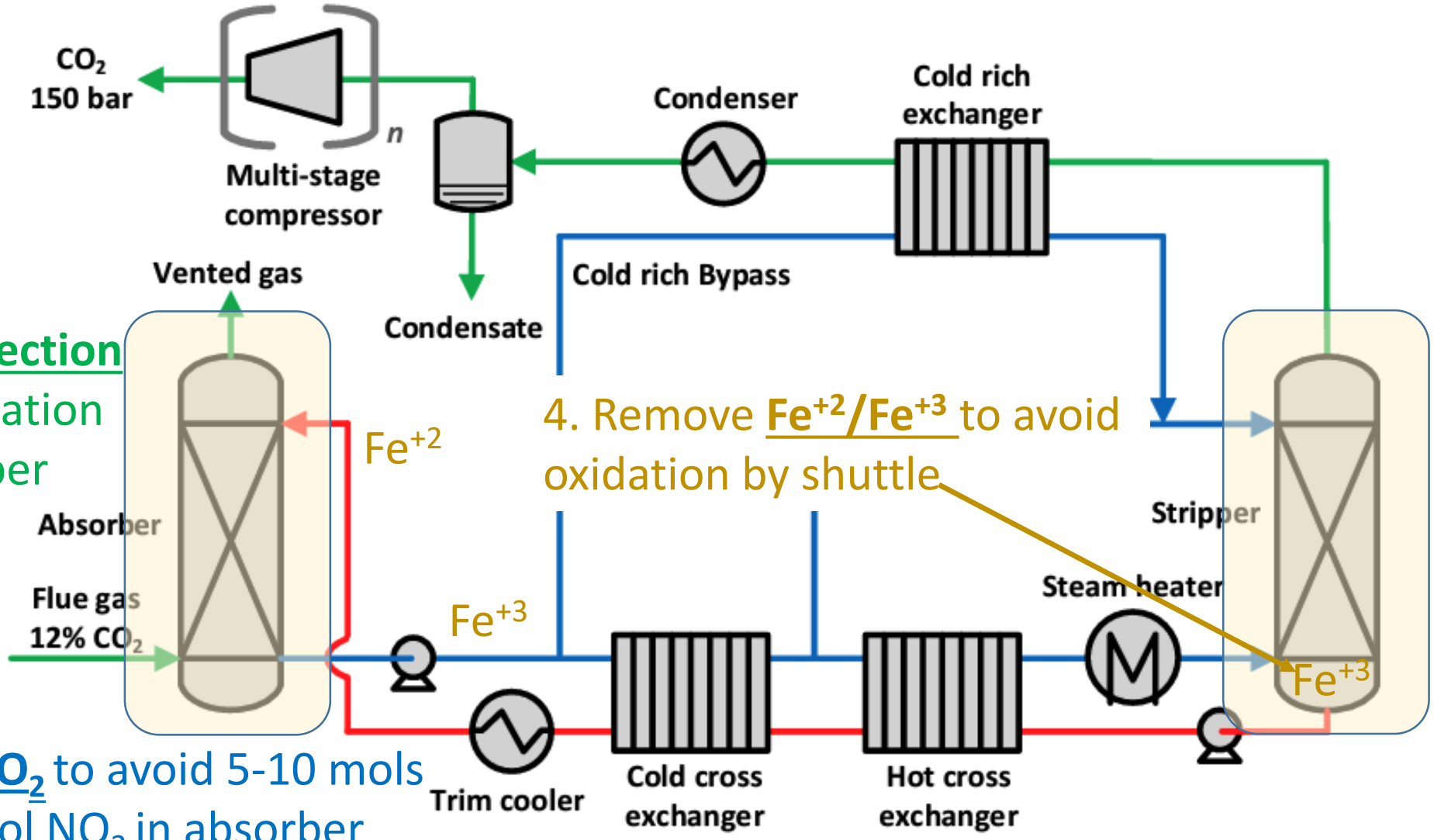
Why the war on amine oxidation?

- Oxidation is a significant environmental and economic risk in the deployment of 2G amine scrubbing
- After 11 PhD students at UT working on PZAS & other 2G solvents we are still asking:
  - How much of what we are going to produce & release? where?
  - What are its health & safety impacts?
- We must <u>understand</u> & <u>mitigate</u> amine oxidation with 2G amines to deploy CCS by PCC

Four Oxidation pathways to be mitigated



Four Oxidation mechanisms to be mitigated



1. **Amine Selection**
To avoid oxidation
In the absorber
& sump

4. Remove **Fe^{+2}/Fe^{+3}** to avoid oxidation by shuttle

2. Remove **NO_2** to avoid 5-10 mols oxidation/ mol NO_2 in absorber

PZ oxidation by NO_2 (catalytic hypothesis)

- **In Absorber (low T)**

$NO_2 + PZH + OH^- \rightarrow PZ\bullet + NO_2^- + H_2O$ (Free radical absorption)

PZ• + $O_2 \rightarrow$ PZOO•

PZOO• + PZH → **PZ•** + PZOOH

Chain reaction

2 **PZ•** + $H_2O \rightarrow$ PZH + PZOH (Termination)

PZOOH+PZH → 2PZOH

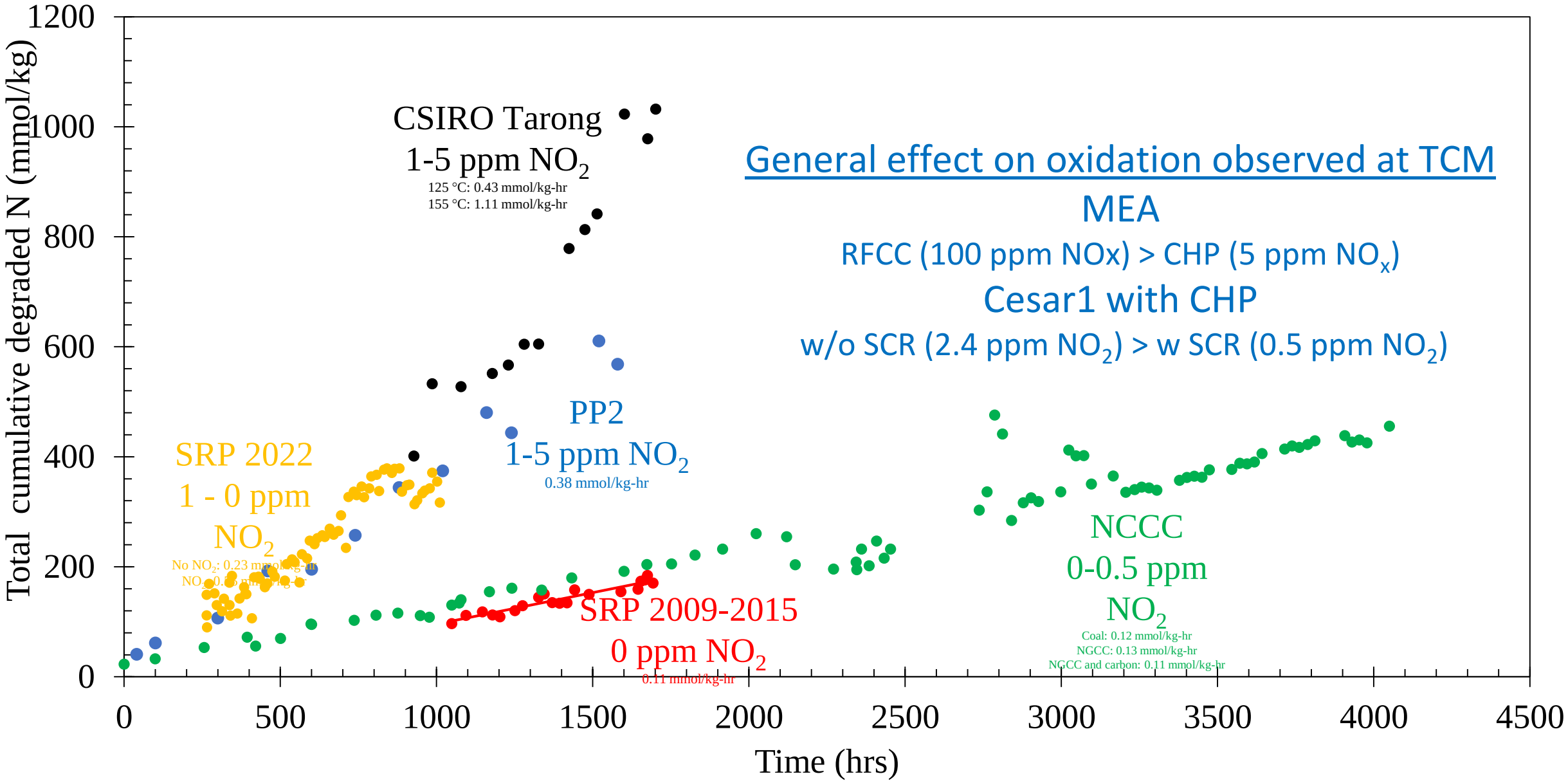
- **In Stripper (high T)**

$NO_2^- + PZCOOH \rightarrow MNPZ + HCO_3^-$

$MNPZ \xrightarrow{\Delta, base} PZOH + N_2O$

- **Sum:** NO_2 + n PZ → n PZOH + other products

PZ Oxidation in pilot plants w and w/o NO_2



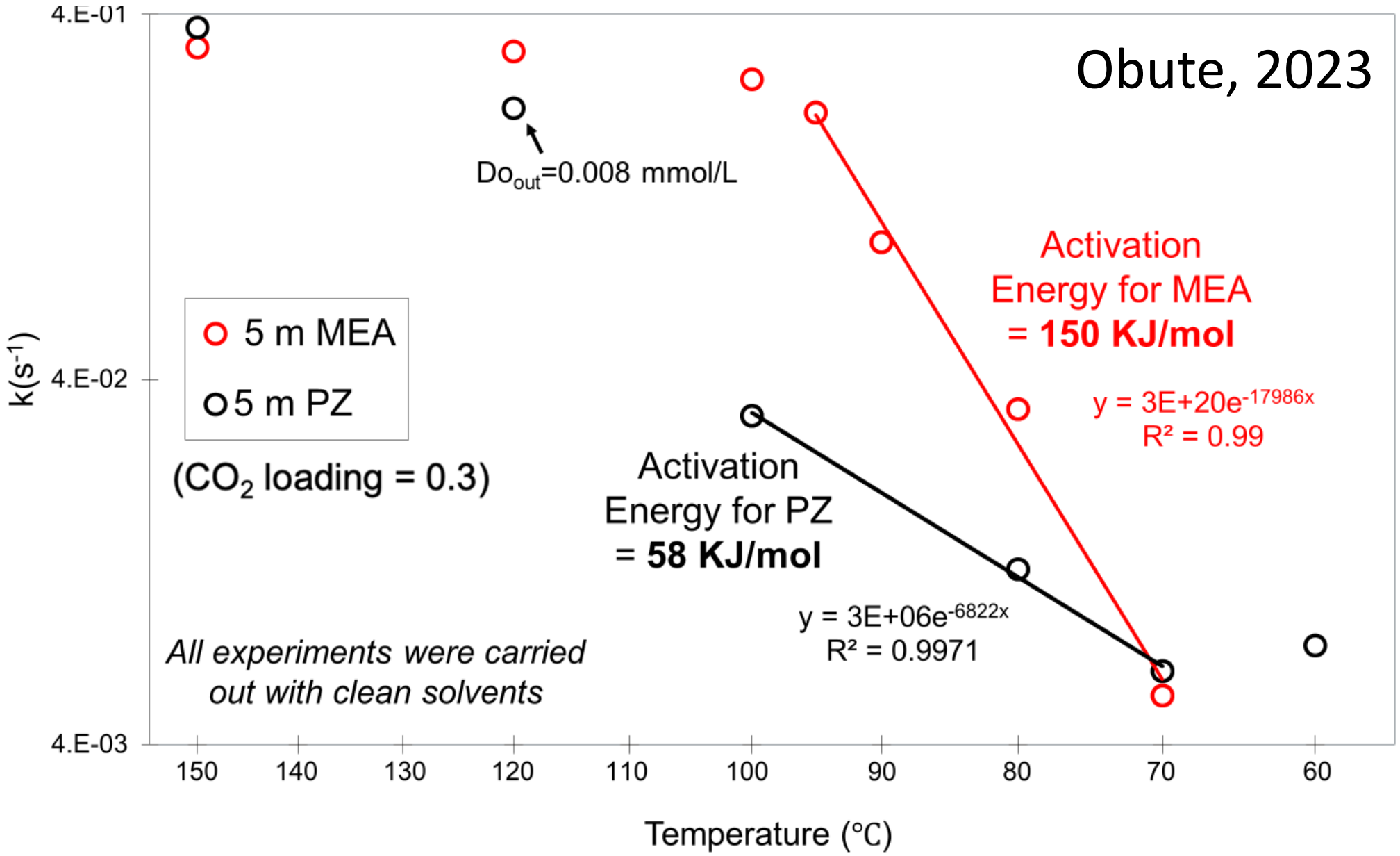
NO_2 may be less of an issue with MEA & CESAR1

- Addition of 15 ppm NO_2 in LAUNCH rig testing with MEA shows a small effect
  - Primary amines remove less NO_2 reducing the amount available for catalysis

- LAUNCH testing at Niederaussem showed no reduction of oxidation with NO_2 removal by sulfite scrubbing
  - NO_2 may be regenerated by NO oxidation in the duct
  - Nitrosamine accumulates in the solvent

DO consumption in 5m PZ and MEA

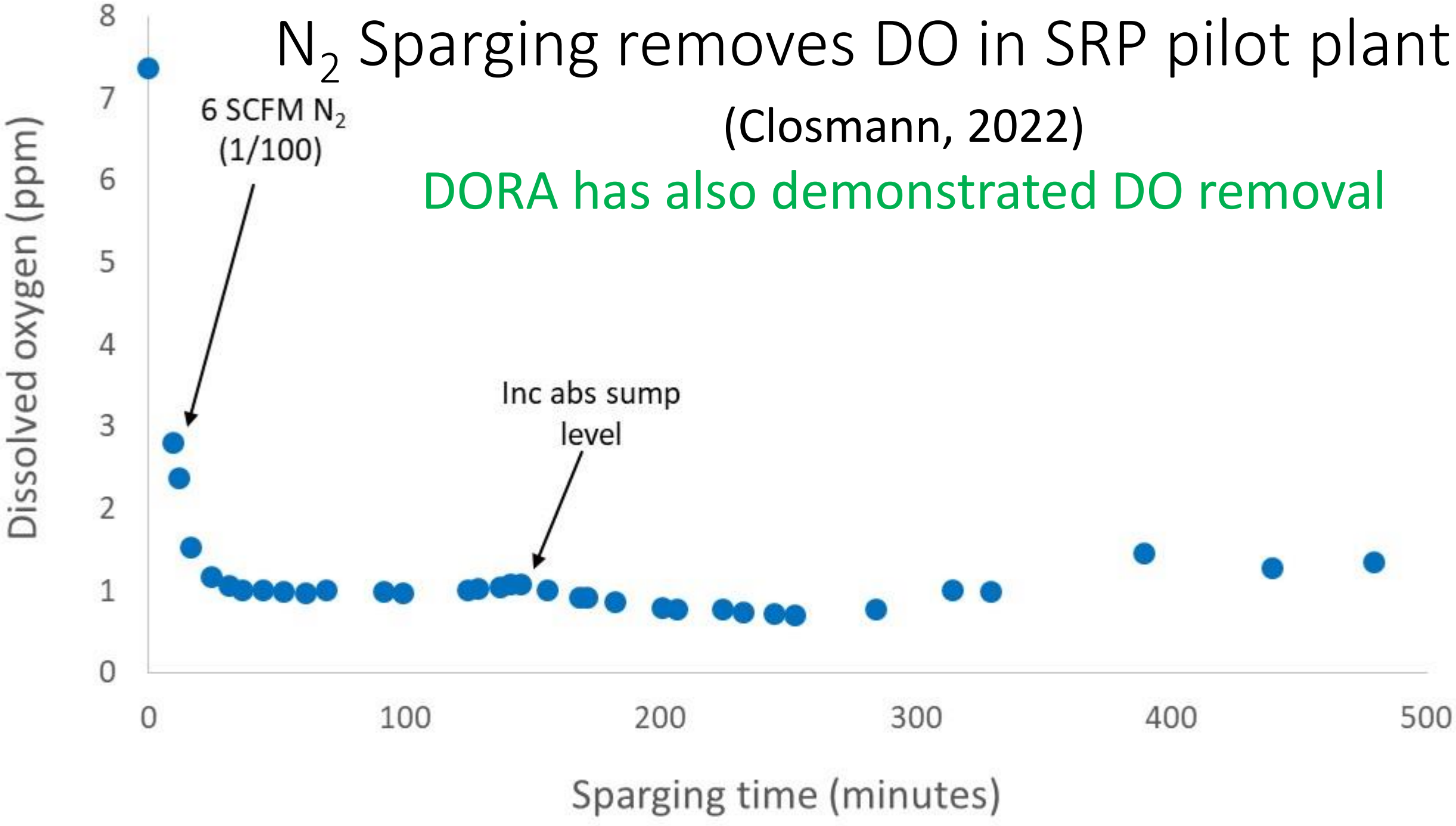
Extension of TNO method with a 20 s plug flow reactor at stripper T/P



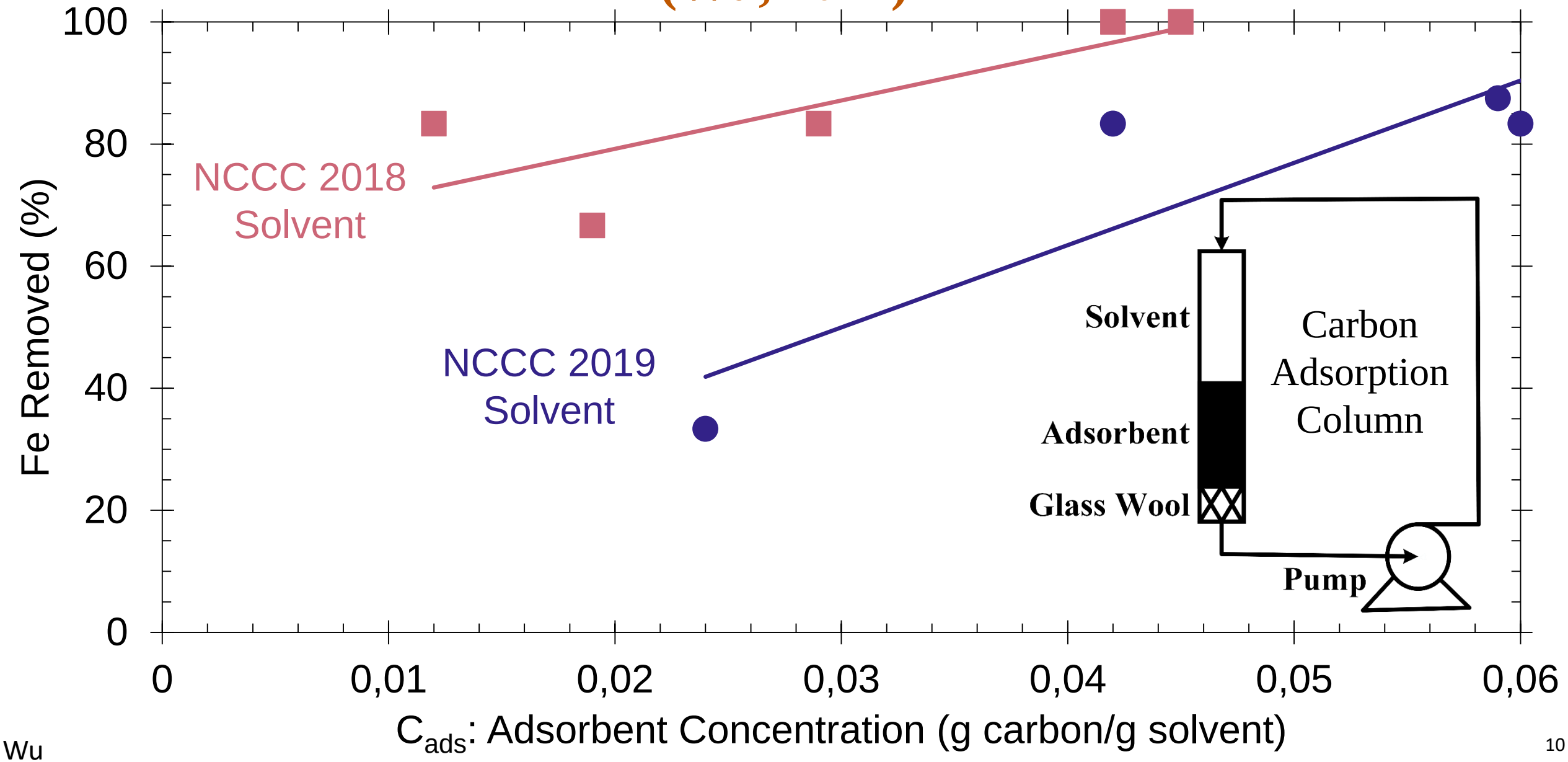
N_2 Sparging removes DO in SRP pilot plant

(Closmann, 2022)

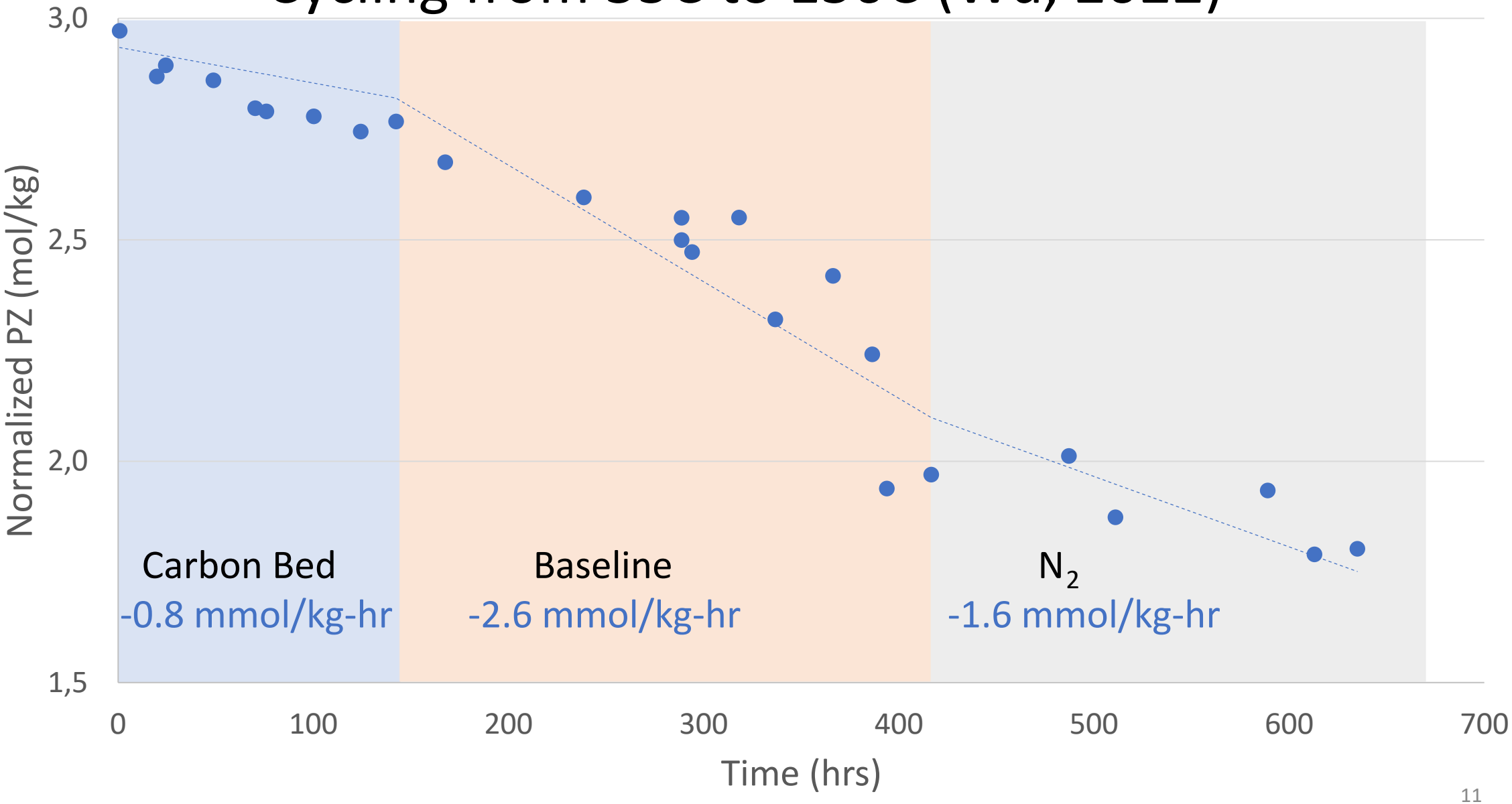
DORA has also demonstrated DO removal



C Treating Significantly Reduces Fe in Batch Experiments (Wu, 2022)



C Treating & N2 Sparging reduce PZ Oxidation in Bench HTOR Cycling from 55C to 150C (Wu, 2022)



2 Steps forward – 1 step back

- None of our current understanding appears to help in excellent long term testing of CESAR1 at Niederaussem (see presentations by Peter Moser)
  - Sulfite scrubbing removes NO_2
    - but has no effect on oxidation
  - Ion exchange removes metals and degradation products
    - but has no effect on oxidation
  - Carbon treating clears solution
    - but has no effect on oxidation
  - BUT CESAR1 oxidizes slowly even in long term testing
    - Solvent selection works

Deep dangerous pits in our understanding

- Volatile aldehydes (Acetaldehyde, Formaldehyde) (HAP's in U.S.)
  - Just starting to understand and report emissions
  - Few reported solution analyses
  - Practically no work on oxidation mechanisms
  - Need reporting of available data so we can all gain understanding
- Health and Safety properties of "cats and dogs"
  - Best to just not make them
- Incomplete solution analyses
  - Very few solvents have comprehensive analysis methods
  - Frequently missing: aldehydes, aminoacids, alcohols
  - We must close the material balance
- Comprehensive air emission results to ppb levels
  - A good start at limited conditions and sites with PTR-TOF-MS
  - Need to use PTR-TOF-MS in U.S.

Needs for pilot plant testing w 2G solvents

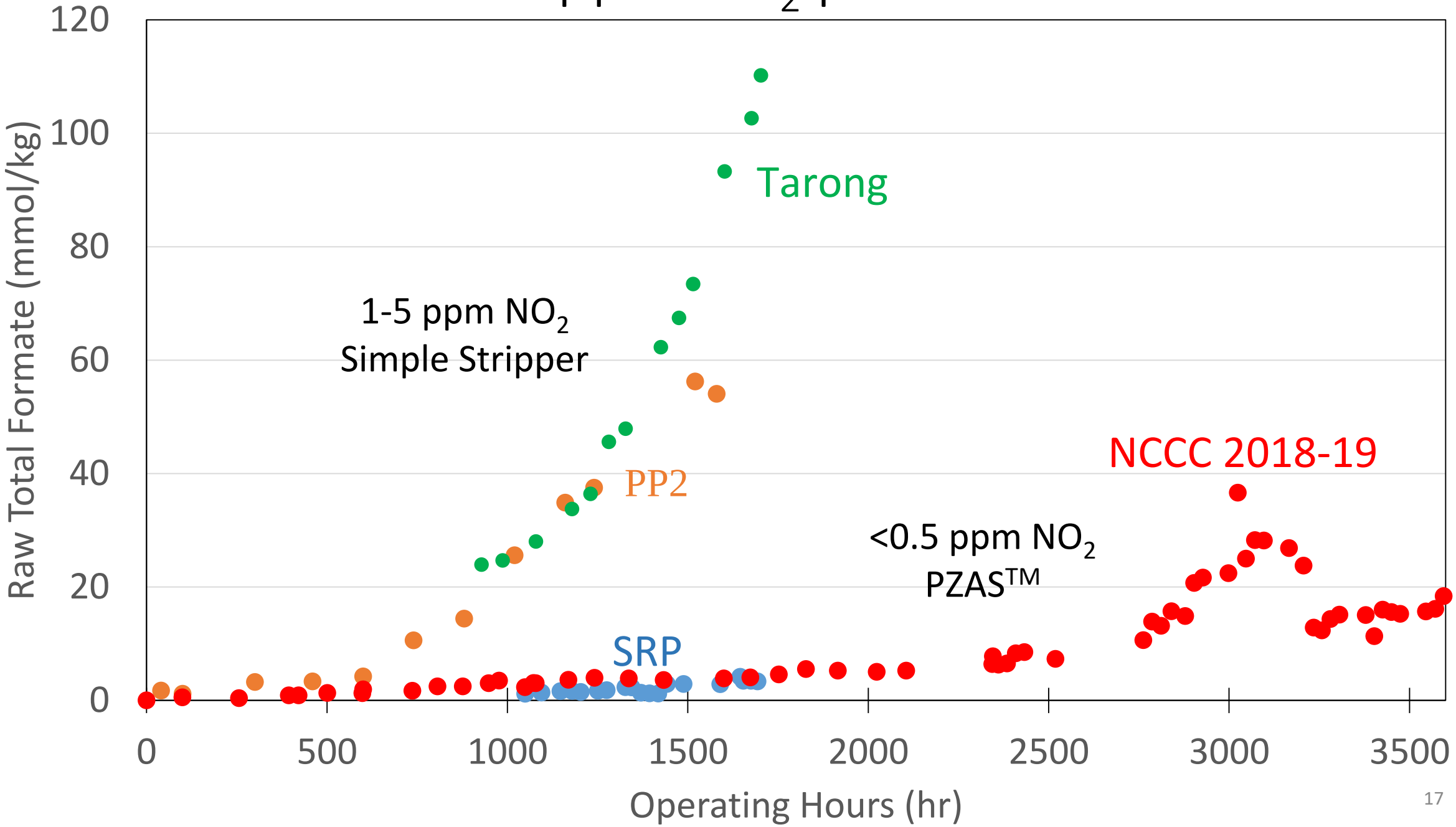
- Longterm testing of reclaiming with 2G solvents
  - WILL it mitigate oxidation (by Fe^{+2}/Fe^{+3})
- Sustained N_2 sparging or DORA
  - Will it reduce oxidation?
- More NO_2 Scrubbing and/or injection
  - Does NO_2 matter with all 2G solvents?

Future Work funded at UT by TxCMP & DOE

- Bench-scale
  - Effect of NO_2 at absorber conditions with multiple solvents
  - DO consumption rate at 40 to 150C with multiple solvents
  - Cyclic oxidation of PZ with C treating and Reclaiming
  - Adding Analytical methods for aldehydes, amino acids
- NCCC pilot – 6 months with PZ – NGCC conditions- 2023
  - N_2 Sparging
  - NO_2 prescrubbing
  - C treating
- Open to any and all Collaborations

Xtra slides

PZAS with<0.5 ppm NO_2 produces less formate



Four important oxidation pathways to be mitigated

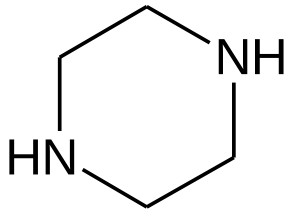
- 1. Poor amine selection (MEA) oxidize at absorber conditions
- 2. NO_2 oxidizes all amines at 0.2 to 5 ppm in the flue gas
- 2. Dissolved oxygen (DO) oxidizes amines at elevated T before the stripper.
- 3. Fe^{+3} oxidizes amines at stripper T & is regenerated from Fe^{+2} in absorber.

PZ oxidation by NO_2 (Fine, 2015)

- **In Absorber (low T)**

  $NO_2 + PZ \rightarrow PZ\bullet + NO_2^-$ (Free radical absorption)

  $2PZ\bullet \rightarrow PZOH + PZ$ (major)

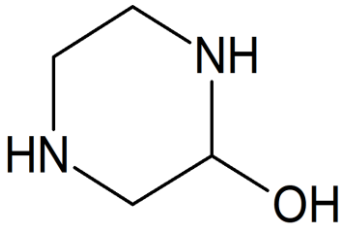


PZ

- **In Stripper (high T)**

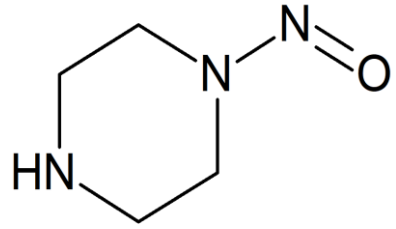
  $NO_2^- + PZCOOH \rightarrow MNPZ + HCO_3^-$

  $MNPZ \xrightarrow{\Delta,\ base} PZOH + N_2O$



PZOH

- **Sum:** $NO_2 + 1.5\ PZ \rightarrow 1.5\ PZOH + 0.5\ N_2O$



MNPZ

Hypotheses Radical propagation and NO_2 synergy with iron

Fine developed the unified mechanism for NO_2 reaction with amines (*)

Absorber

NO_2 absorption:

* $PZ + NO_2 \rightarrow PZ\cdot + NO_2^-$

Radical propagation:

$PZ\cdot + O_2 \rightarrow PZOO\cdot$

$R\cdot + PZ \rightarrow PZ\cdot + P$

$R\cdot$ = PZOO·, PZO· , and ·OH

P = PZOOH, PZOH, and H_2O

Radical termination:

* $2PZ\cdot + H_2O \rightarrow PZOH + PZ$

Iron oxidation:

$$Fe^{2+} \xrightarrow[DO,\ NO_2,\ \cdot OH,\ R\cdot]{} Fe^{3+}$$

Organoperoxy dissociation:

$PZOOH + PZ \rightarrow 2PZOH$

$PZOOH \rightarrow PZO\cdot + \cdot OH$

$Fe^{2+} + PZOOH \rightarrow Fe^{3+} + PZO^- + \cdot OH$

$Fe^{3+} + PZOOH \rightarrow Fe^{2+} + PZOO\cdot + H^+$

PZOH or similar products might go through the same reactions

NO_2 catalyzes free radical oxidation

NO_2 absorption:

* $PZ + NO_2 \rightarrow PZ\cdot + NO_2^-$

Radical propagation:

$PZ\cdot + O_2 \rightarrow PZOO\cdot$

$R\cdot + PZ \rightarrow PZ\cdot + P$

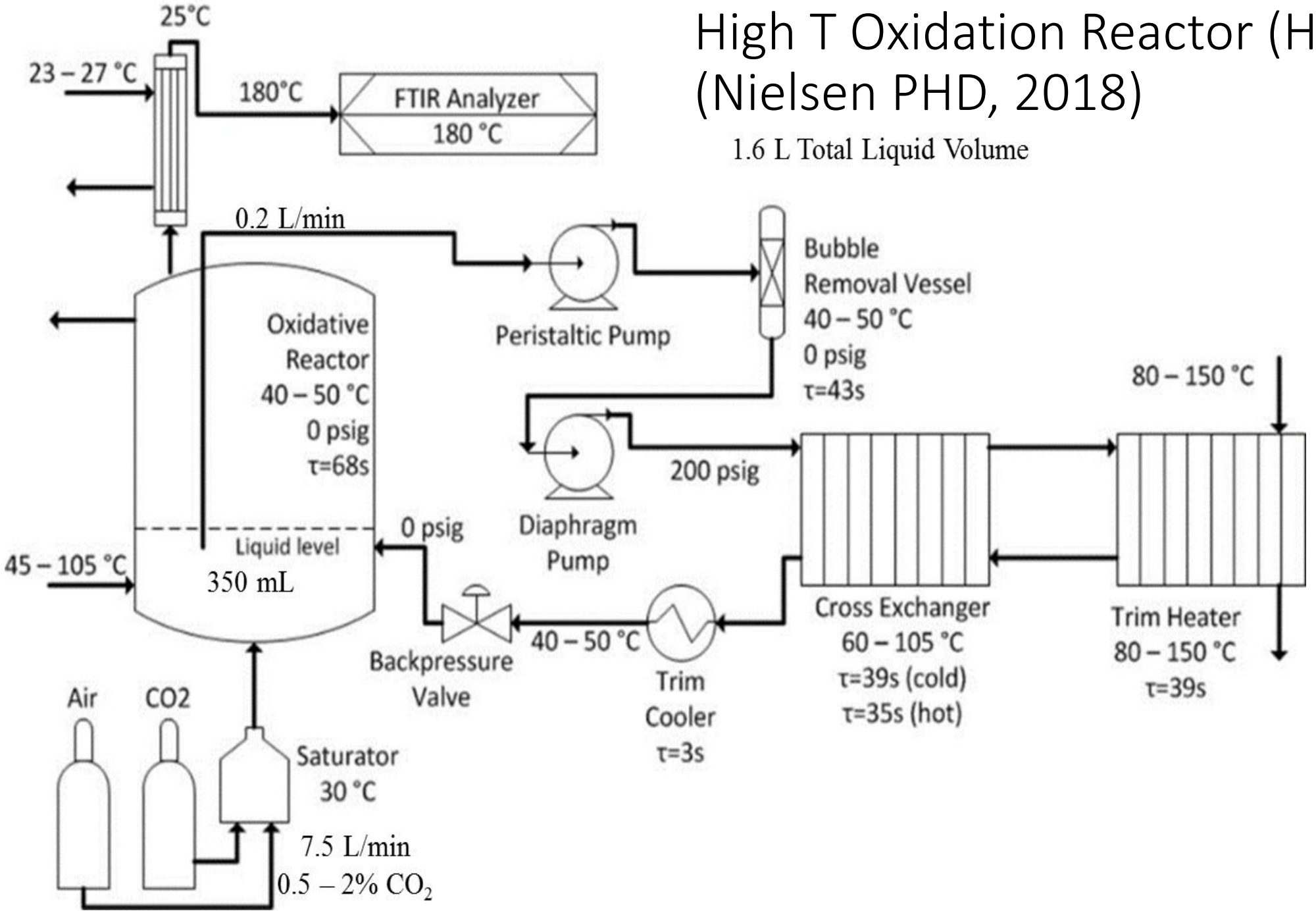
$R\cdot$ = PZOO·, PZO· , and ·OH

P = PZOOH, PZOH, and H_2O

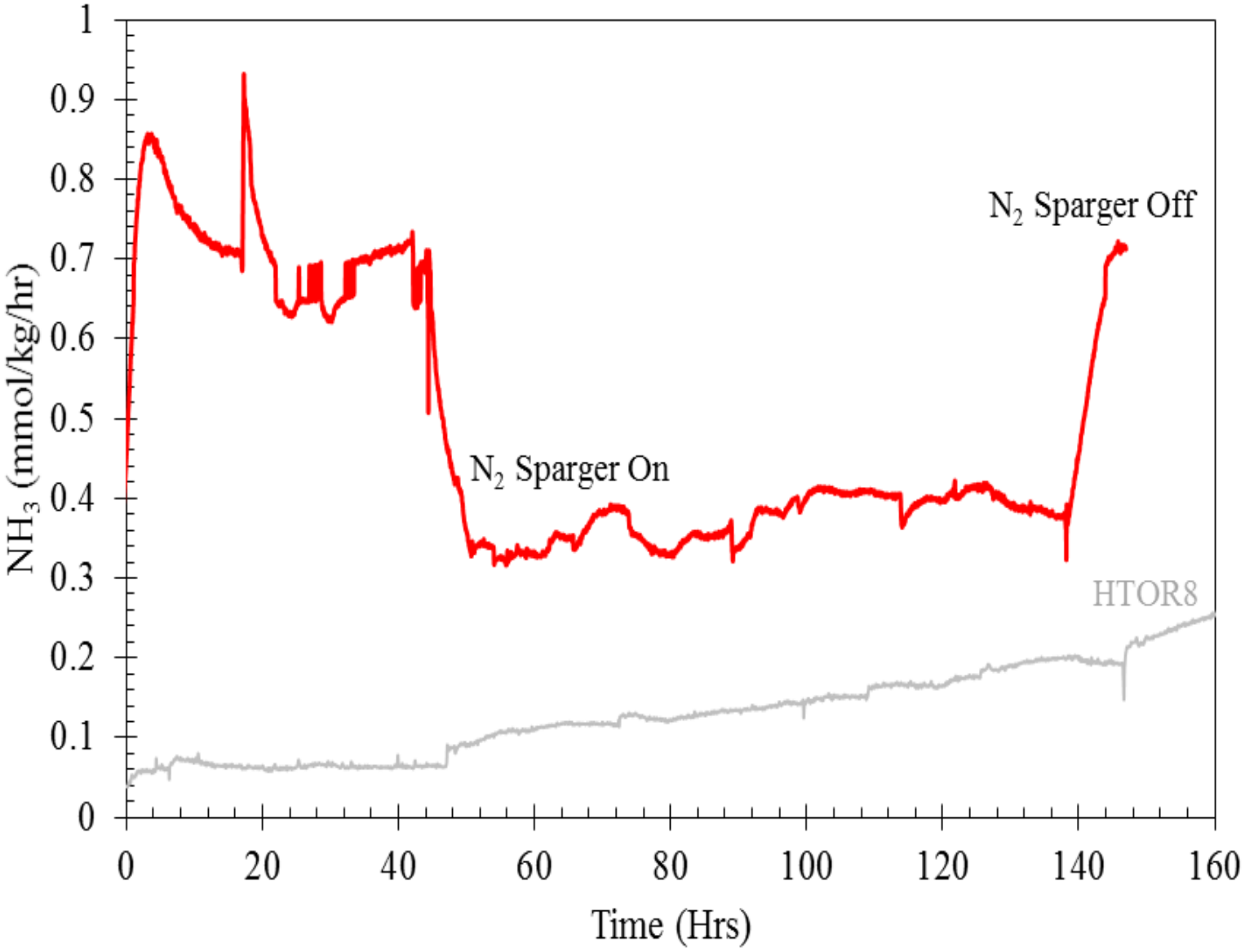
Radical termination:

* $2PZ\cdot + H_2O \rightarrow PZOH + PZ$

High T Oxidation Reactor (HTOR) (Nielsen PHD, 2018)



Oxidation decreases when Dissolved Oxygen is removes
4 m PZ from degraded CSIRO Tarong pilot plant inventory cycled from 40 to 150 °C
Solvent sparged with 1 L/min N_2 to remove dissolved oxygen from t = 44 to 138 hours.



2G amine scrubbing processes are here to stay. 3G processes will not be significantly better for PCC

- Amine Scrubbing was first, capital costs going down
- Energy improvement is done, >50% thermo efficiency
- 97+% CO_2 removal is easily achieved with 2G amine
- But environmental impact must be managed

PhD work at UT on amine oxidation

- Goff (2005) – Catalyzed by dissolved iron (MEA)
- Sexton (2008) – Complicated degradation products (MEA)
- Closmann (2011) – N_2 stripping reduces oxidation by DO at stripper T (MDEA)
- Freeman (2011) – PZ does not oxidize at absorber conditions
- Fine (2015) – NO_2 reacts with all amines to oxidize >1.5 mol/mol NO_2 (PZ et al.)
- Voice (2013) – Inhibitors do not work, oxidation at stripper T w/o dissolved O_2 (MEA, PZ, et al.)
- Nielsen (2018) – Degradation products solubilize iron & catalyze oxidation (PZ)
- Wu (2022) – Oxidation reduced by C treating (PZ)
- Plantz (2025) – Removing dissolved Fe (PZ)
- Chen (2025) – Oxidation catalyzed by NO_2 (PZ et al.)
- Obute (2026) – Kinetics with dissolved oxygen in hot rich solvent (PZ et al.)

Multilevel, multi-investigator simultaneous RD&D to win the war on oxidation (not just LAUNCH)

- Bench-scale - UT, SINTEF, NTNU, UKy, TNO
- Small pilot – UT, NCCC, Tiller, Niederaussem, TNO, Sheffield
- Large pilot – TCM
- Analytical support – SINTEF, Uky, UT

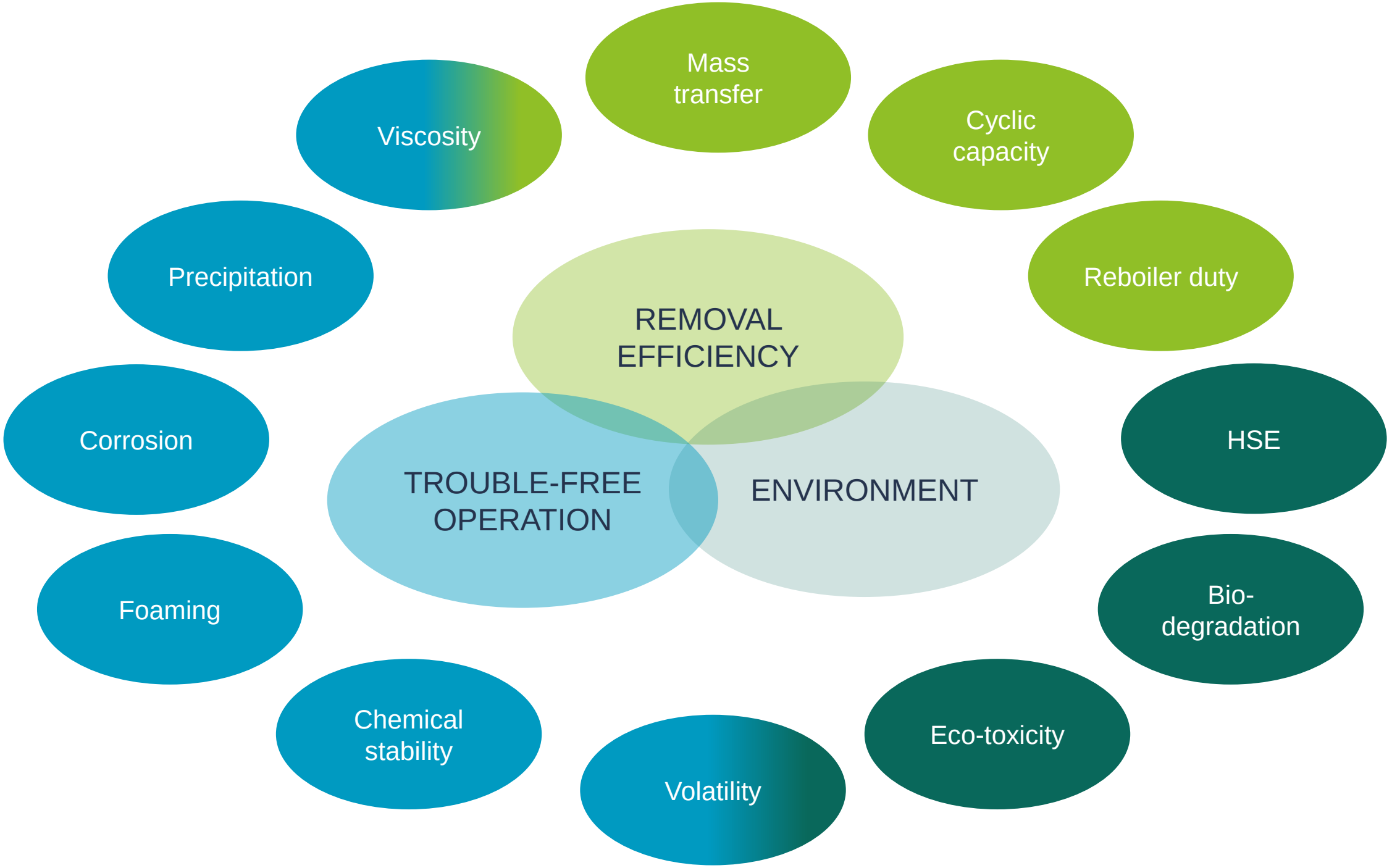
Mitigating Three important oxidation mechanisms

- 1. NO_2 oxidizes all amines at 0.2 to 5 ppm in the flue gas
  - Scrub NO_2 in existing SO_2 polishing scrubber with sulfite/thiosulfate.
    - Demonstrated to reduce NO2 at NCCC and Niederaussem, but did not reduce oxidation at Niederassem
  - Utilize SCR on all sources
- 2. Dissolved oxygen oxidizes amines at elevated T before the stripper.
  - Remove DO in rich solvent with nitrogen with sparging or membrane
    - DO removal demonstrated, impact on oxidation is unclear
  - Reduce exposure time and T (test site specific)
- 3. Fe^{+3} oxidizes amines at stripper T and is regenerated from Fe^{+2} in absorber.
  - Remove dissolved iron by adsorption
    - C treating seems to work with PZ at NCCC, but not at Niederaussem
    - Anion exchange resin at Niederaussem removes iron but does not reduce oxidation
  - Remove degradation products that solubilize iron
    - Continuous reclaiming not tested in LAUNCH

Open event, 15th of February 2023

Solvent qualification

Hanna K. Knuutila

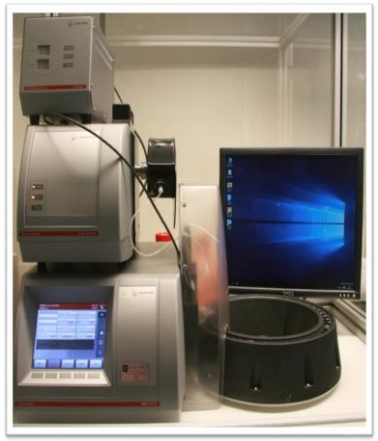

Mass transfer
Viscosity
Cyclic capacity
Precipitation
Reboiler duty
REMOVAL EFFICIENCY
Corrosion
HSE
TROUBLE-FREE OPERATION
ENVIRONMENT
Foaming
Bio-degradation
Chemical stability
Eco-toxicity
Volatility

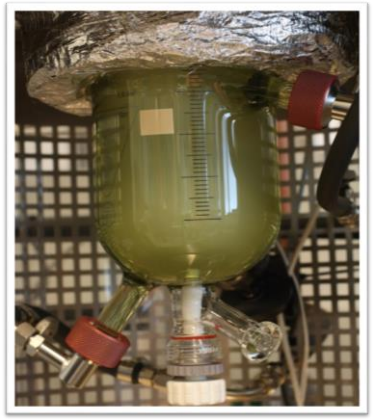
Solvent qualification – Removal efficiency

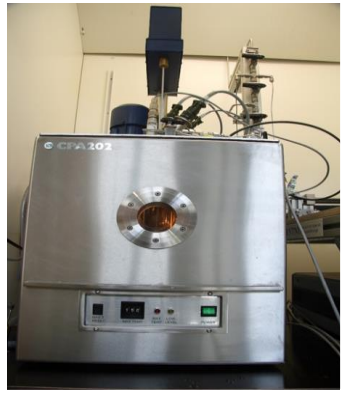
- Rate of reaction/mass transfer → Absorption heights

- Gas/liquid equilibria
- Heat of reaction
- Cyclic capacity

→ Regeneration energy requirement
→ Pumping costs







Solvent qualification – Removal efficiency

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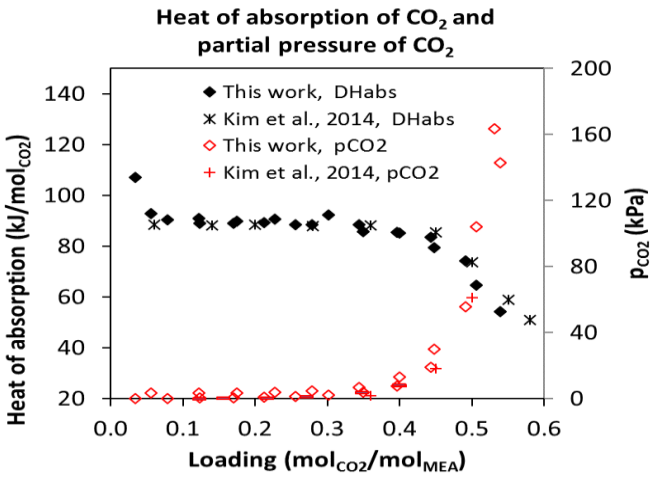
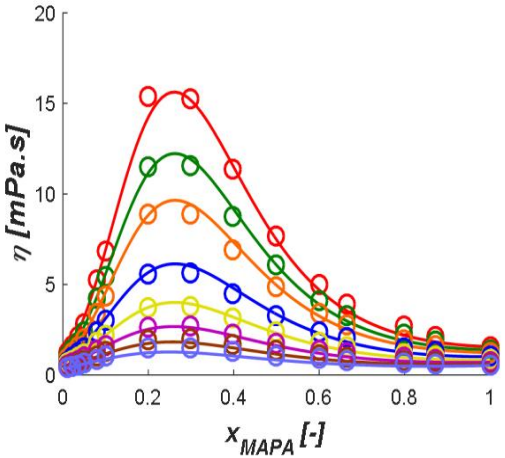
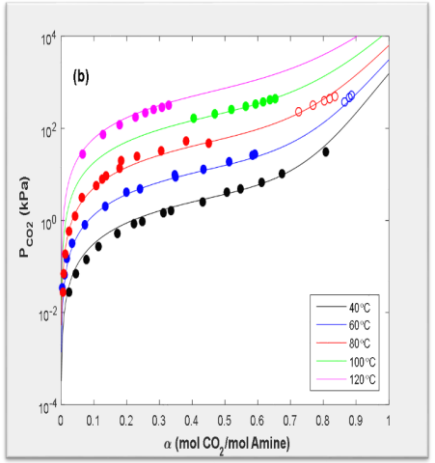
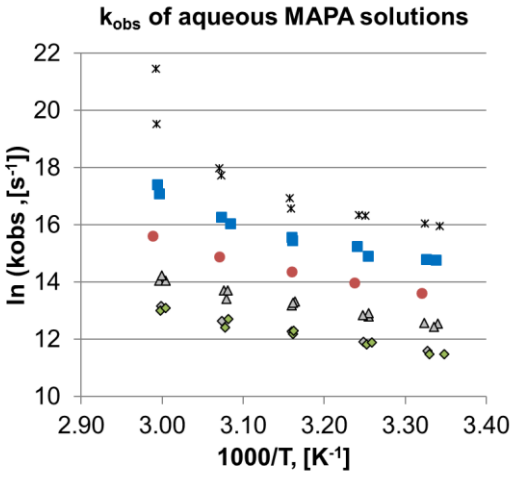








Solvent qualification – solvent vapor pressure and volatility losses

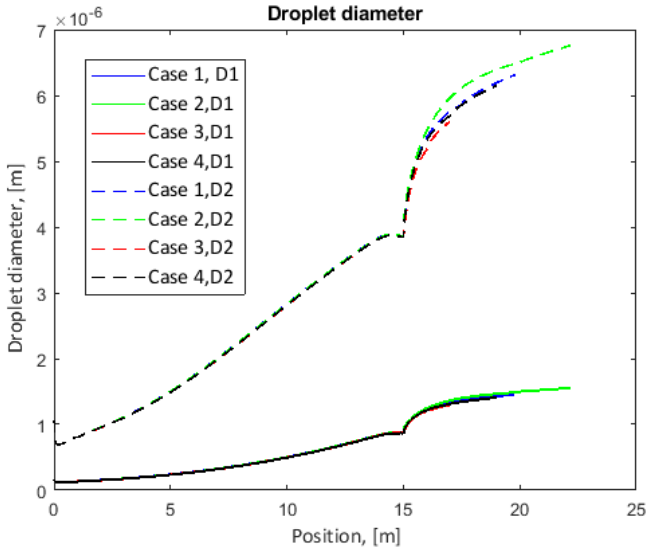
Solvent vapor pressure leads to the cost related to emission control and solvent replacement cost.

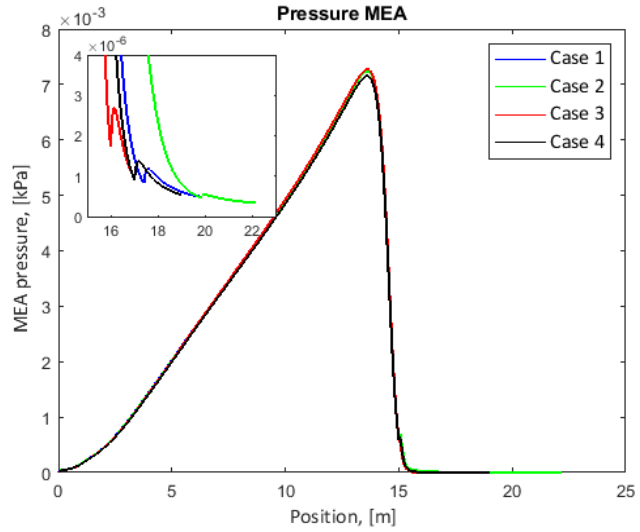
Volatile emissions, like amine, ammonia, aldehydes, can be controlled by

- Good design of water wash
- Use of staged water wash
- Use acid wash

Mist formation can lead to high amine losses and need to be avoided or controlled.

- Can be avoided by removing particles and Sulphur compounds as gas pretreatment
- Can be controlled with several technologies like operating conditions combined dry bed and Brownian demisters.

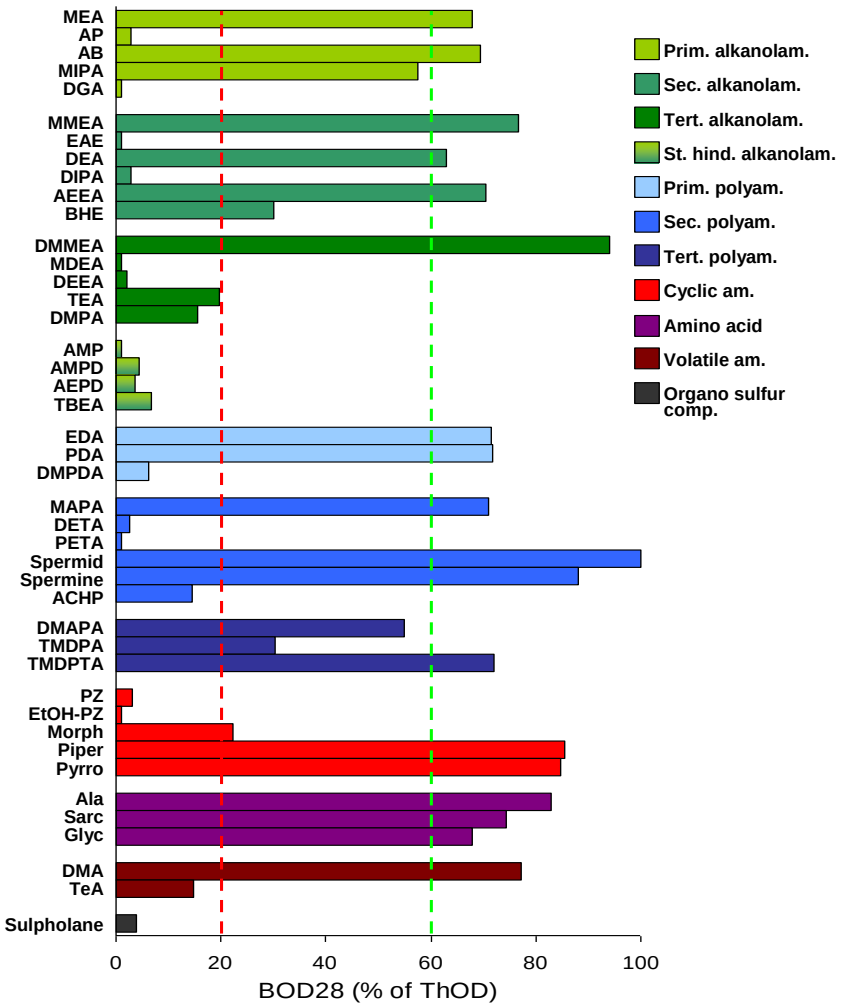




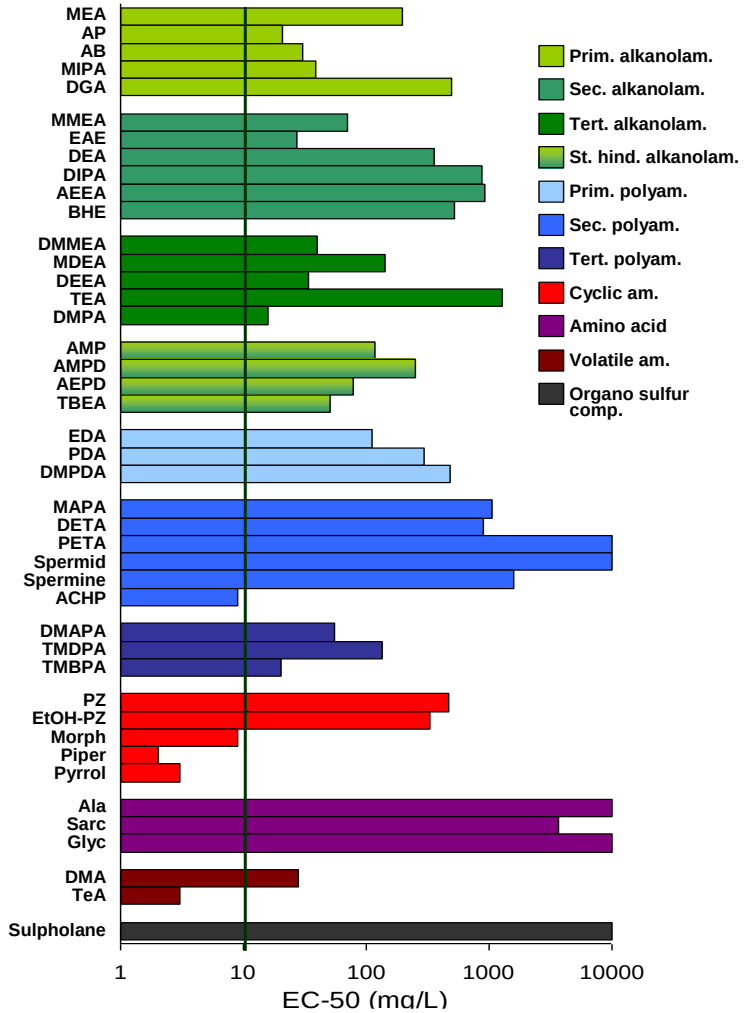
Case 1: WW1 2,4m, WW2 2,4m
Case 2: WW1 4,8m, WW2 2,4m

Solvent qualification – toxicity and biodegradation

(Eco)toxicity



Biodegradation



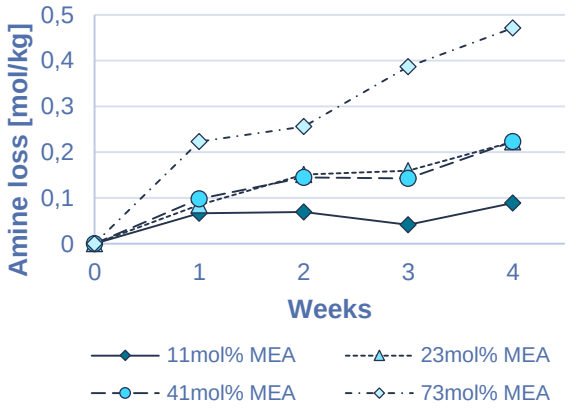
Solvent qualification – solvent stability and corrosion

Laboratory tests:

- Oxidative degradation tests
- Thermal degradation tests
- Cyclic lab systems with artificial gas

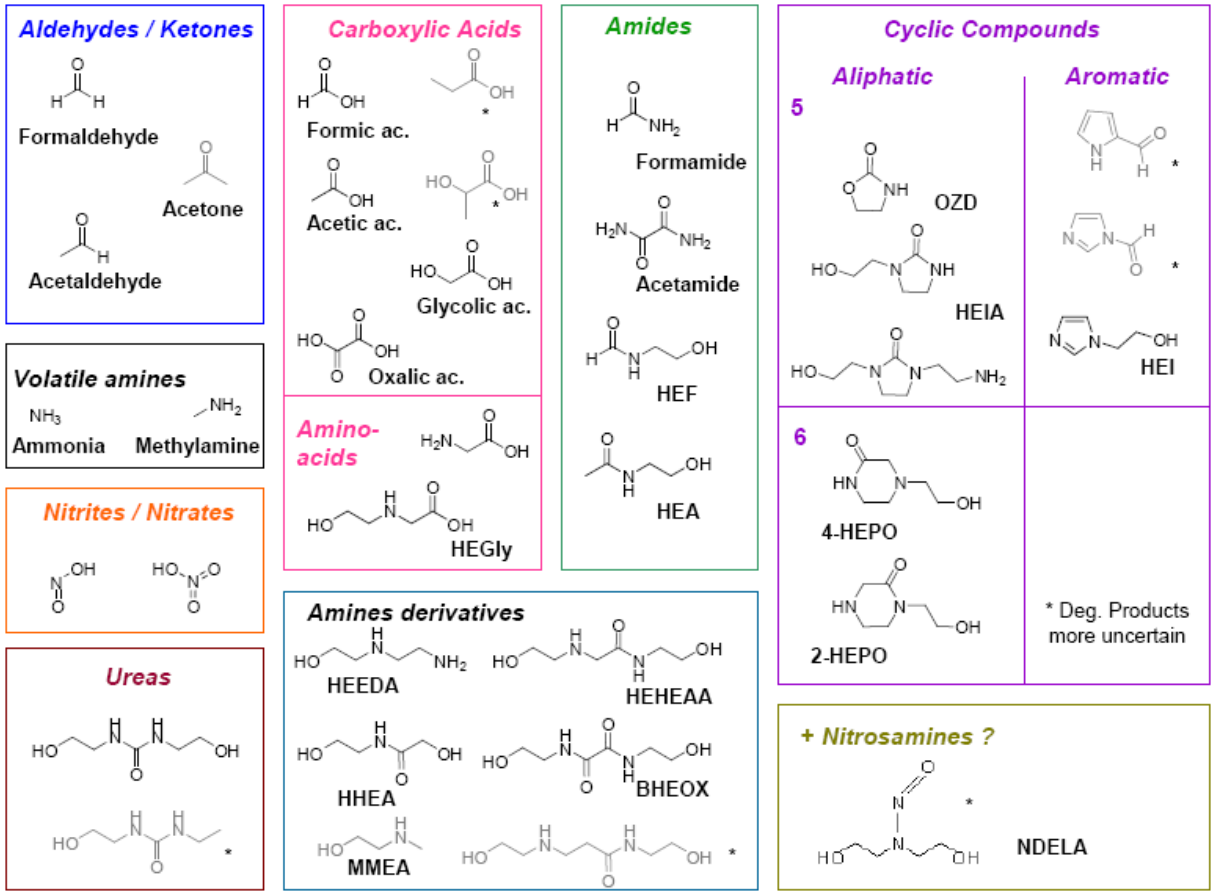
Focus on

- Identification of degradation compounds
- Closing nitrogen balance (ensure all major degradation compounds are known)
- Identify degradation compounds that are important from an HSE or operational point of view (like volatile degradation compounds) to know what needs to be monitored and/or controlled



Solvent qualification – solvent stability and corrosion

Understanding degradation requires (the development of) analytical methods to identify and quantify the main degradation compounds



Solvent qualification – piloting

Small pilots for long-term testing to understand degradation under real and relevant gas

Larger pilots

- Testing and qualifying emissions of amine and volatile degradation compounds
- Formation of degradation compounds (and reclaiming)
- Energy numbers

Conclusions

Solvent qualification should include both process and solvent performance.

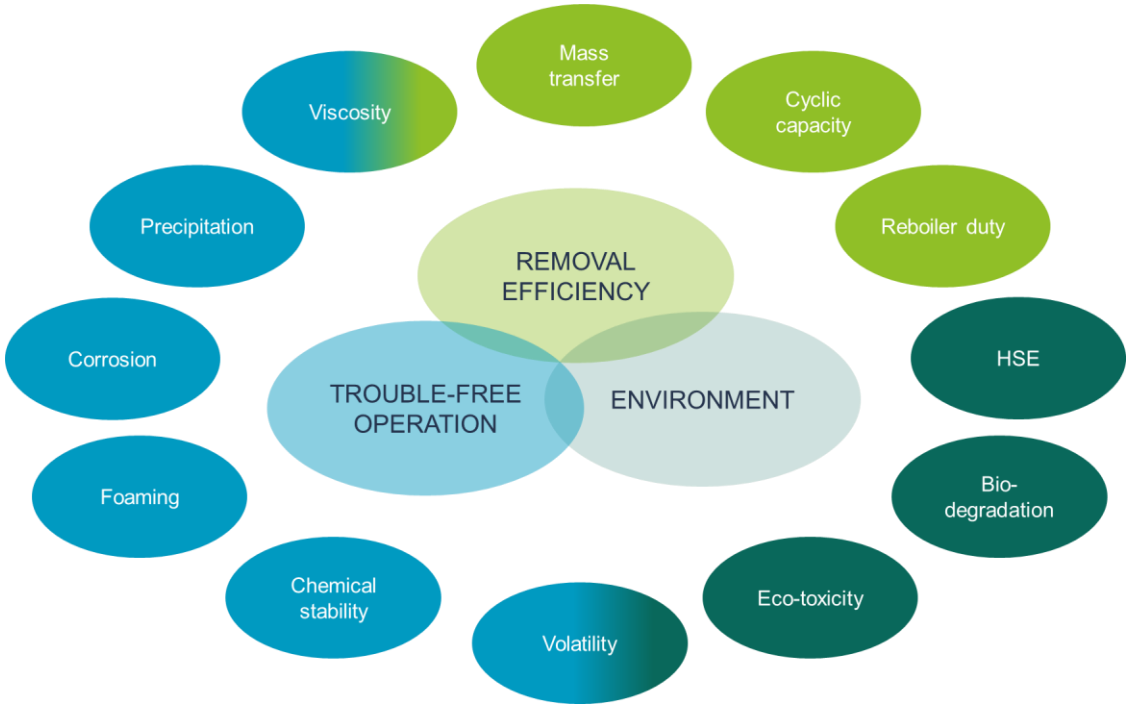
Solvent degradation is a potential show-stopper and should be addressed early during solvent development.

Lab-scale degradation experiments are needed to get an overview of degradation and to understand trends.

Piloting with real flue gas is needed to qualify the solvents.

All main degradation compounds should be identified (close nitrogen balance), and methods to quantify those must be developed.

Emission control of amine and volatile degradation compounds



Acknowledgements

The LAUNCH project (Project No. 299662) is co-funded by the ERA-NET Accelerating CCS Technologies initiative, which supports the delivery of safe and cost-effective carbon capture, utilisation and storage. The governments of each participating country have contributed funding through the ACT2 initiative.

Financial contributions have been made by Netherlands Enterprise Agency, the Netherlands; The Federal Ministry for Economic Affairs and Energy, Germany; The Research Council of Norway, Norway; and the UK Department for Business, Energy & Industrial Strategy, UK, with extra funding from the US Department of Energy. All funders are gratefully acknowledged.

LAUNCH

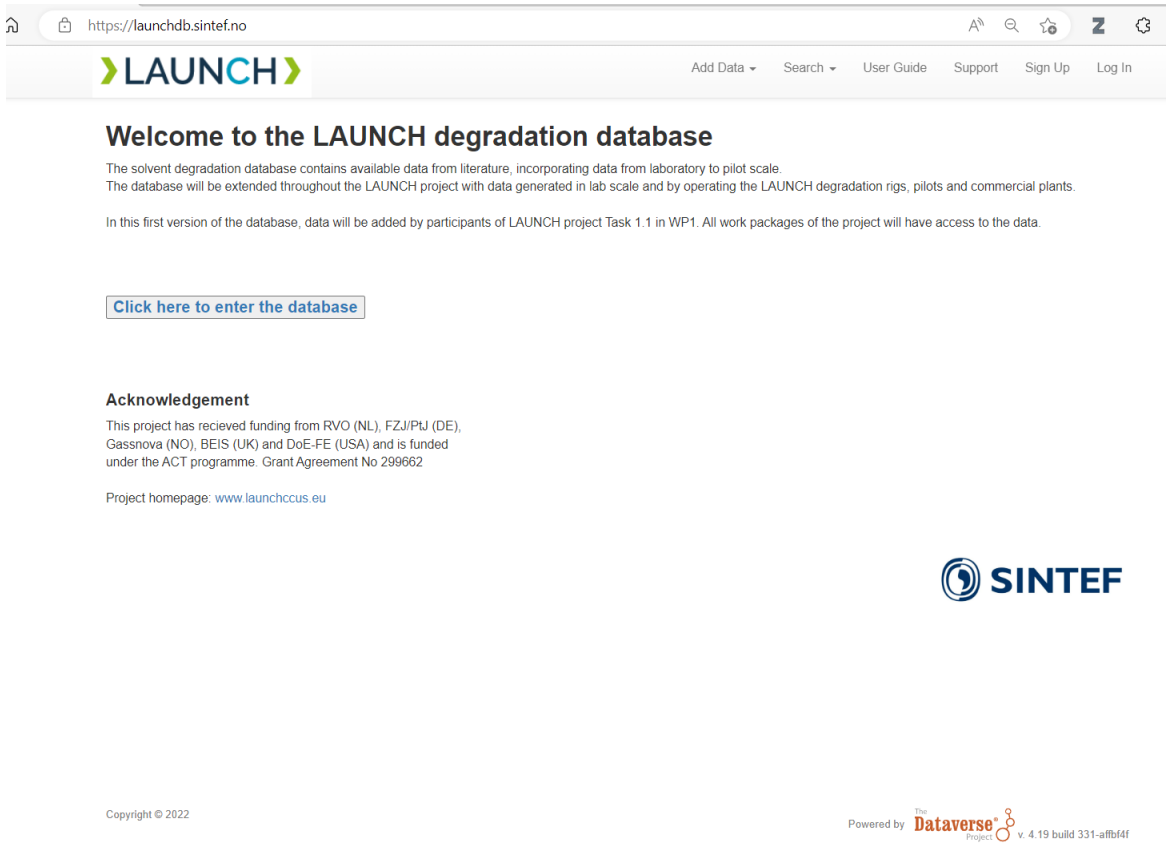
LAUNCH Open Event 15th February 2023

Solvent degradation Database

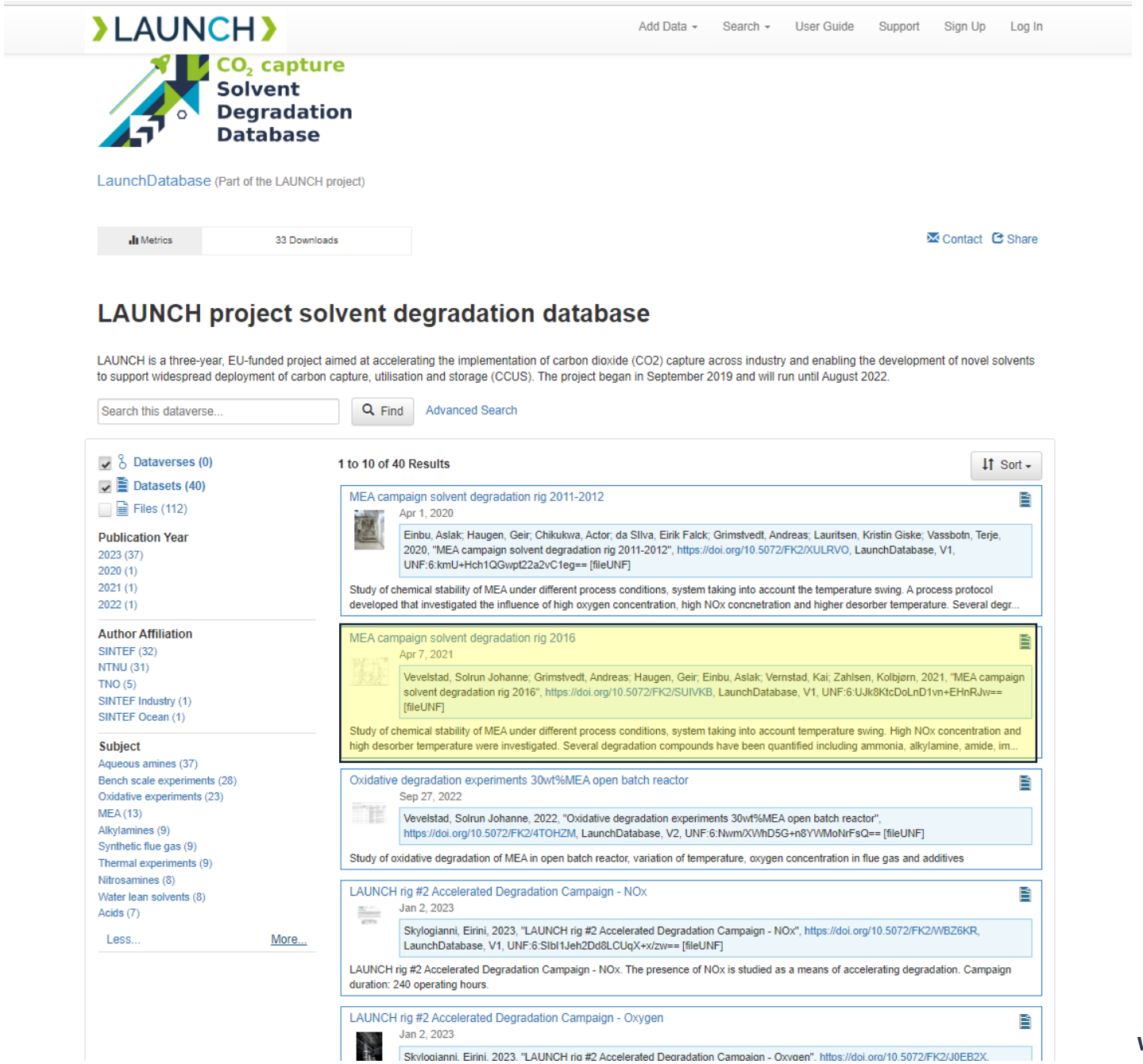
Andreas Grimstvedt - SINTEF Industry

Solvent degradation Database

- Dataverse - Open source research data repository software framework (https://dataverse.org/)
- Custom LAUNCH Dataverse repository established
- Database with web portal and REST-API deployed on Azure Cloud with SINTEF access control (https://launchdb.sintef.no/)
- Will be made public at the end of LAUNCH project



The Dataverse® Project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LAUNCH project solvent degradation database
LAUNCH is a three-year, EU-funded project aimed at accelerating the implementation of carbon dioxide (CO2) capture across industry and enabling the development of novel solvents to support widespread deployment of carbon capture, utilisation and storage (CCUS). The project began in September 2019 and will run until August 2022.
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MEA campaign solvent degradation rig 2011-2012
Apr 1, 2020
Einbu, Aslak; Haugen, Geir; Chikukwa, Actor; da SIlva, Eirik Falck; Grimstvedt, Andreas; Lauritsen, Kristin Giske; Vassbotn, Terje, 2020, "MEA campaign solvent degradation rig 2011-2012", https://doi.org/10.5072/FK2/XULRVO, LaunchDatabase, V1, UNF:6:kmU+Hch1QGwpt22a2vC1eg== [fileUNF]
Study of chemical stability of MEA under different process conditions, system taking into account the temperature swing. A process protocol developed that investigated the influence of high oxygen concentration, high NOx concnetration and higher desorber temperature. Several degr...
MEA campaign solvent degradation rig 2016
Apr 7, 2021
Vevelstad, Solrun Johanne; Grimstvedt, Andreas; Haugen, Geir; Einbu, Aslak; Vernstad, Kai; Zahlsen, Kolbjørn, 2021, "MEA campaign solvent degradation rig 2016", https://doi.org/10.5072/FK2/SUIVKB, LaunchDatabase, V1, UNF:6:UJk8KtcDoLnD1vn+EHnRJw== [fileUNF]
Study of chemical stability of MEA under different process conditions, system taking into account temperature swing. High NOx concentration and high desorber temperature were investigated. Several degradation compounds have been quantified including ammonia, alkylamine, amide, im...
Oxidative degradation experiments 30wt%MEA open batch reactor
Sep 27, 2022
Vevelstad, Solrun Johanne, 2022, "Oxidative degradation experiments 30wt%MEA open batch reactor", https://doi.org/10.5072/FK2/4TOHZM, LaunchDatabase, V2, UNF:6:Nwm/XWhD5G+n8YWMoNrFsQ== [fileUNF]
Study of oxidative degradation of MEA in open batch reactor, variation of temperature, oxygen concentration in flue gas and additives
LAUNCH rig #2 Accelerated Degradation Campaign - NOx
Jan 2, 2023
Skylogianni, Eirini, 2023, "LAUNCH rig #2 Accelerated Degradation Campaign - NOx", https://doi.org/10.5072/FK2/WBZ6KR, LaunchDatabase, V1, UNF:6:SIbl1Jeh2Dd8LCUqX+x/zw== [fileUNF]
LAUNCH rig #2 Accelerated Degradation Campaign - NOx. The presence of NOx is studied as a means of accelerating degradation. Campaign duration: 240 operating hours.
LAUNCH rig #2 Accelerated Degradation Campaign - Oxygen
Jan 2, 2023
Skylogianni, Eirini, 2023, "LAUNCH rig #2 Accelerated Degradation Campaign - Oxygen", https://doi.org/10.5072/FK2/J0EB2X,

MEA campaign solvent degradation rig 2016

Version 1.0

Vevelstad, Solrun Johanne; Grimstvedt, Andreas; Haugen, Geir; Einbu, Aslak; Vernstad, Kai; Zahlsen, Kolbjørn, 2021, "MEA campaign solvent degradation rig 2016", https://doi.org/10.5072/FK2/SUIVKB, LaunchDatabase, V1, UNF:6:UJk8KtcDoLnD1vn+EHnRJw== [fileUNF]

Cite Dataset

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Dataset Metrics

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Description Study of chemical stability of MEA under different process conditions, system taking into account temperature swing. High NOx concentration and high desorber temperature were investigated. Several degradation compounds have been quantified including ammonia, alkylamine, amide, imidazole, piperazinone, nitrosamine, pyrazine, urea and several more. (2016-08-23)

Subject Aqueous amines; MEA; Synthetic flue gas; Bench scale pilot; HSE; Nitrosamines; Alkylamines; Ammonia

Keyword Chemical stability, cycled system, MEA, degradation compounds

Related Publication Results from MEA campaign using SDR rig. A large set of degradation compounds from MEA have been quantified, for example MEA urea. Nitrogen and nitrosamine balance included. issn: 1876-6102

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Rekvisisjon	Journal nr	Sample id	Week	Operation days (total days)	Desorber T	Unit	Flue gas oxygen content	Unit	Flue gas Nox content	Unit	Absorber liquid temperature	Unit	Lean loading	Unit	Rich loading	Unit	Alkalinity	Unit	Ar
20110244	P112631	Unused	0	0													4.513	mol amine/kg	
20110244	P112632	LEAN Week 0	0		120	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	4.410	mol amine/kg	
20110244	P112633	LEAN Week 1	1		120	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	4.267	mol amine/kg	
20110244	P112635	LEAN Week 3	3		120	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	not analyzed	mol amine/kg	
20110244	P112637	LEAN Week 5	5	26 (35)	120	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	not analyzed	mol amine/kg	
20110286	P113055	LEAN Week 7	7		120	°C	18	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	not analyzed	mol amine/kg	
20110286	P113056	LEAN Week 8	8	16 (17)	120	°C	18	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	3.855	mol amine/kg	
20120013	P12119	LEAN Week 10	10		140	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	not analyzed	mol amine/kg	
20120013	P12120	LEAN Week 11	11	21 (21)	140	°C	12	mol%	5	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	3.540	mol amine/kg	
20120041	P12400	LEAN Week 13	13		120	°C	12	mol%	50	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	3.225	mol amine/kg	
20120041	P12401	LEAN Week 14	14	20 (22)	120	°C	12	mol%	50	ppmv	40	°C	0.2	molCO2/mol amine	0.45	molCO2/mol amine	3.226	mol amine/kg	

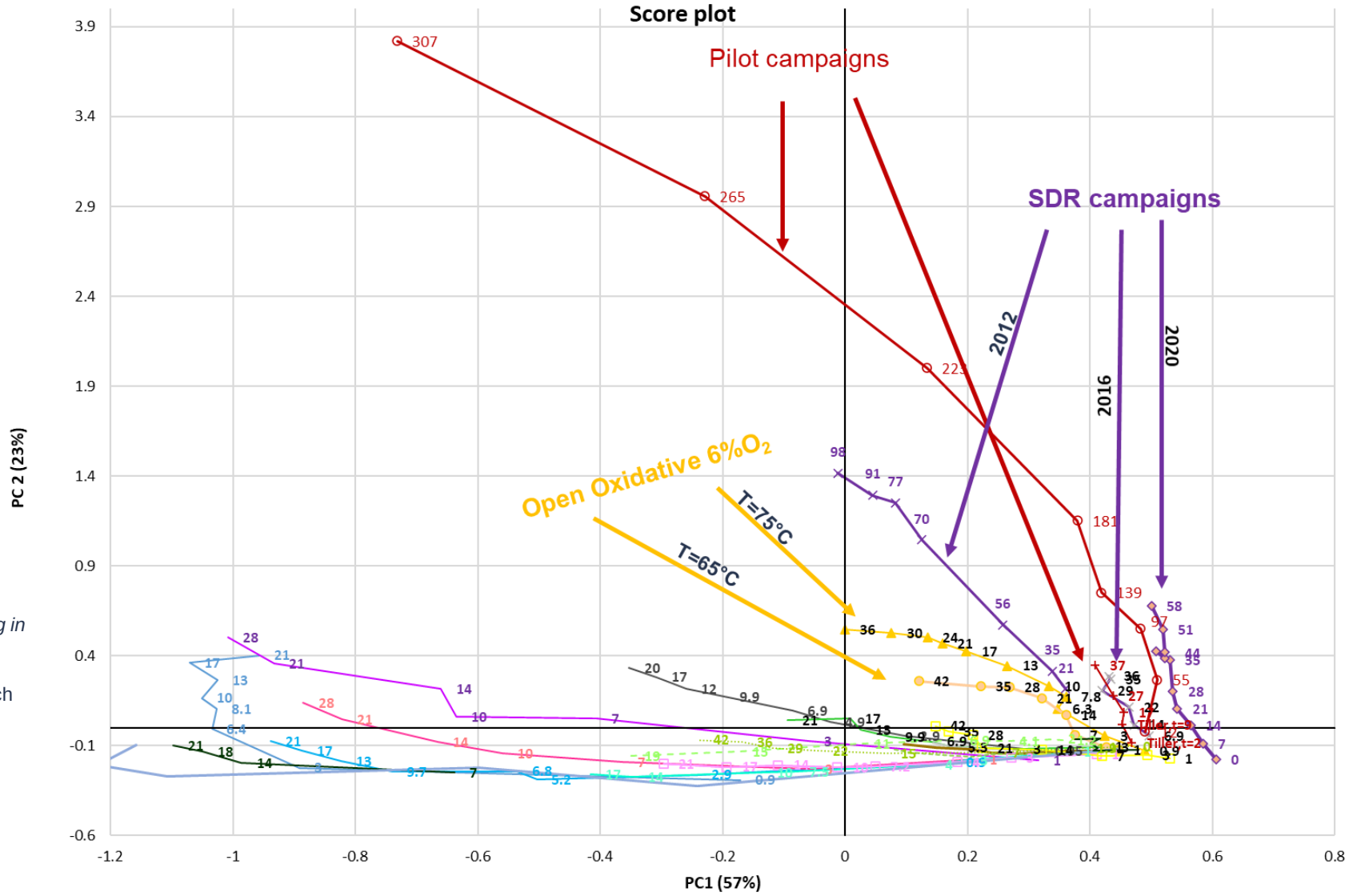
BHEOX	Unit	CAS	Molecular weight (g/mol)	Analytical instrument	HEA	Unit	CAS	Molecular weight (g/mol)	Analytical instrument	HeGly	Unit	CAS	Molecular weight (g/mol)	Analytical instrument
< 100	µg/mL	1871-89-2	176.17	LC-MS	< 10	µg/mL	142-26-7	103.12	LC-MS	< 10	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	20	µg/mL	142-26-7	103.12	LC-MS	460	µg/mL	5835-28-9	119.12	LC-MS
ot analyze	µg/mL	1871-89-2	176.17	LC-MS	ot analyze	µg/mL	142-26-7	103.12	LC-MS	ot analyzed	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	180	µg/mL	142-26-7	103.12	LC-MS	3211	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	237	µg/mL	142-26-7	103.12	LC-MS	4098	µg/mL	5835-28-9	119.12	LC-MS
ot analyze	µg/mL	1871-89-2	176.17	LC-MS	ot analyze	µg/mL	142-26-7	103.12	LC-MS	ot analyzed	µg/mL	5835-28-9	119.12	LC-MS
29	µg/mL	1871-89-2	176.17	LC-MS	443	µg/mL	142-26-7	103.12	LC-MS	6152	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	898	µg/mL	142-26-7	103.12	LC-MS	7117	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	1146	µg/mL	142-26-7	103.12		7494	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	1166	µg/mL	142-26-7			75	µg/mL	5835-28-9	119.12	LC-MS
< 100	µg/mL	1871-89-2	176.17	LC-MS	1386	µg/mL	142-2				µg/mL	5835-28-9	119.12	LC-MS

HeGly	Unit	CAS	Molecular weight (g/mol)	Analytical instrument
< 10	µg/mL	5835-28-9	119.12	LC-MS
460	µg/mL	5835-28-9	119.12	LC-MS
hot analyzed	µg/mL	5835-28-9	119.12	LC-MS
3211	µg/mL	5835-28-9	119.12	LC-MS
4098	µg/mL	5835-28-9	119.12	LC-MS
hot analyzed	µg/mL	5835-28-9	119.12	LC-MS
6152	µg/mL	5835-28-9	119.12	LC-MS
7117	µg/mL	5835-28-9	119.12	LC-MS
7494	µg/mL	5835-28-9	119.12	LC-MS
8475	µg/mL	5835-28-9	119.12	LC-MS
9288	µg/mL	5835-28-9	119.12	LC-MS

Example of use of data*

PCA analysis

This analysis based on data for the degradation compounds HeGly, HEF, BHEOX, HEA, HEPO, OZD and HEI



*) Taken from open webinar presentation *Solvent degradation testing in post combustion CO2 capture. The importance of fundamental knowledge and test strategies. A. Grimstvedt and S. J. Vevelstad,* Webinar, October 29th 2020 arrange by the Norwegian CCS research centre (NCCS).

Acknowledgements

The LAUNCH project is funded through the ACT programme (Accelerating CCS Technologies, Horizon2020 Project No 299662).

Financial contributions have been made by Ministry of Economic Affairs and Climate Policy, the Netherlands; The Federal Ministry for Economic Affairs and Energy, Germany; Gassnova of Norway through the CLIMIT program; and the Department for Business, Energy & Industrial Strategy, UK, with extra funding from the US Department of Energy. All funders are gratefully acknowledged.

Open event – Degradation Network Model

Juliana Monteiro, TNO

Delft, The Netherlands, 15th February 2023

Previous models (Literature)

Leónard et al.:

$$MEA + 1.3O_2 \rightarrow 0.6NH_3 + 0.1\,C_5H_8N_2O(HEI) + 0.1\,C_6H_{12}N_2O_2(HEPO) + 0.1CH_2O_2 + 0.8CO_2 + 1.5H_2O$$

$$-r_{MEA} = 5.35 \times 10^5 e^{-41.730/RT}[O_2]^{1.46}$$

Semi flow batch reactor → gas flow through the reactor (mimics absorber)
Temperature range: 55ºC (only 1 experiment), 100, 120 and 140ºC
Typical experiment lasted for 21 days

Degradation Network Model

Upsides:

Generic → can be applied to any solvent

Pragmatic → needs relatively simple and inexpensive lab data

Not dependent on knowledge on degradation products, or kinetics of individual reactions

Downsides:

Needs to be calibrated against pilot plant data

$$Amine + aO_2 \rightarrow DegP$$

$$\frac{d[O_2]}{dt} = -a\frac{d[amine]}{dt}$$

$$\frac{d[O_2]}{dt} = k[O_2]^n$$

$$k = k_0 e^{E_a/RT}$$

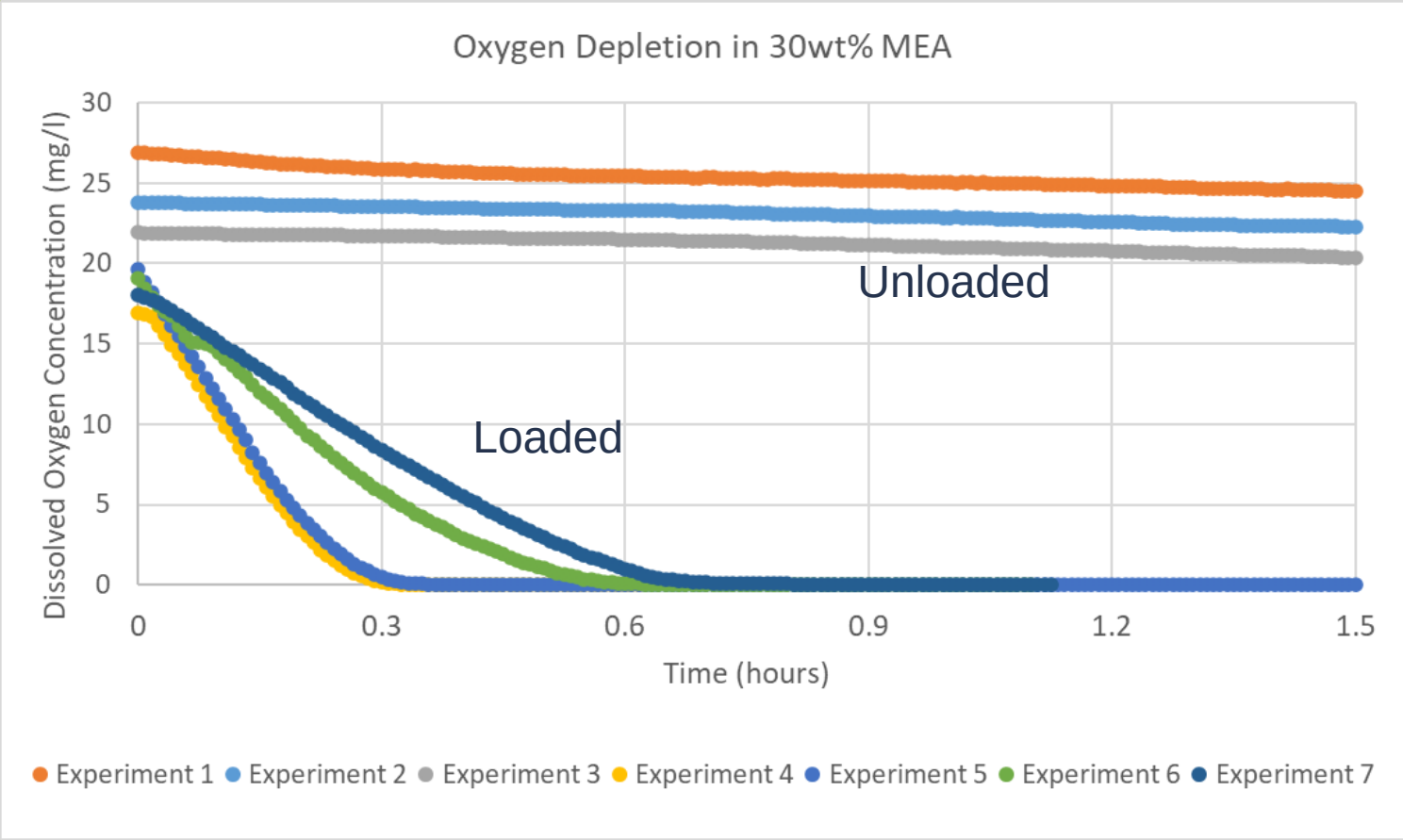
Experiments

Oxygen decay measured over time

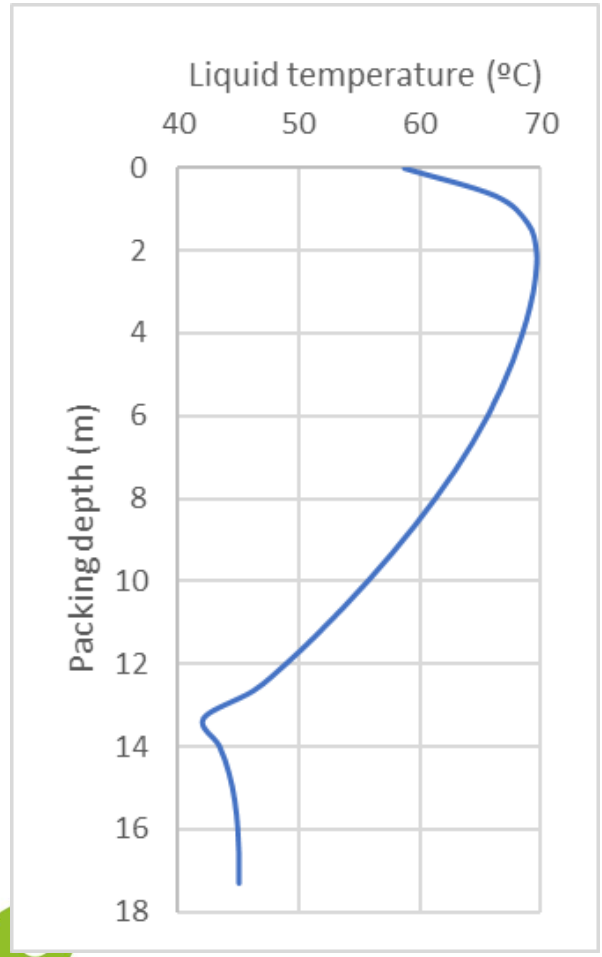
$$\frac{d[O_2]}{dt} = k[O_2]^n$$

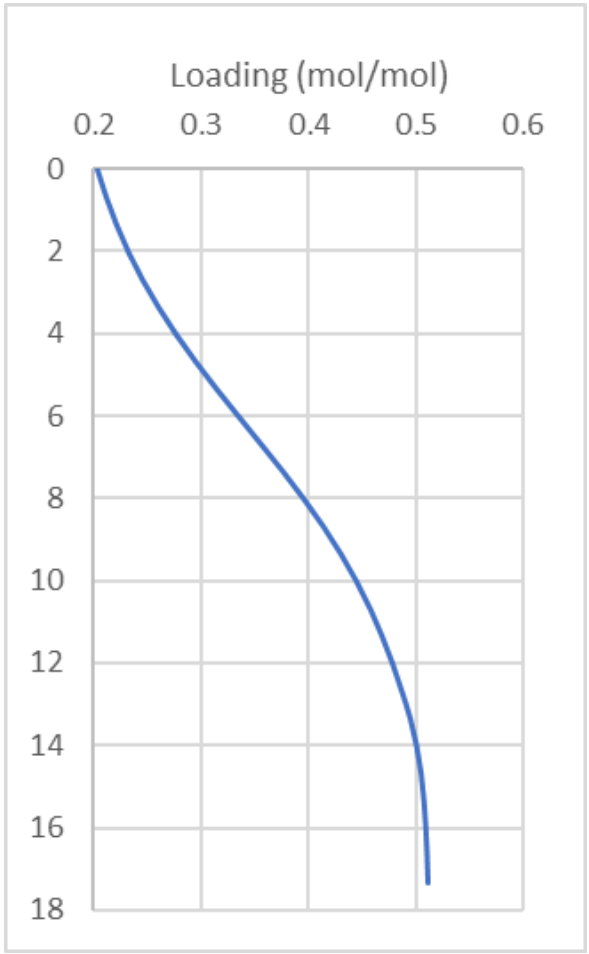
Typical experiment duration: from a couple of hours to 1 day

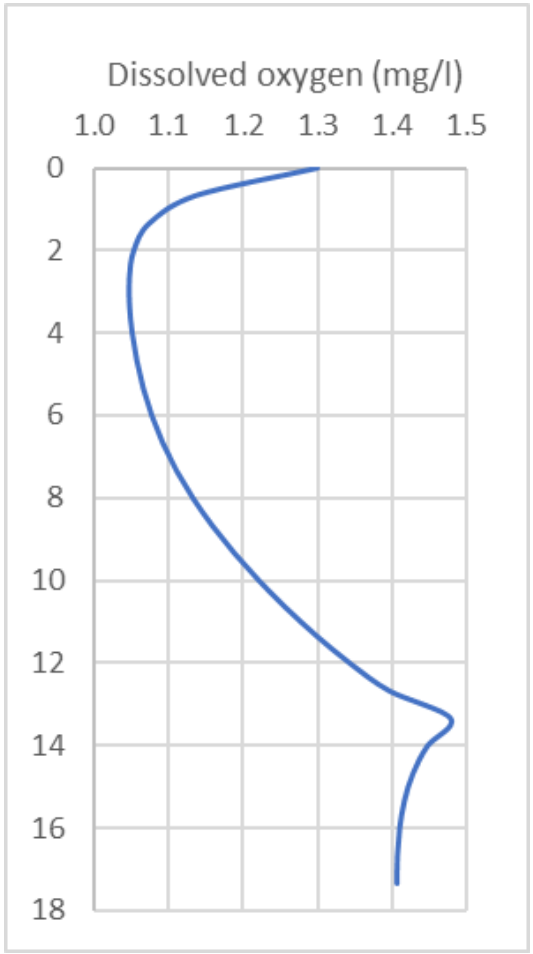
Typical results

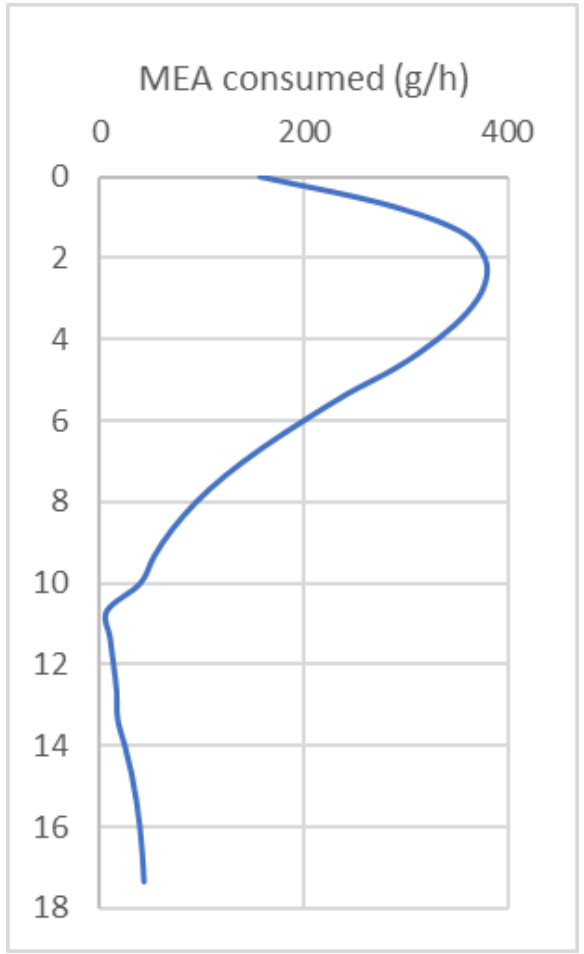


DNM application results – MEA









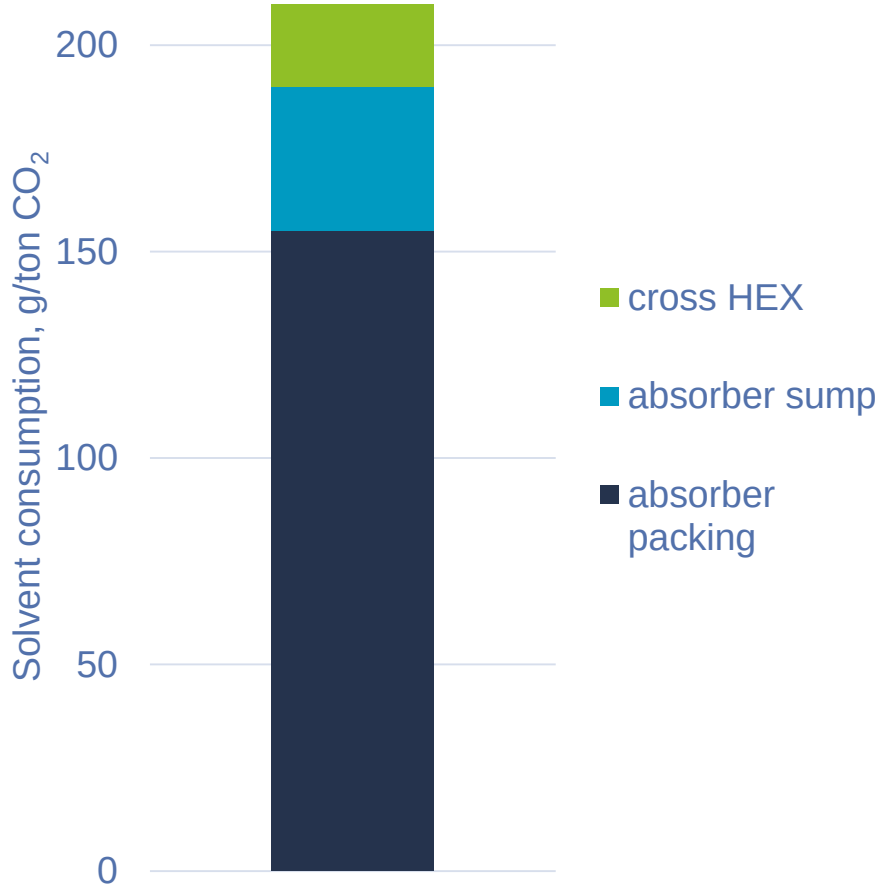
DNM application results – MEA

MEA: most degradation occurs in the absorber packing → control absorber temperature to control degradation

Next steps:
Model + report to be published in LAUNCH's website (Feb-Mar 2023)

Currently MEA and CESAR1 available

More data for both solvents being generated, updated model to be released later in 2023



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Open Event
15.02.2023

Accelerated Solvent Degradation

TNO: **Eirini Skylogianni**, Roberta Figueiredo, Arjen Huizinga, Paul Gravesteijn, Juliana Monteiro, Peter van Os

SINTEF: Andreas Grimstvedt, Solrun Johanne Vevelstad

UoS: **Muhammed Akram**, Jon Gibbins, Mohammed Pourkashanian

Supported by:
Federal Ministry for Economic Affairs and Climate Action
on the basis of a decision by the German Bundestag

Motivation of the work

- **Identify** accelerated degradation techniques to be used in lab scale rigs (LAUNCH rig) to…
- **Mimic** degradation trends of larger pilot plants

- **Currently,** the process of **solvent qualification requires thousands of operational hours of tests** in a pilot environment which is a complicated and expensive task.
- Having a **fast way of assessing a range of solvents and degradation countermeasures** at industrially-relevant timescales is needed and could help **accelerate CO_2 capture implementation**.

Accelerated degradation techniques

Increased oxygen content	→	O_2 decrease from 7 to 18 vol%
Increased amine concentration	→	MEA increase from 35 to 40 wt%
Increased stripping temperature	→	Reboiler temperature increase from 120 °C to 130 °C
Addition of elevated NOx levels	→	Addition of NOx

→ Combination of the above.

LAUNCH rig#2

- → Capture capacity: 25 kg CO_2/day
- → Mobile small-scale plant
- → Continuous operation in cyclic mode (absorption-desorption)
- → FT-IR on top of the absorber for online monitoring
- → Synthetic flue gas (N_2, O_2, CO_2, H_2O)
- → gas turbine representative gas composition (5.5 vol% CO_2)
- → cMEA; 35 wt% aqueous MEA
- → no water washes available during these campaigns

LAUNCH rig

TERC pilot plant

→ Capture capacity: 1000 kg CO_2/day

→ Continuous operation in cyclic mode (absorption-desorption), using 2 absorbers and 1 stripper

→ water wash after the 2nd absorber

→ synthetic flue gas (N_2, O_2, CO_2, H_2O)

→ gas turbine representative gas composition (5.5 vol% CO_2)

→ cMEA; 35 wt% aqueous MEA

TERC pilot plant

Accelerated degradation campaigns

Campaigns #	Description	Comment
Baseline	35 wt% MEA, **7.6 vol% O_2**, 120 °C	
High O_2	35 wt% MEA, **19.8 vol% O_2**, 120 °C	
High MEA wt%	**40 wt% MEA**, 19.8 vol% O_2, 120 °C	LAUNCH rig: ~37wt% MEA
High T	35 wt% MEA, 19.8 vol% O_2, **130 °C**	TERC: 128 °C
NOx	35 wt% MEA, 19.5 vol% O_2, 120 °C, **NOx**	LAUNCH rig: ~250, TERC: 15ppmv

→ Fresh solvent was used in each campaign.

→ Cleaning procedure consisted of washing the column with water & adding compressed air.

Sampling and Analysis

Sampling: Twice a week for the whole duration of the campaigns

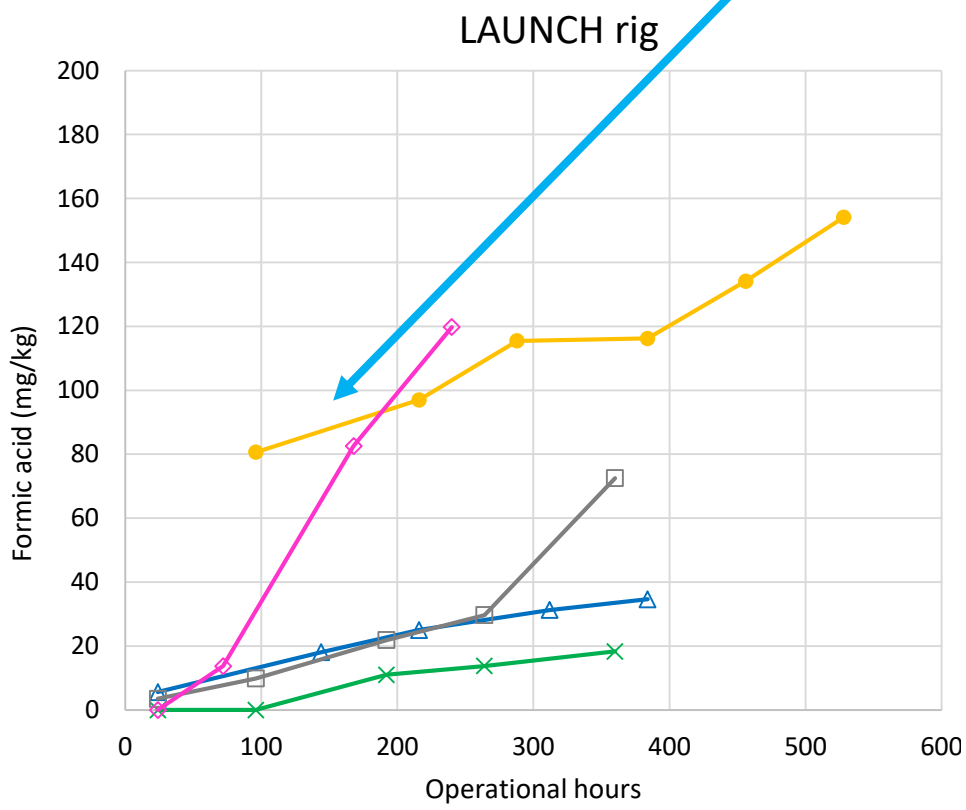
Components analyzed:

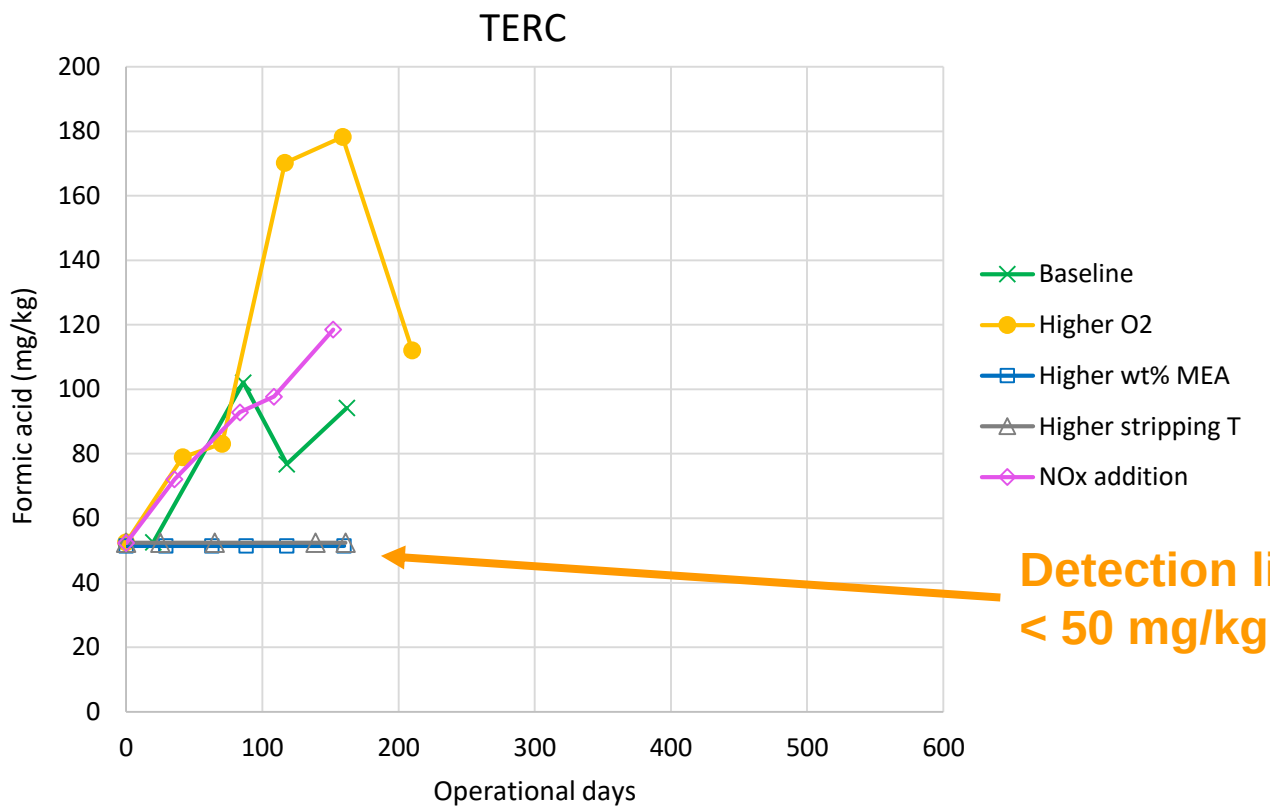
- MEA, water, CO_2
- Typical oxidative degradation products: acids (acetic, formic etc.), nitrate and nitrite
- Typical MEA degradation products: MEA-Urea, HEHEAA, HEEDA, HEI, HEF, HEPO, HeGly, HEA, BHEOX, HEIA, OZD and Nitroso-HeGly
- Metals: Fe, Cr, Ni, Mn, Cu, Zn, Mo, Ba, Pb

Results - Acids

High Fe concentration in the start of "Higher O2" campaign that seems to have affected the degradation trends

- **Formic acid is the most dominant acid** (acetic acid next in TERC, but undetected in LAUNCH rig)
- The *"Baseline"* campaign has the lowest concentration in the LAUNCH rig.
- Formic acid under detection limit for *"Higher wt% MEA"* and *"Higher stripping T"* campaigns.

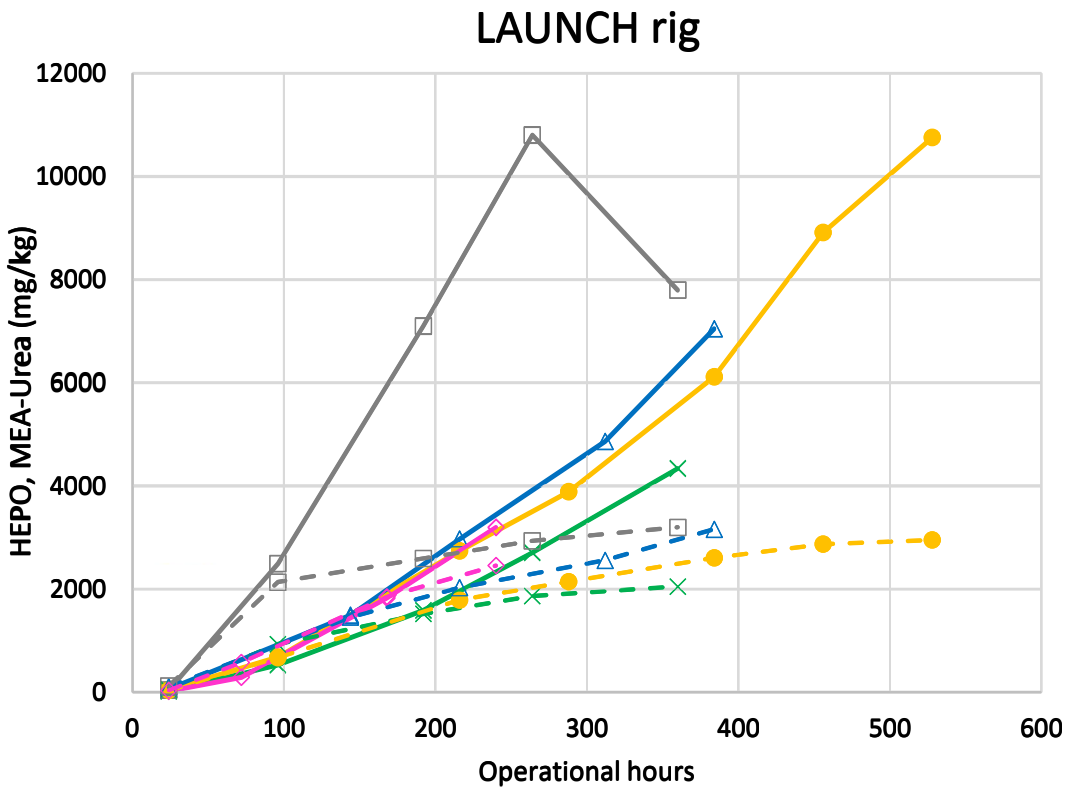


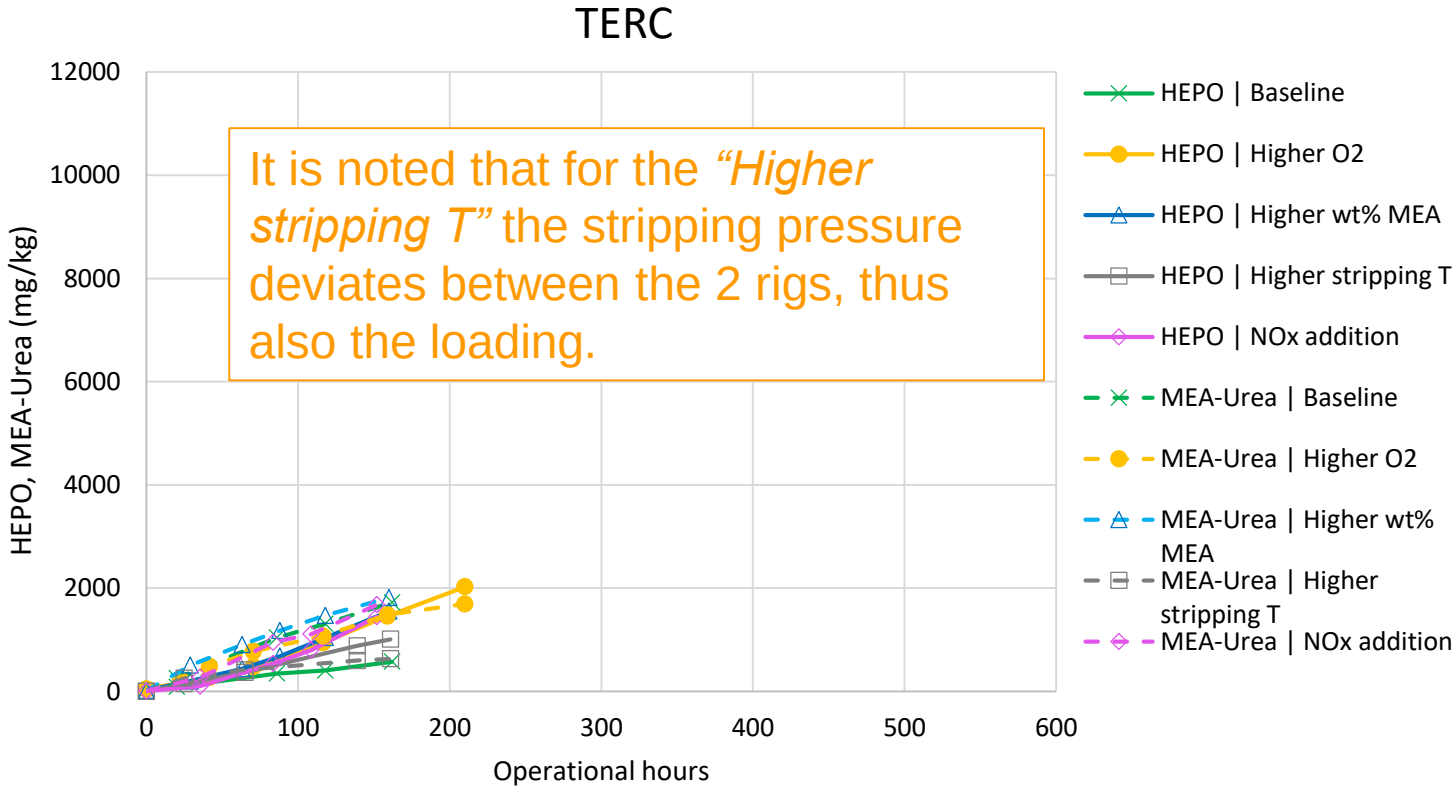


Detection limit < 50 mg/kg

Results – Major degradation components

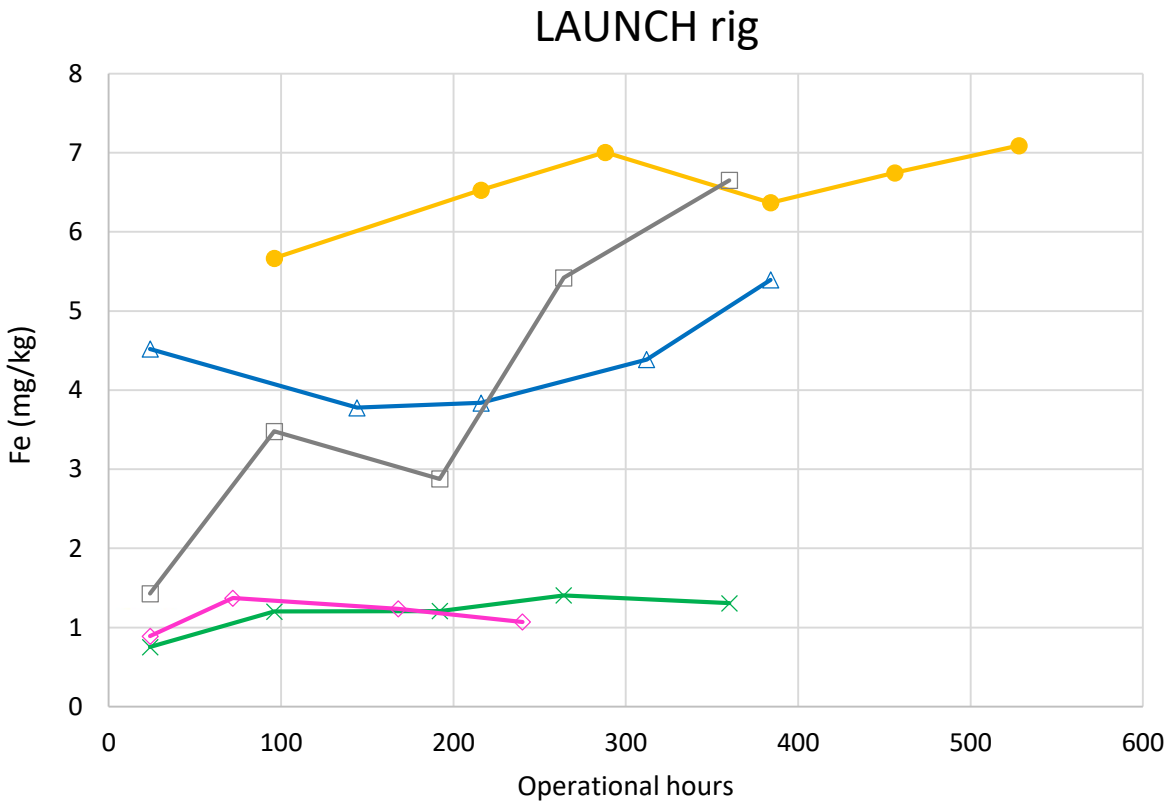
- **Major degradation products are HEPO, MEA-Urea and HEGly in both LAUNCH rig and TERC** (in agreement with *Vevelstad et al., 2017)*
- HEPO and MEA-UREA: highest at *"Higher stripping T"* in LAUNCH rig and at *"Higher wt% MEA in TERC"*.
- The LAUNCH rig shows similar concentrations as TERC.

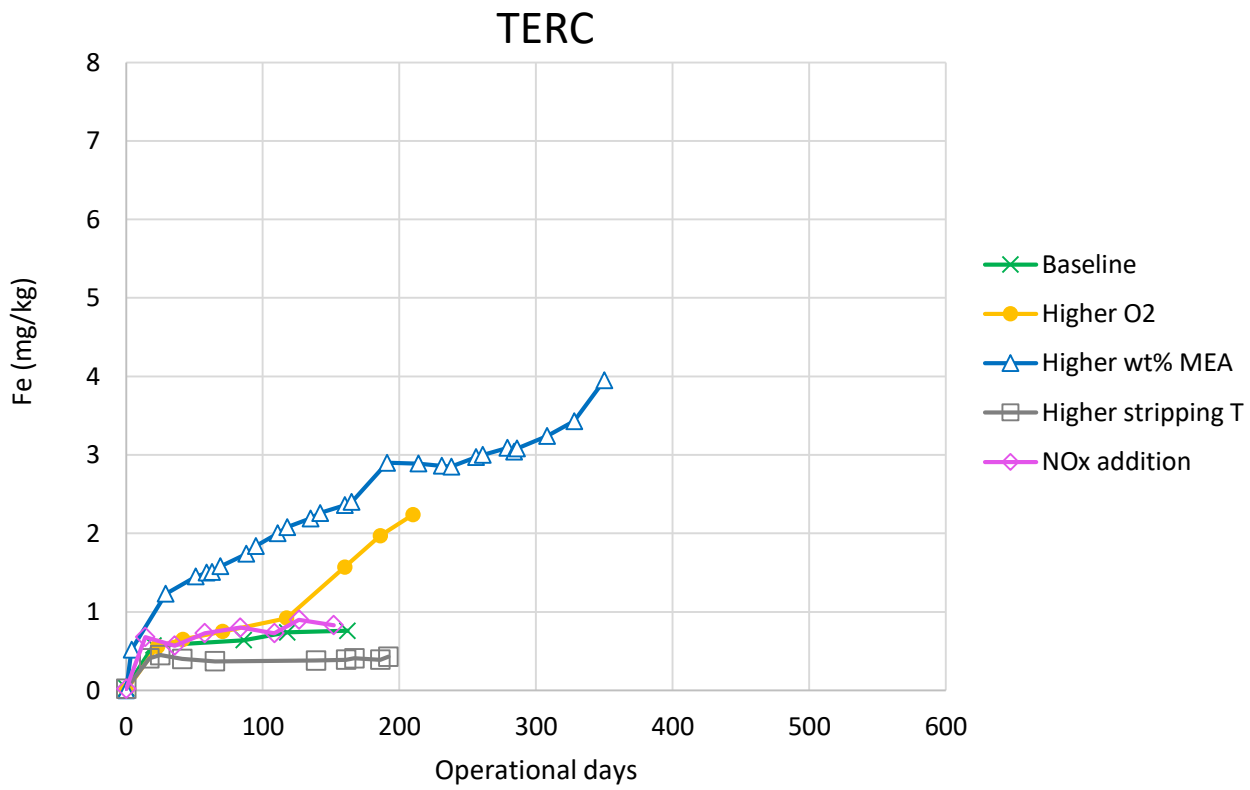




Results - Metals

- Presence of metals indicate corrosion issues.
- Fe is found at the highest concentration in LAUNCH rig, increasing sharper at *“Higher stripping T”*
- Fe is found highest at the *“Higher O2”* campaign and *“Higher wt% MEA”* in TERC.





Summary and Conclusions

→ Different accelerated degradation techniques were tested and the results were compared between TERC pilot plant (1000 kg CO_2/day) and the LAUNCH rig (25 kg CO_2/day).

→ Increasing the O_2 content in the gas is an efficient accelerated degradation technique. Additional acceleration is seen, in most cases, when combined with the other accelerated degradation techniques tested.

→ The major degradation products were HEPO, MEA-Urea and HEGly, while formic acid was the most dominant acid.

→ Highest HEPO, MEA-Urea and Fe concentration were found at the "Higher stripping T" in the LAUNCH rig and at the "Higher wt% MEA" at TERC.

→ Overall, the LAUNCH rig seems to follow the behaviour of the degradation products of TERC, though the iron values were higher.

The LAUNCH rig was capable of predicting the major degradation components found in the larger TERC pilot plant. Longer tests and more similar operating conditions can possibly explain the differences seen in the degradation trends.

Acknowledgements

The LAUNCH project (Project No. 299662) is co-funded by the ERA-NET Accelerating CCS Technologies initiative, which supports the delivery of safe and cost-effective carbon capture, utilisation and storage. The governments of each participating country have contributed funding through the ACT2 initiative.

Open event – The LAUNCH Rig

Juliana Monteiro, TNO

Delft, The Netherlands, 15th February 2023

Objective

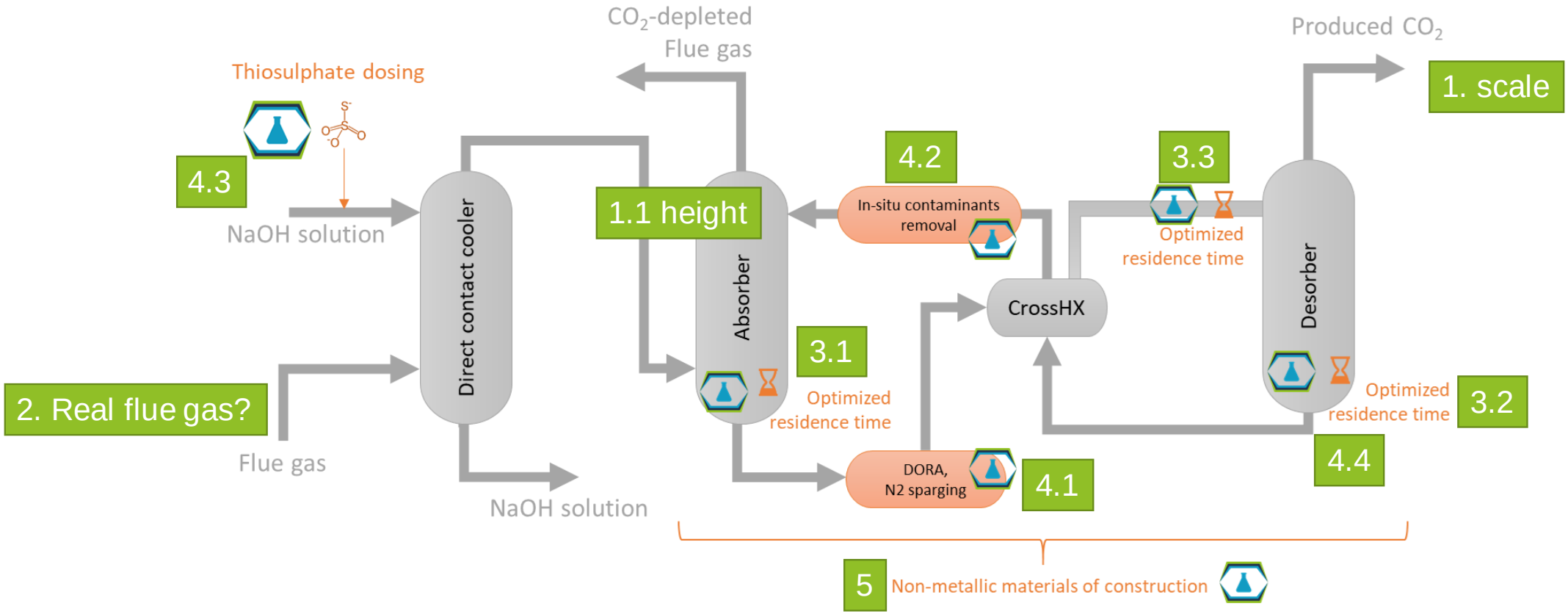
Design a test rig to be used to evaluate solvent degradation

Realistic results

Direct results

Minimal cost

The LAUNCH Rig – Design questions



1 - Scale

As small as possible to have realistic, direct results at the lowest possible cost

LAUNCH rig proposed size

3-5 Nm^3/h of gas

1 $kgCO_2/h$

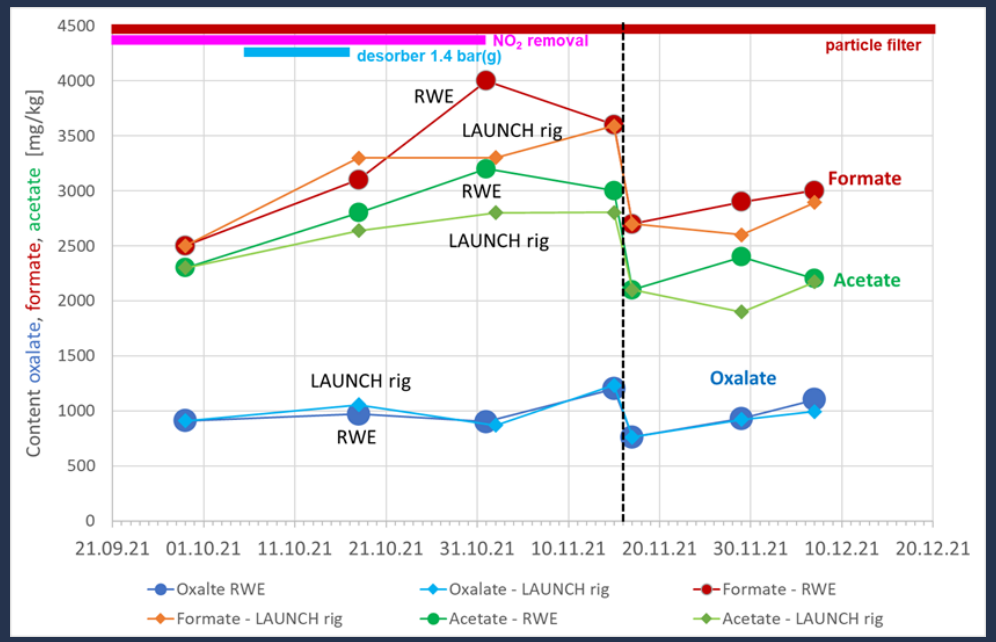
1.1 Absorber height:

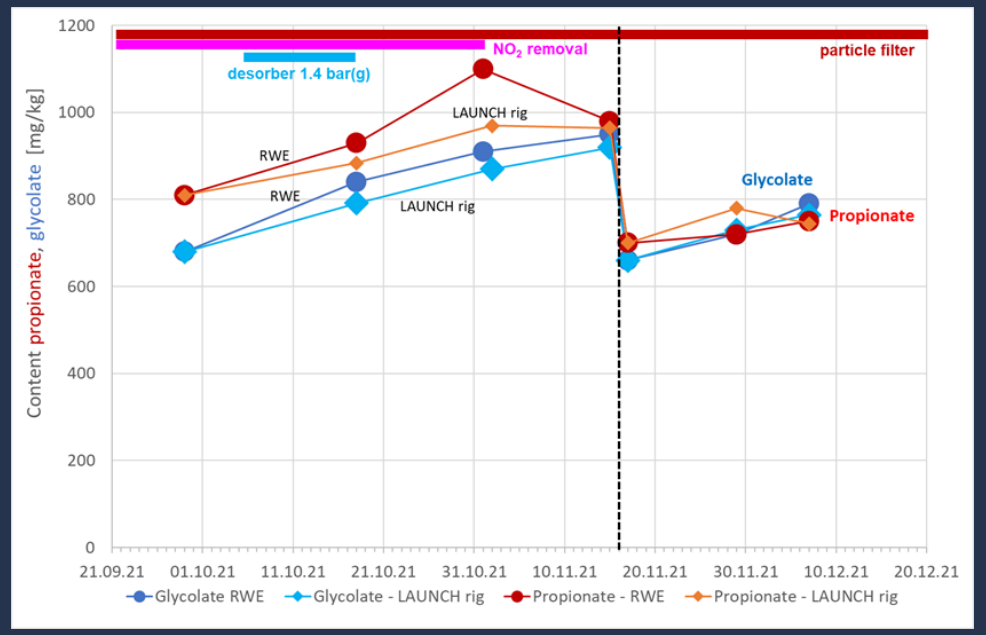
5-10 meters “lab” packing

depends on desired solvent kinetics

Important: absorber operates at the full scale lean to rich loading range → realistic degradation profile

1 – Scale: validation





RWE: 300 kg/h; LR: 1 kg/h → head-to-head operation for 3 months
Degradation products formate, acetate, glycolate, propionate and oxalate in both plants followed the same trend throughout the campaign

2 – Real Flue Gas

Flue gas minor components will influence degradation

Design specs: easily transportable, weather-proof, winterized

Important: treat flue gas as it would be treated in full scale: quench, remove SOx, remove particles, etc.

3 – Residence times

Realistic absorber and stripper sump residence times

Nice to have → sumps and hot pipping with flexible residence times to adapt to different plant configurations

4 – Degradation mitigation technologies

Include all that would be included in full scale (e.g., active carbon bed, oxygen removal)

In principle, no reclaiming

5 – Materials of construction

Same as planned for full scale (usually stainless steel) → realistic behaviour, de-risking

6 – Instrumentation

FTIR in the gas, O_2 analyser in the gas and liquid

Must have: offline solvent analysis for amines, CO_2, water and degradation compounds

Nice to have: online solvent monitoring

Acknowledgements

The LAUNCH project is funded through the ACT programme (Accelerating CCS Technologies, Horizon2020 Project No 299662).

Financial contributions have been made by Ministry of Economic Affairs and Climate Policy, the Netherlands; The Federal Ministry for Economic Affairs and Energy, Germany; Gassnova of Norway through the CLIMIT program; and the Department for Business, Energy & Industrial Strategy, UK, with extra funding from the US Department of Energy. All funders are gratefully acknowledged.

Open Event

Controlling degradation

Mitigation technologies used at RWE

Peter Moser,
Emission Reduction Technologies, R&D, RWE Power

Delft, 15th February 2023

Supported by:
Federal Ministry for Economic Affairs and Climate Action
on the basis of a decision by the German Bundestag

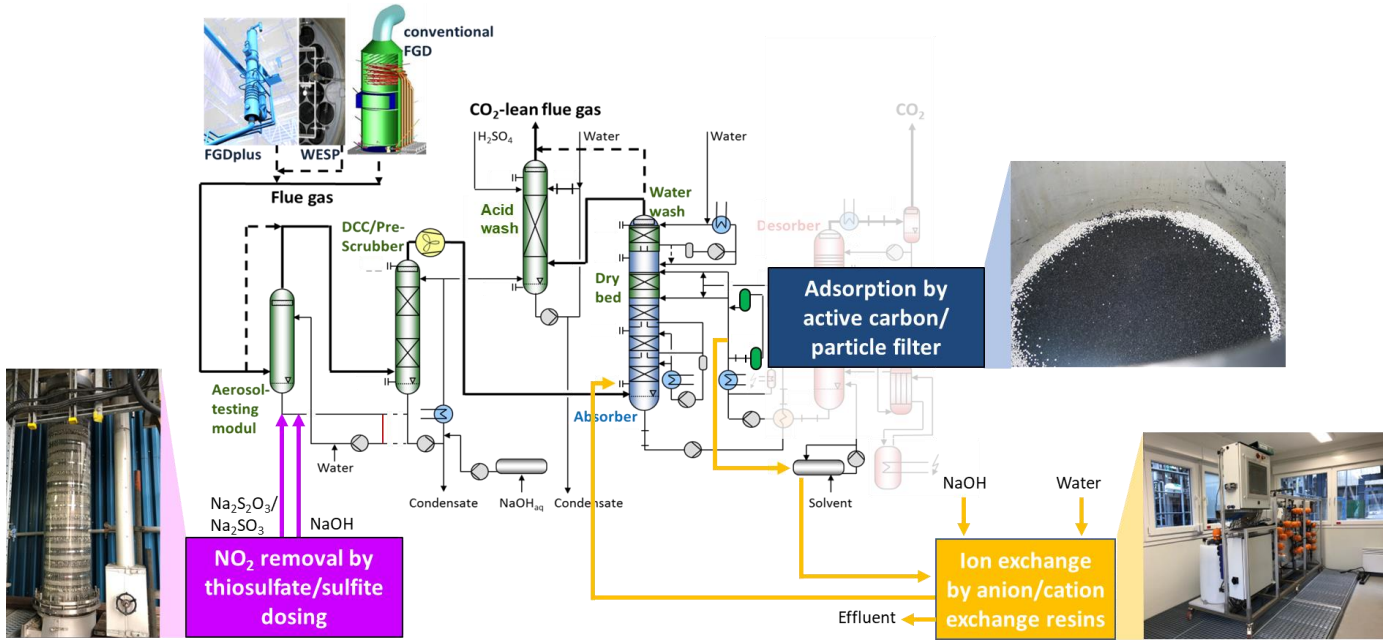
Mitigation technologies used at RWE

Capture pilot plant at RWE's Innovation Center at Niederaussem, Germany

Flue gas source: 1,000 MW lignite-fired power plant
Operation mode: 24/7, 7.2 t_{CO2}/d@capture rate 90%, 120°C/1.75 bar(a)
Solvent: CESAR1, aqueous blend of 3.0 M AMP and 1.5 M PZ
9 times batch-wise (10-25% of the inventory) addition of fresh solvent
Testing time: start on 09.04.2019, ongoing campaign, 34,000 testing hours

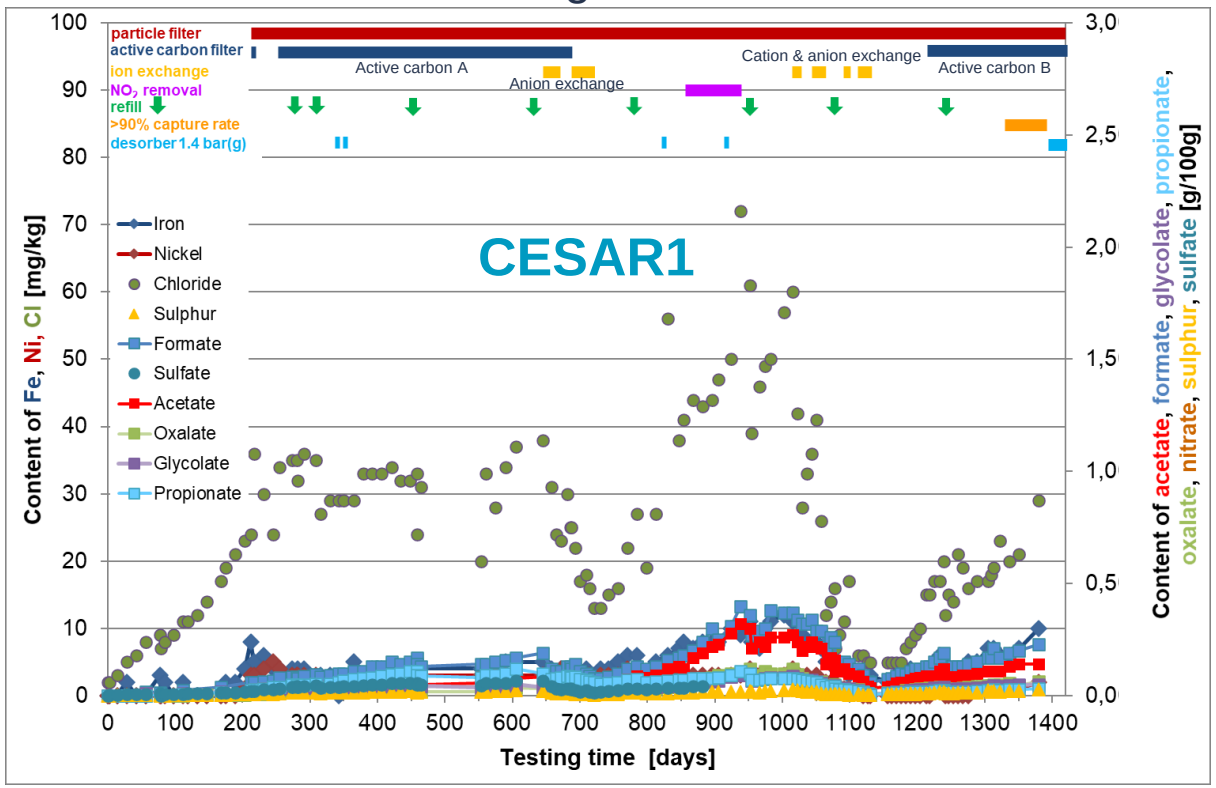
Evaluation of control technologies with different effect mechanisms:

- removal of NO_2 from flue gas by **thiosulfate dosing**
- removal of **ionic trace elements** from solvent by **ion-exchange**
- adsorptive removal of **trace elements** from solvent by **active carbon**

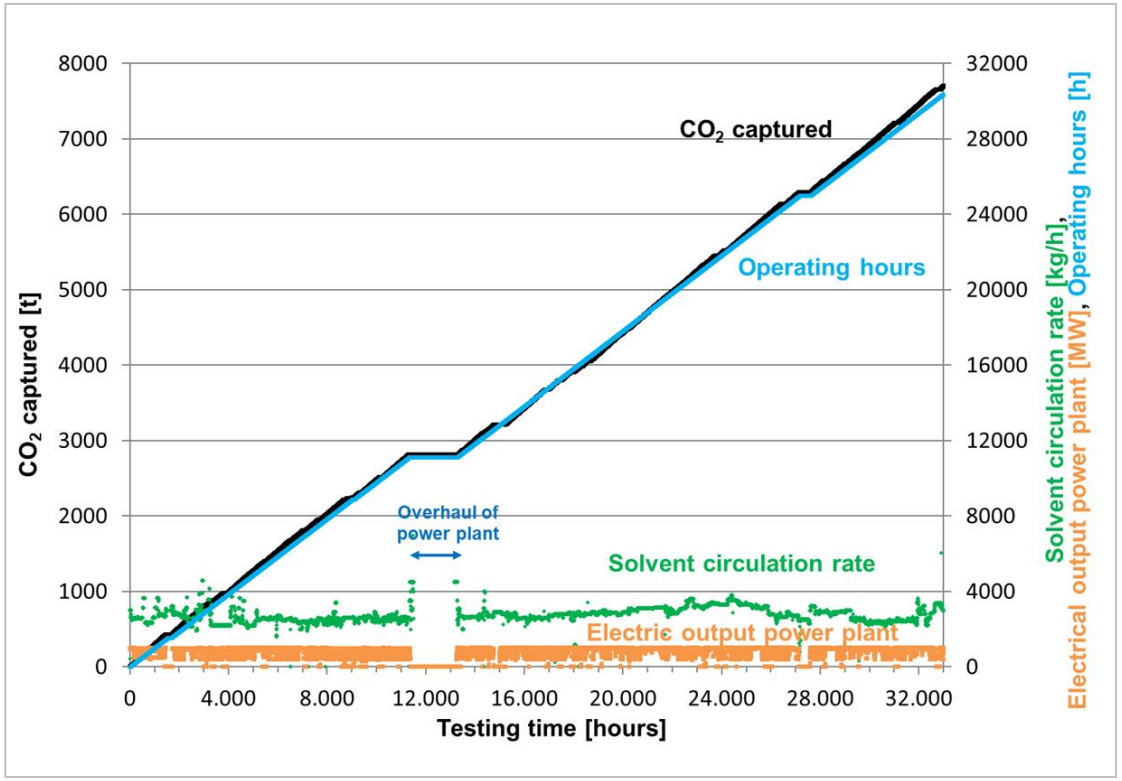


Start CESAR1 campaign on 9th April 2019, today testing day 1409
34,000 testing hours without exchange of the solvent inventory

CESAR1 - linear degradation behaviour

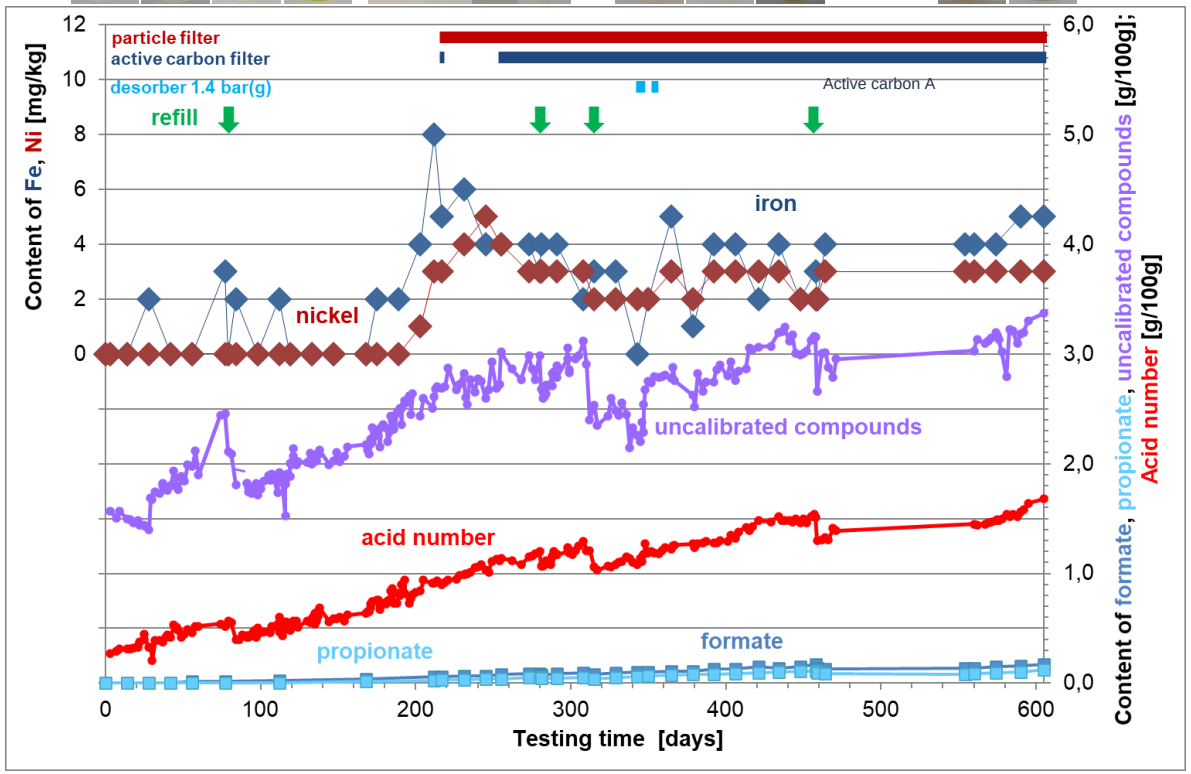


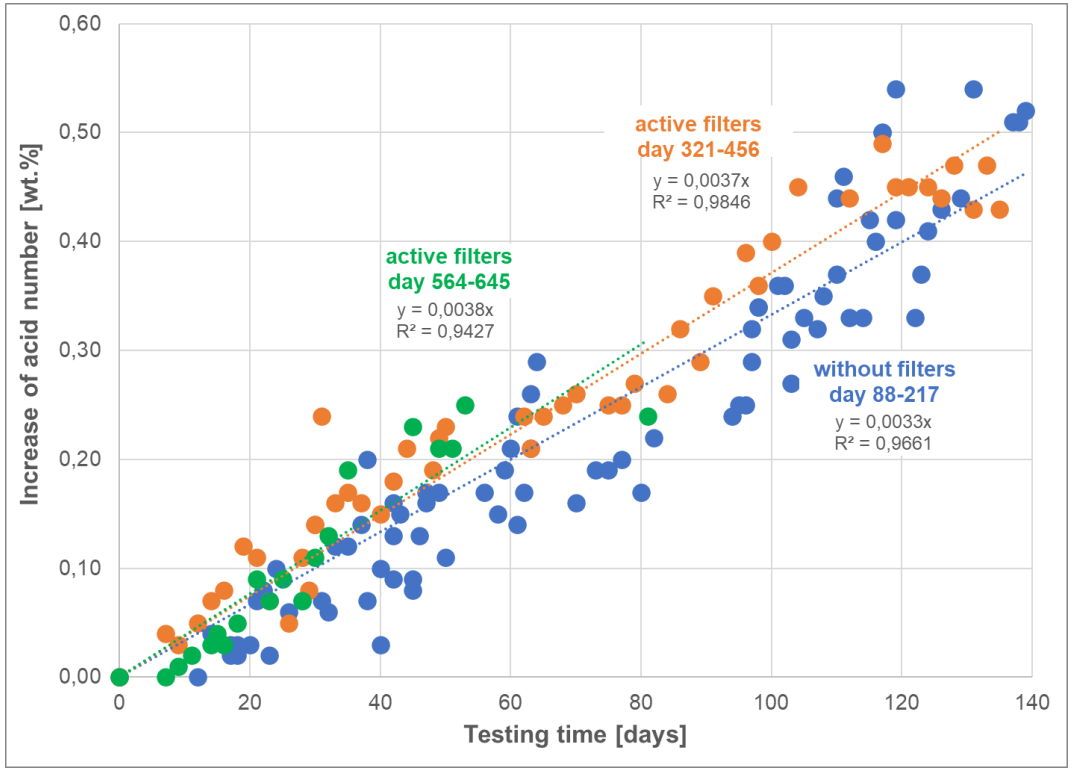
24/7 operation



The solvent degradation and the evaluation of solvent management technologies need sufficient testing time.

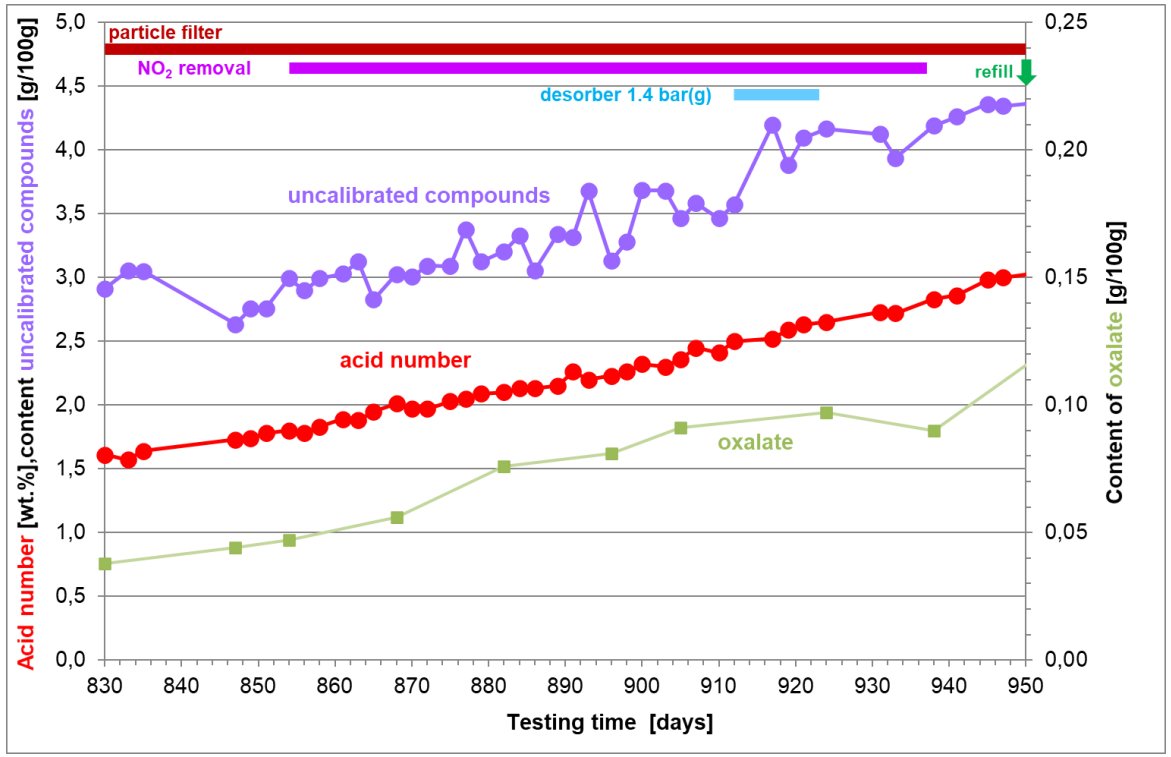
Adsorptive removal of trace elements by active carbon – no effect on the increase rate of acidic contaminants

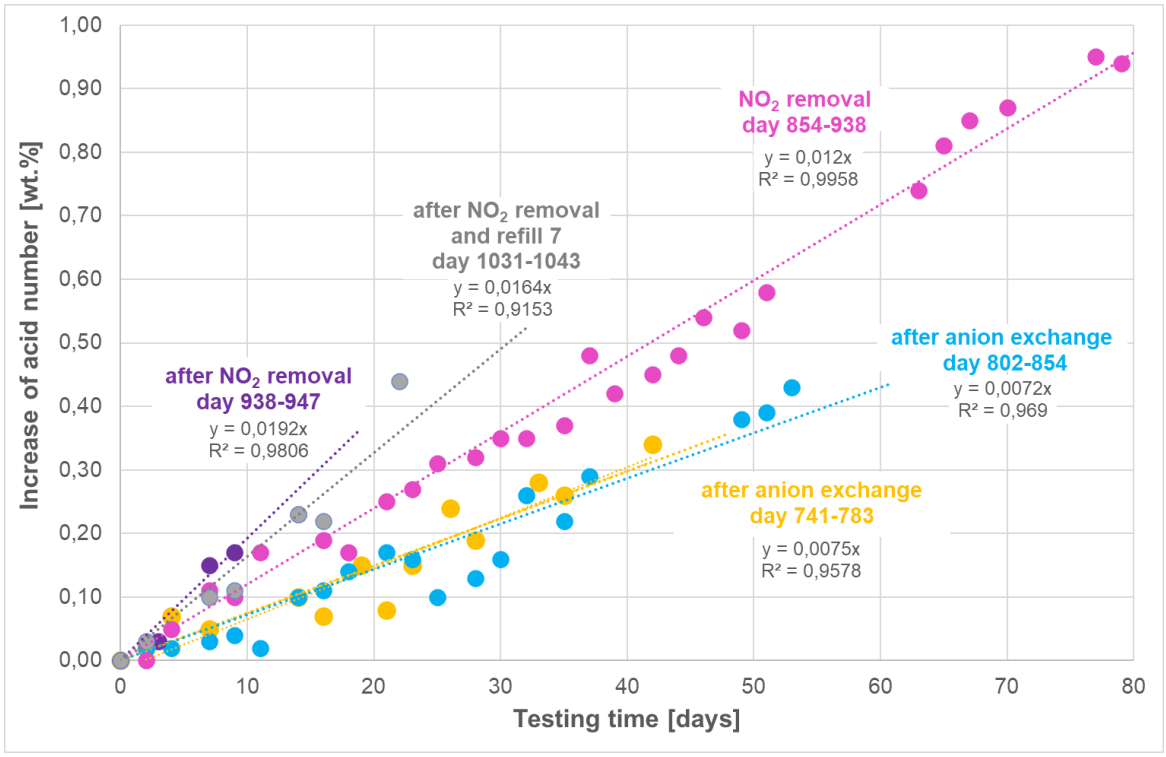




- Active carbon A can reduce the visible absorbance of the solvent and the iron and nickel concentration, but no clear effect on the solvent degradation rate was found.
- Active carbon B reduced visible absorbance and caused foaming.

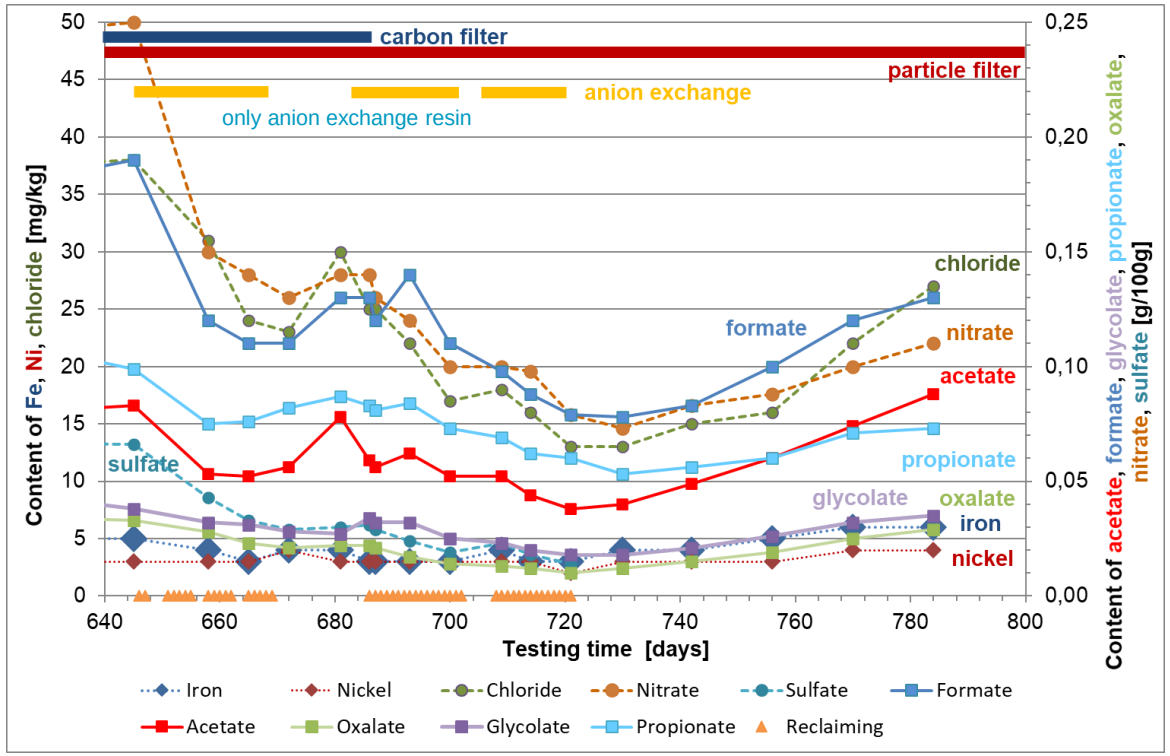
NO_2 removal from the feed gas by thiosulfate dosing

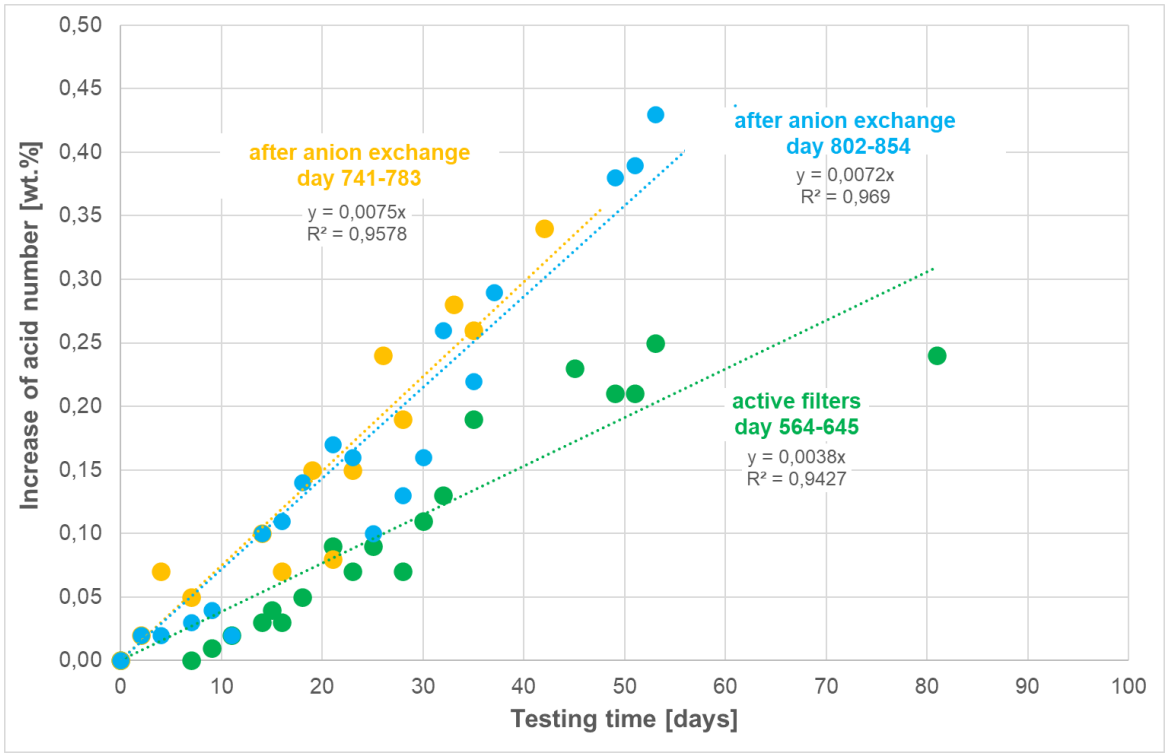




- **NO_2 removal by >80% in the flue gas at the entrance of the absorber had no decreasing effect on the increase rate of the acid number and of the formation of "uncalibrated compounds"**

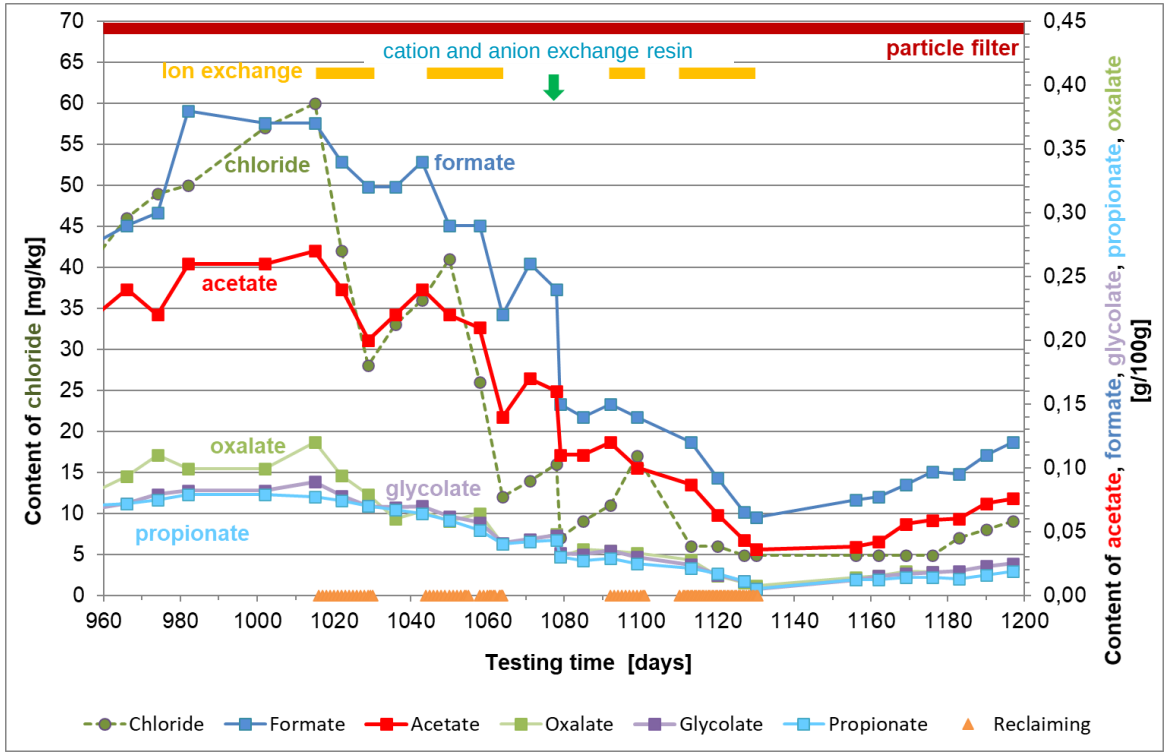
Ion exchange with anionic exchange resin – removal of anionic acid residues, trace components, and complexes

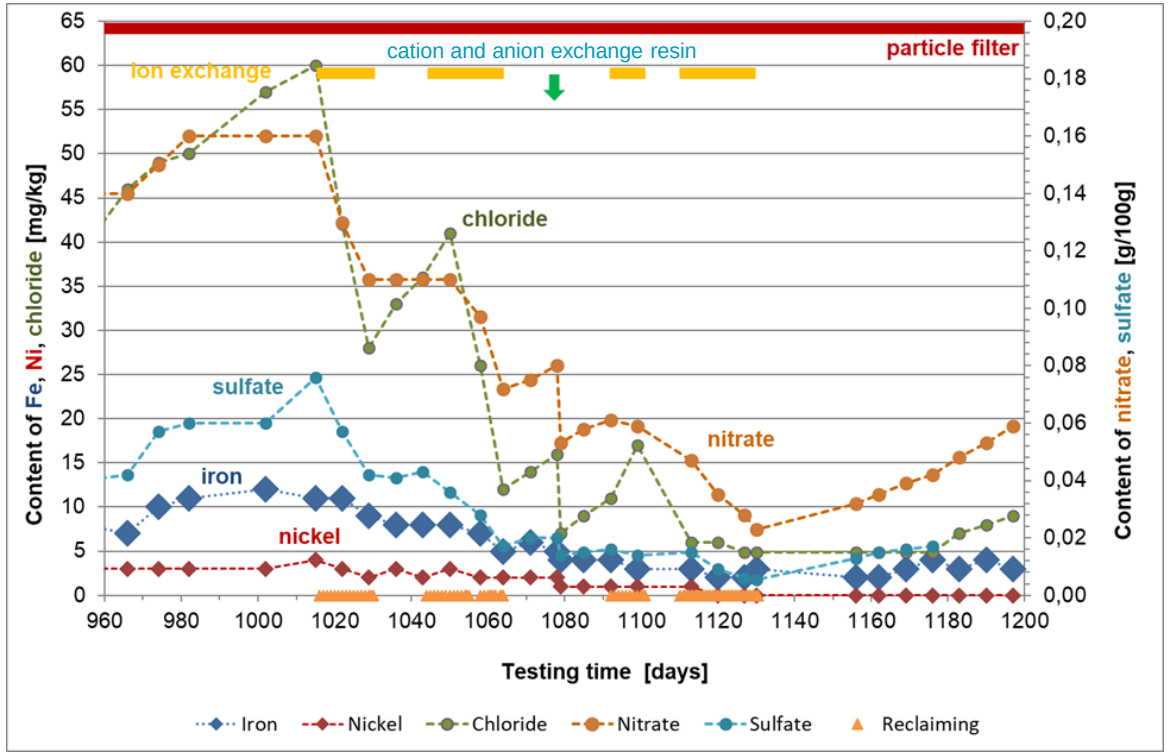




- Anion concentrations were reduced by more than 50% (inventory was treated in total two times), but after “cleaning” the solvent the rate of increase of the acid number doubled.
- Anionic ion exchange resins can remove especially iron (40%) from the solvent, but no nickel.
- Iron cations can exist as anionic complexes in the solvent depending on the solvent matrix.

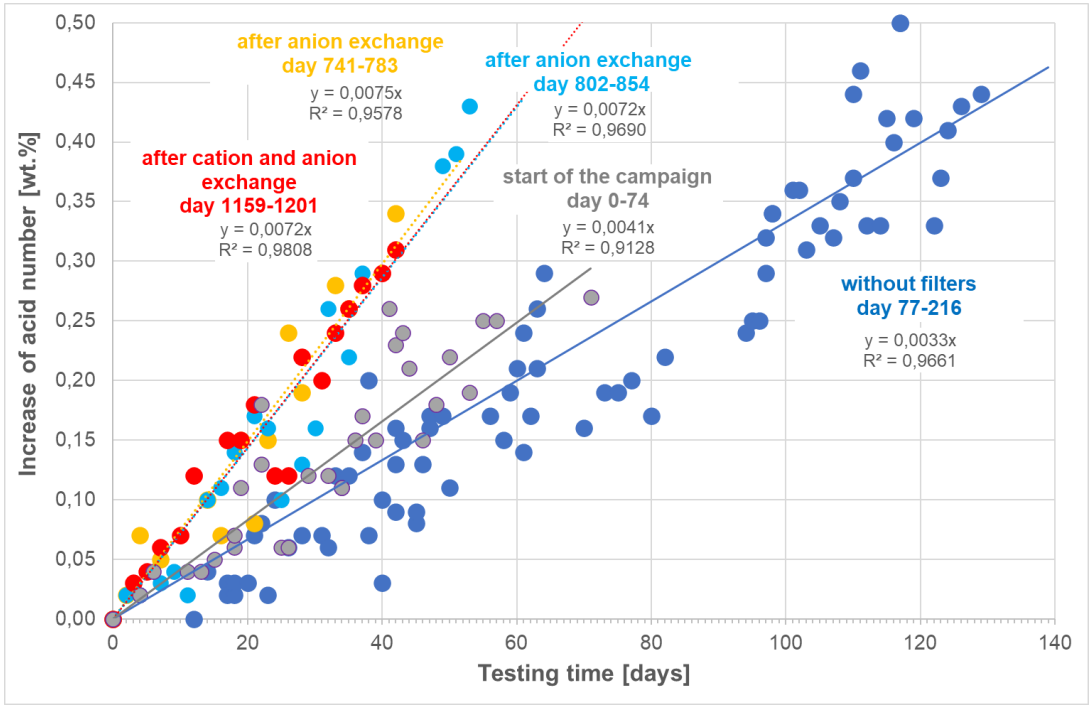
Removal of ionic trace elements from the solvent by anion and cation ion exchange





- **The ion concentrations could be reduced by 70% to 100%, to levels below the concentrations determined in the period <210 days of operation at the beginning of the overall campaign (before application of active carbon).**

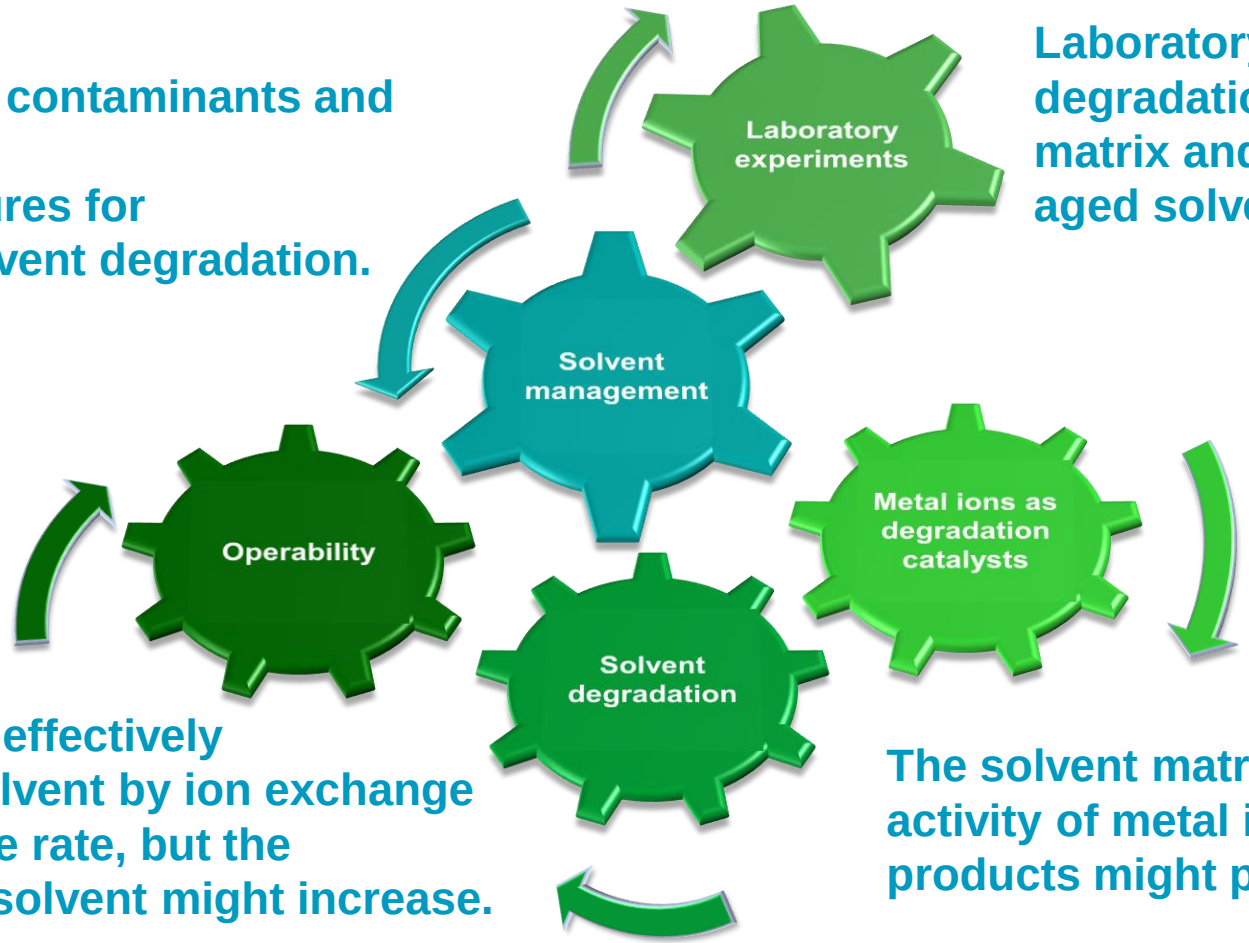
Removal of ionic trace elements from the solvent by anion and cation ion exchange



- After application of cation and anion ion exchange the increase rate of the acid number is similar to the rate after anion exchange.
- The lowest increase rate is not found for the fresh, most clean solvent, but for mildly aged solvent without any application of solvent management.
- As catalyst acting metals in the solvent are complexed and their accessibility for reaction partners and redox behavior are tuned by the ligands of the solvent matrix.

Conclusion from Niederaussem – 4 years of operation without exchange of the solvent inventory is possible and without excessive solvent “cleaning”

Adsorptive removal of contaminants and NO_2 removal are no effective measures for CESAR1 to reduce solvent degradation.



Laboratory experiments on solvent degradation should consider the solvent matrix and should comprise tests with aged solvent.

Results received from long-time testing with real flue gases are important to evaluate degradation models.

Ionic contaminants can effectively be removed from the solvent by ion exchange to retain the CO_2 capture rate, but the degradation rate of the solvent might increase.

The solvent matrix is important for the catalytic activity of metal ions: Some degradation products might protect the solvent.

Acknowledgements

The LAUNCH project is funded through the ACT2 programme (Accelerating CCS Technologies, Horizon2020 Project No 294766). Financial contributions have been made by Ministry of Economic Affairs and Climate Policy, the Netherlands; The Federal Ministry for Economic Affairs and Climate Action, Germany; Gassnova of Norway through the CLIMIT program; and the Department for Business, Energy & Industrial Strategy, UK, with extra funding from the US Department of Energy. All funders are gratefully acknowledged.

https://launchccus.eu/

Ministerie van Economische Zaken en Klimaat

Supported by:

Federal Ministry for Economic Affairs and Climate Action

on the basis of a decision by the German Bundestag

GASSNOVA

Department for Business, Energy & Industrial Strategy

U.S. DEPARTMENT OF ENERGY

Final event

Oxidative degradation

Roberta Figueiredo and Fred Closmann

Delft, The Netherlands, 15th February 2023

Oxygen in the flue gas

Metals in solution

High temperatures

Flue gas impurities

Particles in the flue gas

Solvent degradation

Emissions

Reduced solvent capacity

High costs

Irreversible reactions

Corrosion

Solvent handling

Main objective: How to solve the oxidative degradation issues?

DORA

Membrane contactor

Indirect contact with the solvent

Sweep gas + vacuum

After the sump

Use of N_2

Decrease in degradation products

Increase CO_2 quality

N_2 Sparging

In abs sump or separate vessel

Direct contact with the solvent

Stripping gas (N_2)

Final meeting

Oxygen removal Highlights - DORA

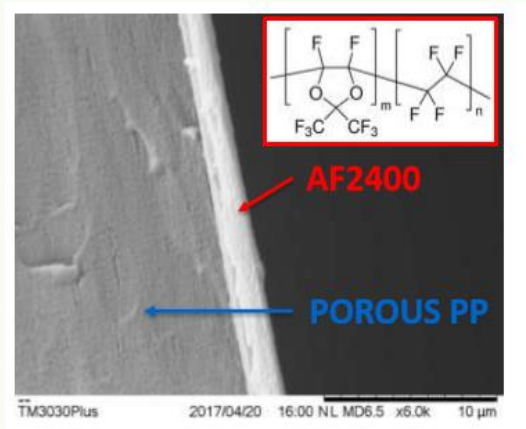
DORA - Membrane choice

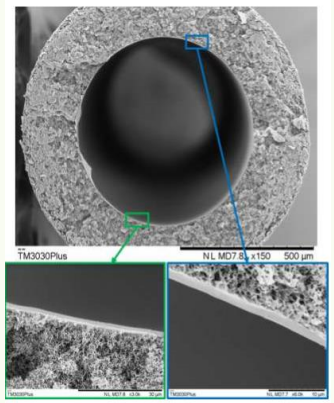
POROUS

Liqui-Cell X40 membrane SP Series (2.5x8 extra-flow model)

TRL6

DENSE LAYER



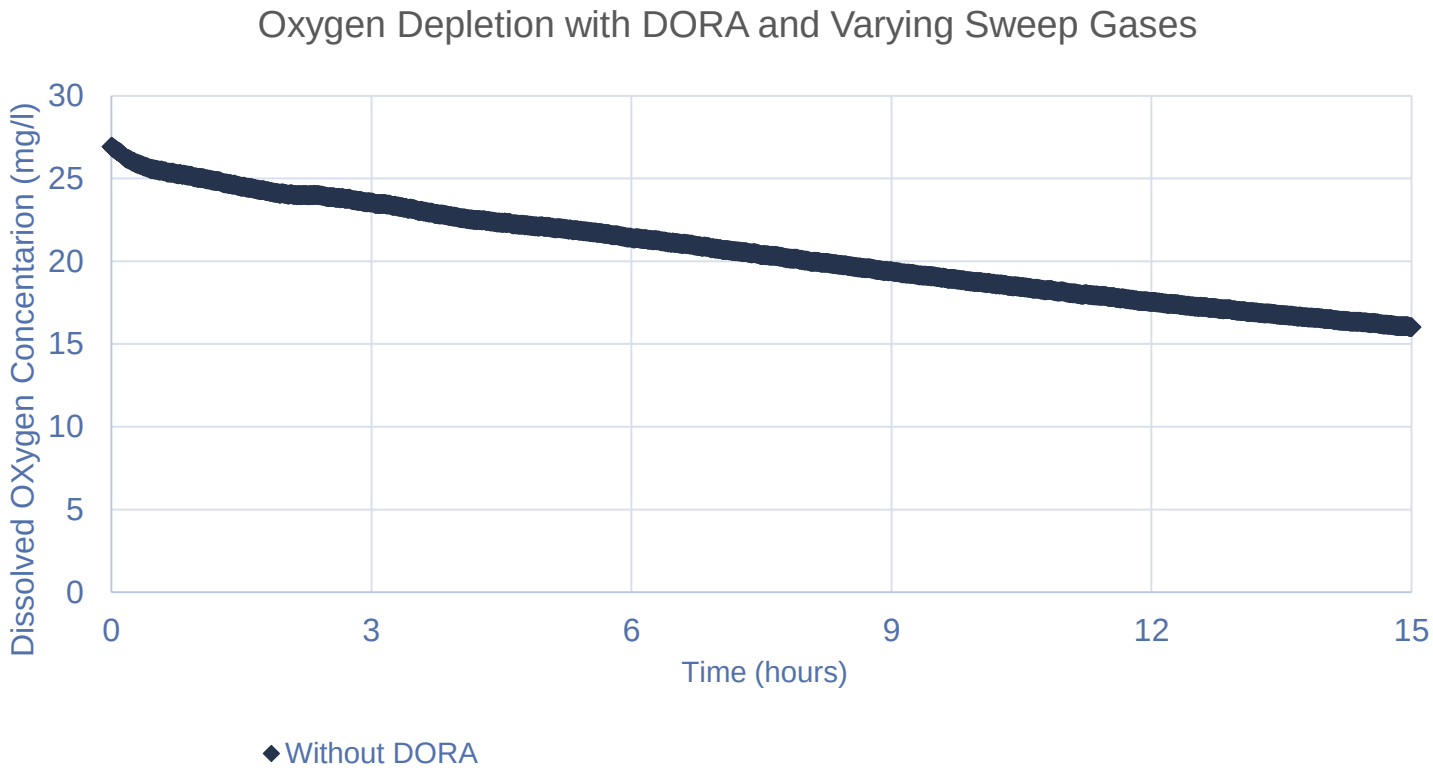


Cross sectional image of the hollow fibre membrane with AF2400 coating

TRL4

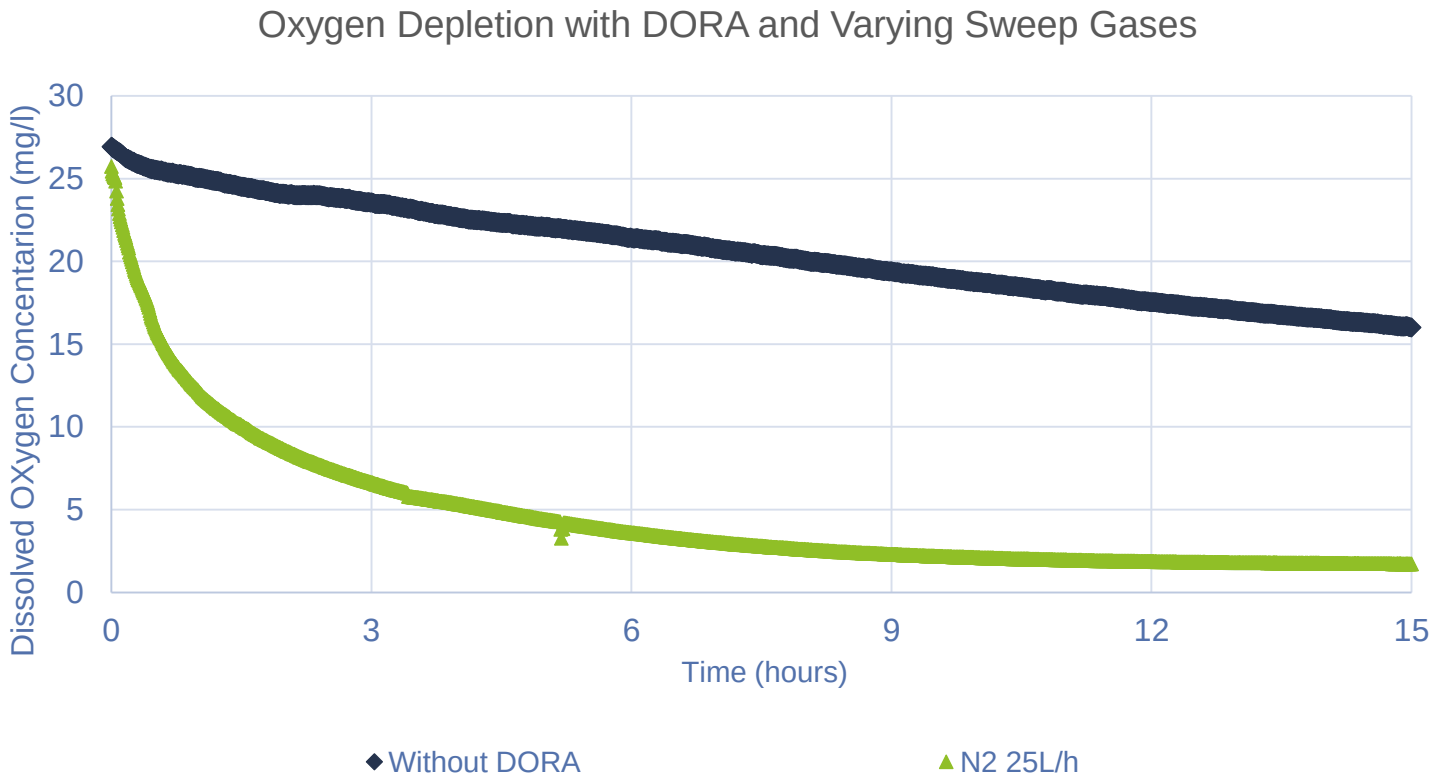
L. Ansaloni, A. Hartono, M. Awais, H. K. Knuutila, and L. Deng, “CO2 capture using highly viscous amine blends in non-porous membrane contactors,” *Chem. Eng. J.*, vol. 359, no. 2019, pp. 1581–1591, 2019

Impact of vacuum on N_2 use



Tests done with MEA and pure oxygen in the Oxygen Depletion Installation with and without the DORA

Impact of vacuum on N_2 use



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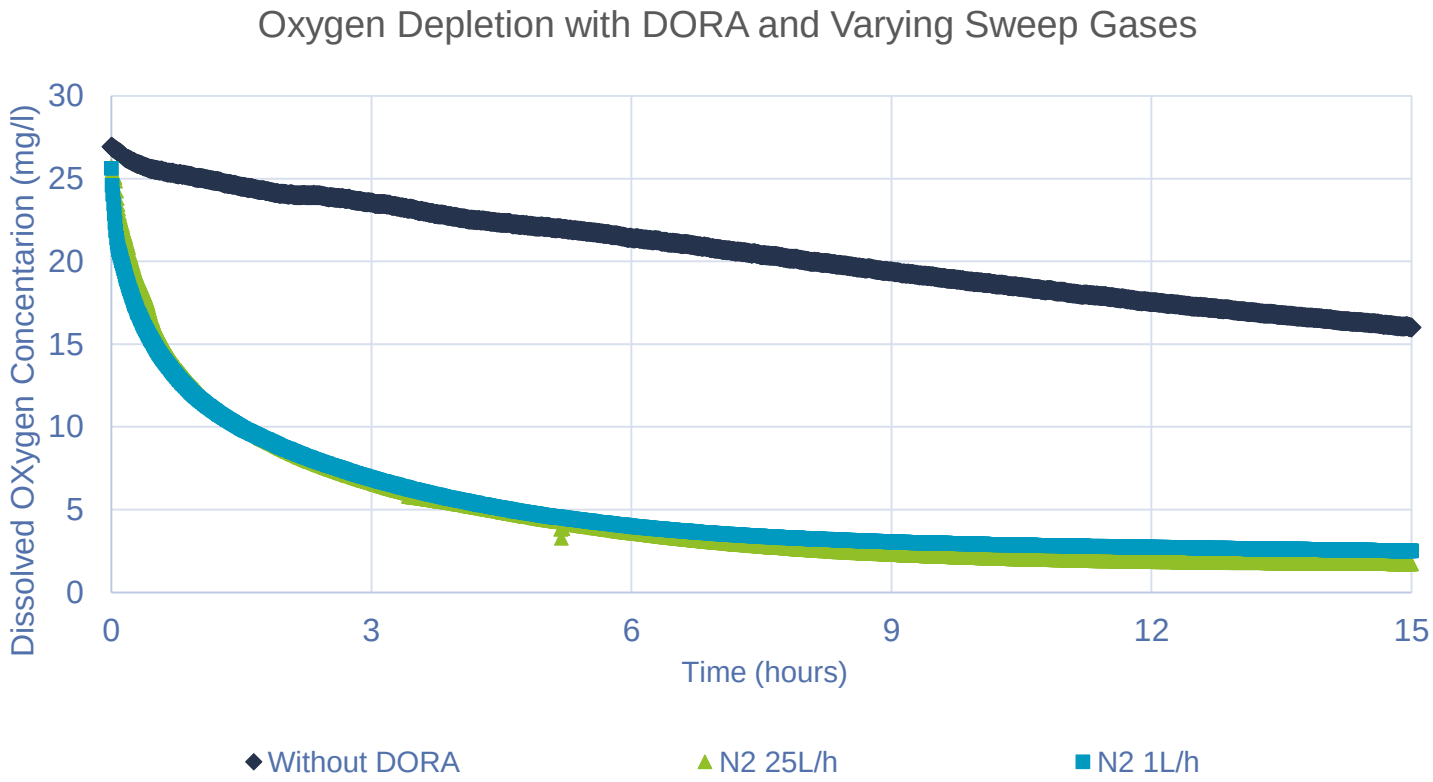
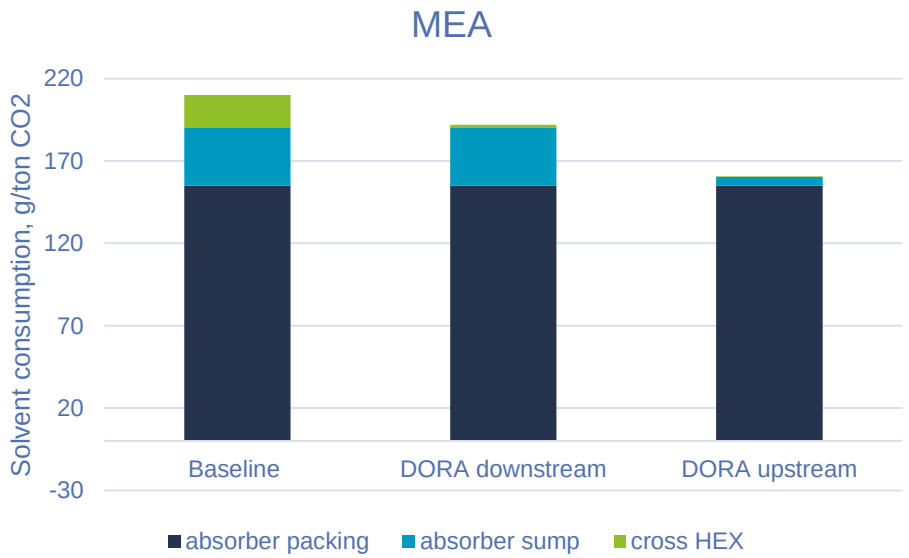
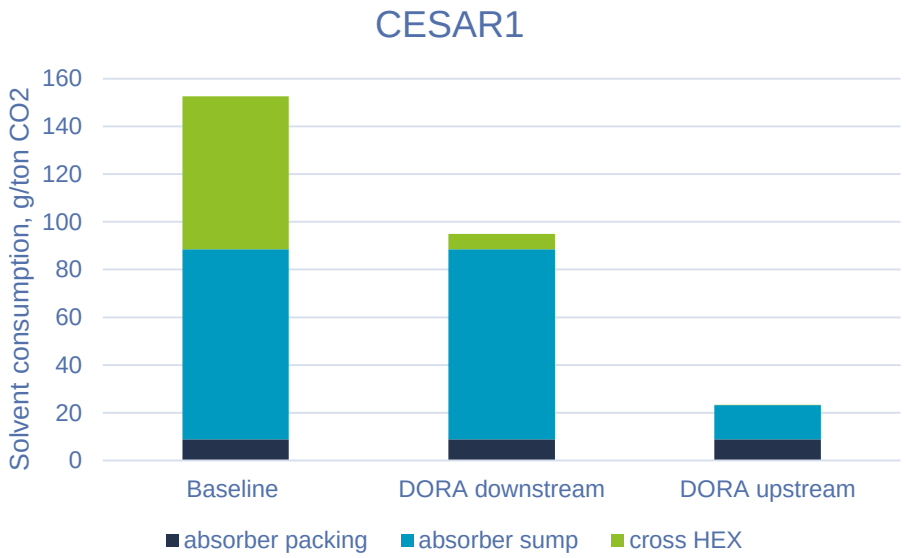


Tests done with MEA and pure oxygen in the Oxygen Depletion Installation with and without the DORA

DORA results from the Degradation Network Model





From the preliminary results, the impact of DORA is even more significative for solvents that are more stable such as CESAR1

Oxygen removal Highlights – N2 sparging

Nitrogen sparging

- N_2 sparging proposed for stripping dissolved oxygen from solvent before it is transferred to the hot side of the absorption/stripping process.

Laboratory tests demonstrated:

- N_2 sparging achieved **> 90% DO removal**
- N_2 sparging achieved **> 50% reduction in degradation rate** (formate, ammonia)
- Absorber sump sparging at SRP demonstrated **~ 90% reduction in DO**
- Absorber sump sparging is currently being tested at NCCC

Pilot testing at the SRP

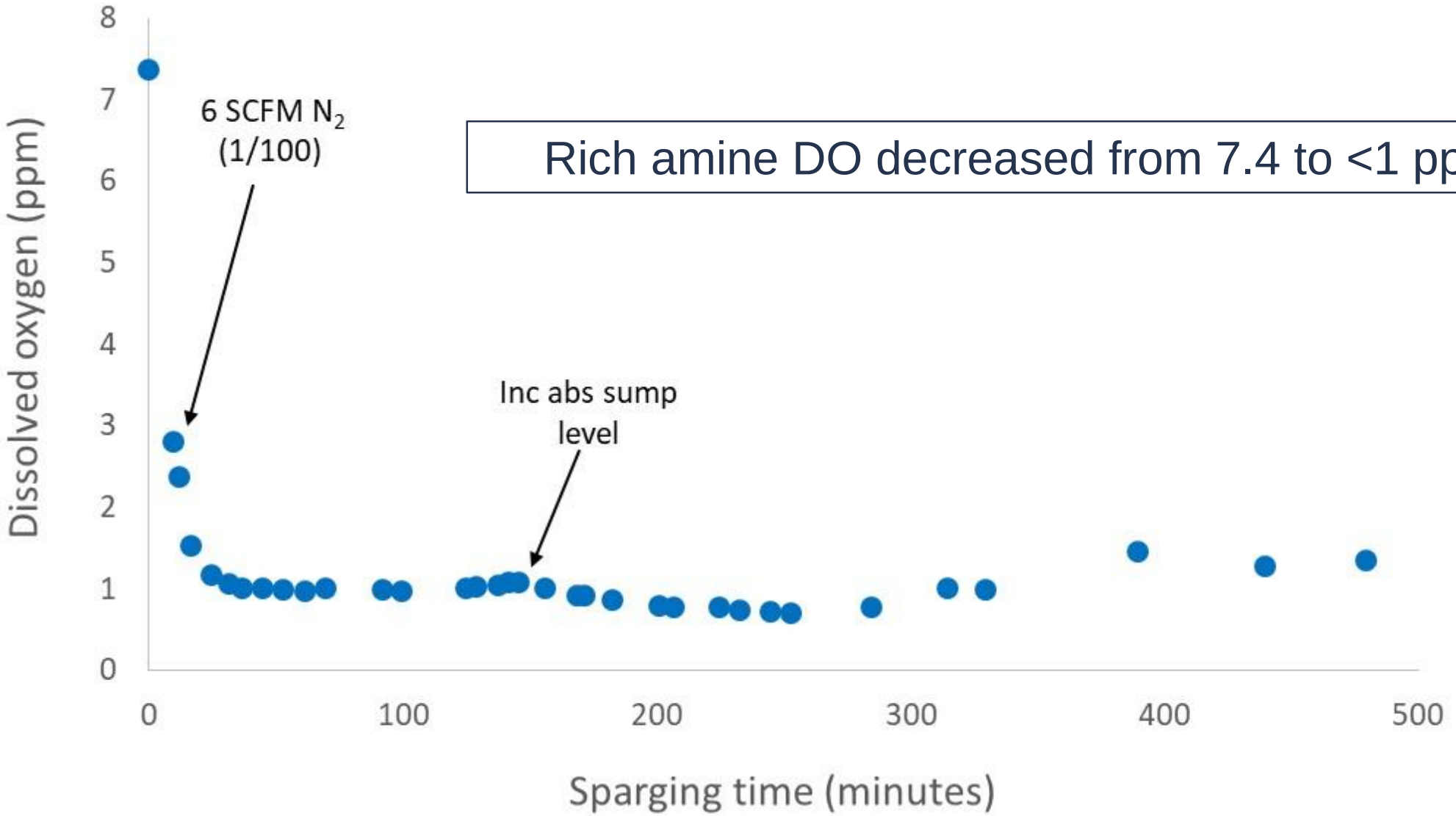
SRP plant University of Texas

Wire mesh mist eliminator

Mist eliminator and sparge pipe installed in absorber

N_2 ~1% of total gas traffic in absorber column

Pilot testing at SRP results

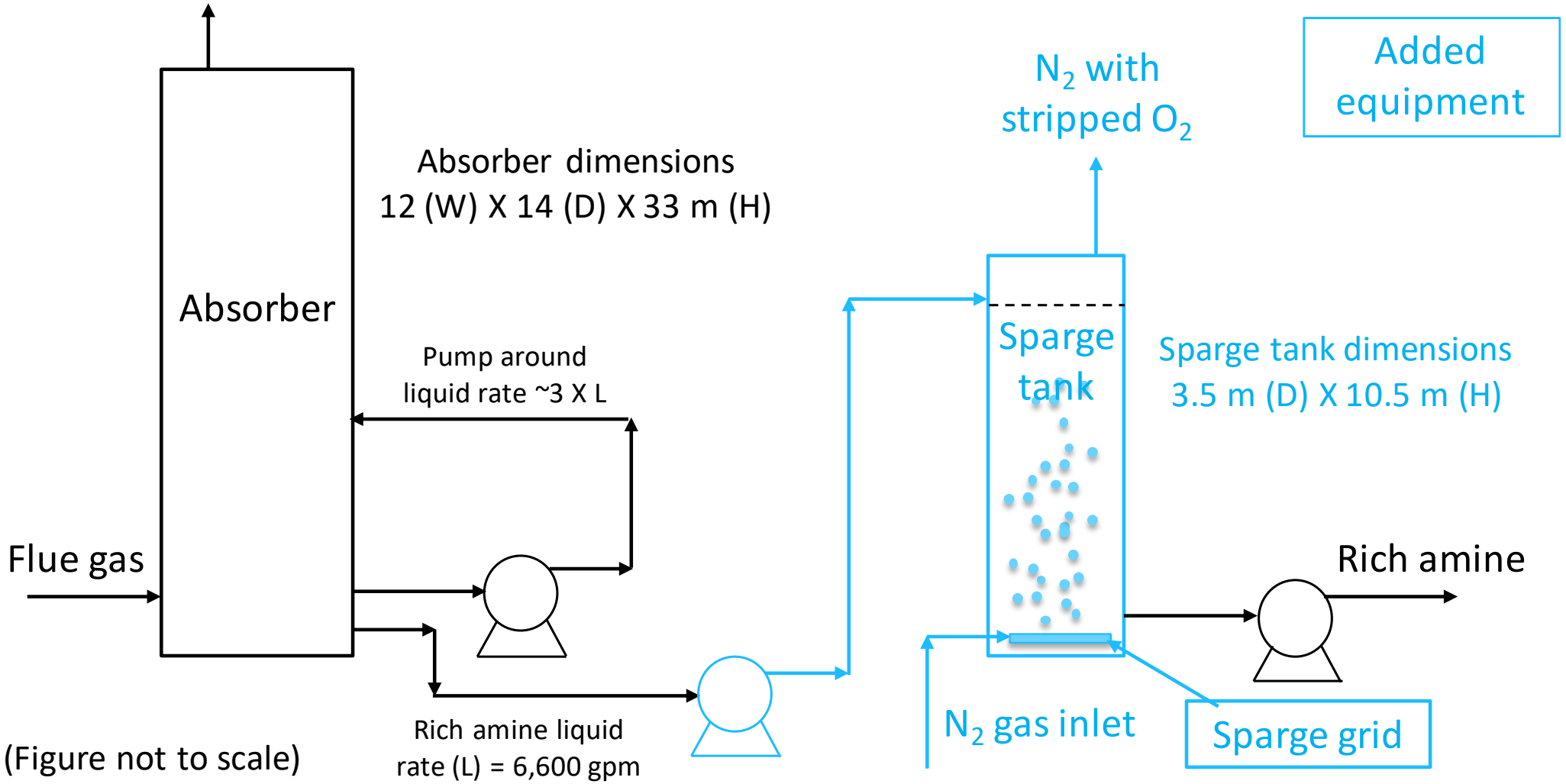


Rich amine DO decreased from 7.4 to <1 ppm

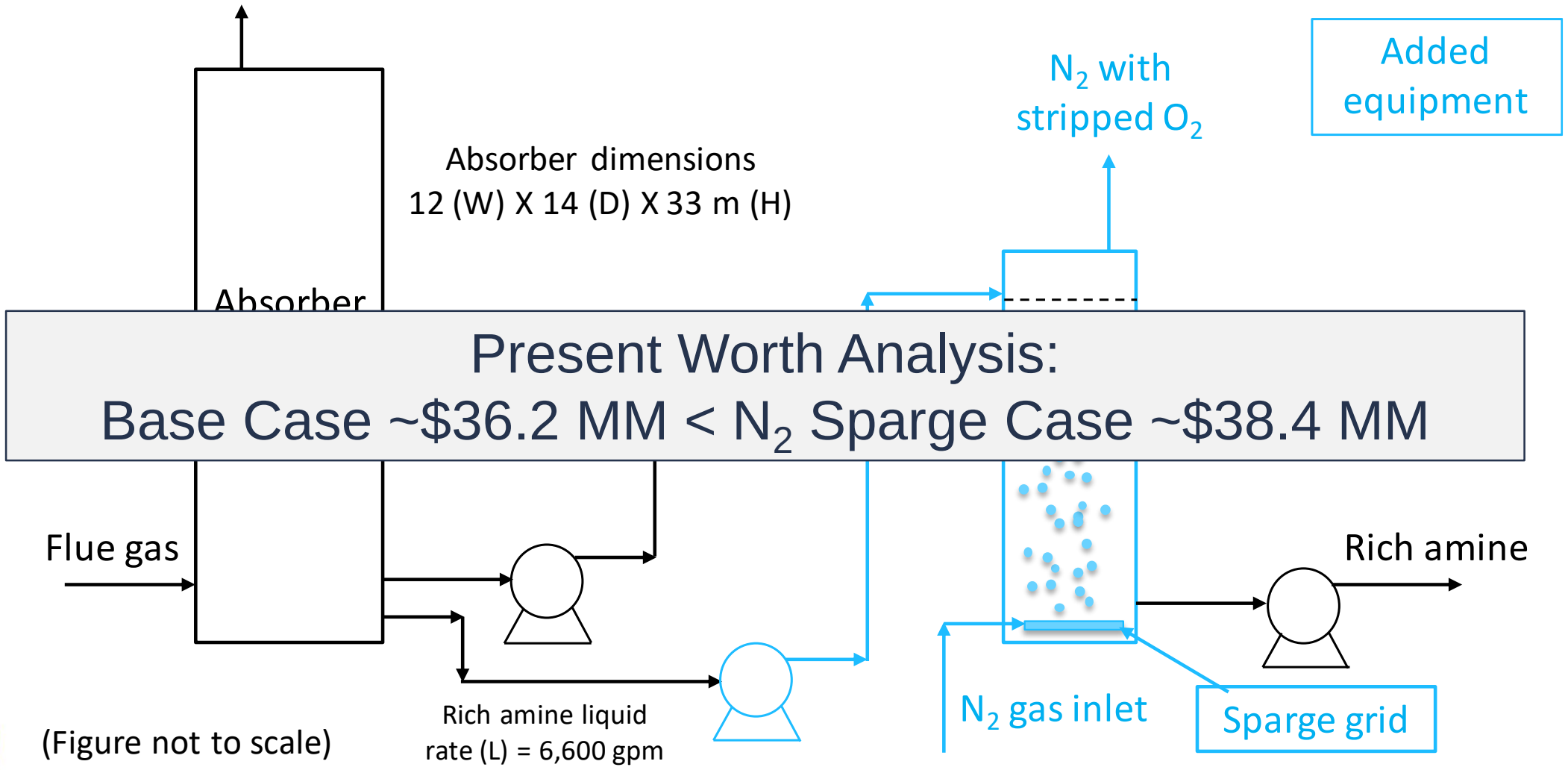
Full scale application analysis

- N_2 sparging model developed to estimate DO stripping in separate vessel
- Assumed mass transfer with a straight equilibrium line
- Mass transfer k_La and liquid hold up correlations from literature
- Used Henry's Law to estimate incoming DO concentration
- Modeled tank diameter & height as *f*(N_2 gas rate) at fixed DO removal

Full scale application – analysis using Mustang FEED as base case

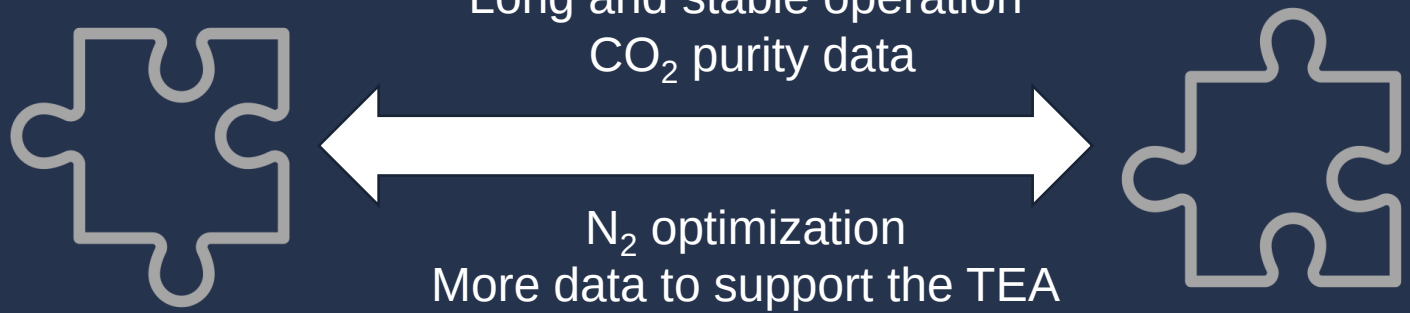


Full scale application results mixed on economic benefit



Gaps and way forward

Remaining gaps and way forward



DORA

Demonstrate DORA's versatility with different solvent and flue gases

Improve material compatibility

N_2 sparging

More mass transfer data to improve design

Minimize sparge rate

Effect of foaming

Acknowledgements

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LAUNCH

Open Meeting
15th February 2023

Controlling degradation (cost estimation)

Accelerating CCS Technologies | Bundesministerium für Wirtschaft und Energie | The Research Council of Norway | Rijksdienst voor Ondernemend Nederland | Department for Business, Energy & Industrial Strategy | U.S. DEPARTMENT OF ENERGY

Controlling degradation (cost estimation) - objectives

Techno-economics and guidelines

- Develop a techno-economic evaluation categorizing a number of solvent degradation control options
- Compare techno-economic evaluations against benchmark solvent degradation control options i.e. regular replacement, bleed and feed, thermal reclaim, ion exchange
- Determine optimum solvent degradation control options

Techno-economic evaluation - baseline plant

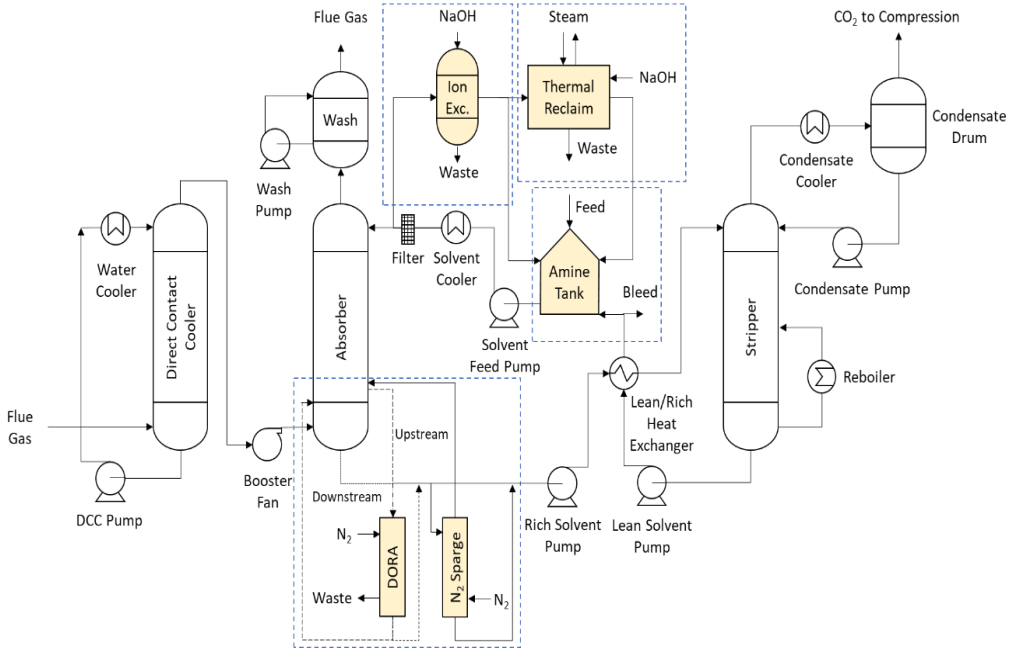
Case	1	2	3	4	5	6
Plant	PC			EfW		CCGT
Rating (MW_e)	1000			200 kt waste/yr		420
Reference	ALIGN Class 4/5			ALIGN Class 4/5		Sherman FEED
O_2 ,CO_2 (%vol)	3.5, 14.6			7.9, 9.8		12.1, 3.9
Solvent	MEA	CESAR1		MEA	CESAR1	MEA
Concentration (%wt)	29.5	39.3	39.3	29.5	39.3	35
Capture (%)	90	90	95	90	90	85
Capture ($ktCO_2/yr$)	5,900	5,900	6,200	215	212	1132
Availability (%)	85	85	85	90	90	57
Capture Cost (€/tCO_2)[1]	30.3	25.6	25.1	35.8	34.8	85.9[2]

[1] With original solvent management OPEX removed
[2] Excluding cost of CO_2 compression

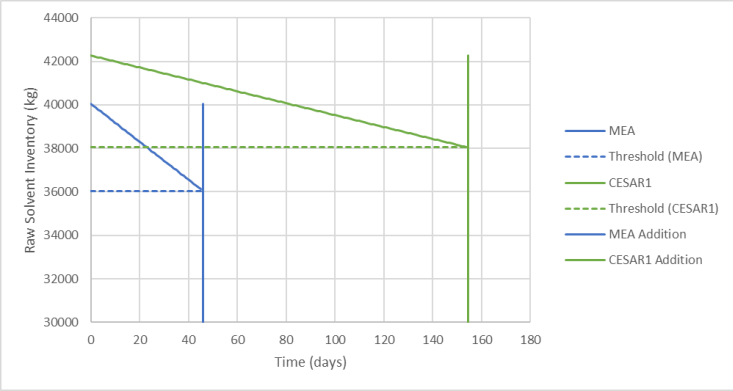
Techno-economic evaluation - solvent management

- The selection of solvent management options to be included followed discussion between the partners of the findings and extent of data available from testing

	Technique	Test Finding	Data Source
Corrective (Benchmarks)	Bleed and Feed	Limited effect MEA	-
	Thermal Reclaim	-	TNO
	Ion Exchange	Ionic + cationic	RWE Pilot
Preventative (Advanced)	DORA	90% DO reduction	TNO
	N_2 Sparging	90% DO reduction	UoT
	Thiosulphate Dosing	Ambiguous	TNO
	Non-metallic MOC	Insufficient data	TNO/BEFORM
	Residence Time	Ambiguous	-



Techno-economic evaluation - basis



- Oxidative rate is calculated using TNO's DNM; $r = -\frac{d[MEA]}{dt} = -\frac{1}{a}\frac{d[O_2]}{dt}$
- Oxidative/Thermal/Heat Stable Salts defined as 50/25/25%
- Total degradation rate (kg/day) can be reduced by DORA or N_2 sparge
- Benchmark options applied to avoid complete replacement of solvent

Process Input
- Process model output
- DNM parameters
- Economic parameters

→ Oxidative degradation in Absorber/ Sump/HEX

Total degrade rate

- Stoichiometric factor
- Degrade product split
- Preventative option

→ Time to threshold (cycle time)

Solvent, chemicals, waste, utilities

- Clean threshold
- Corrective option

→ Fixed and Variable OPEX

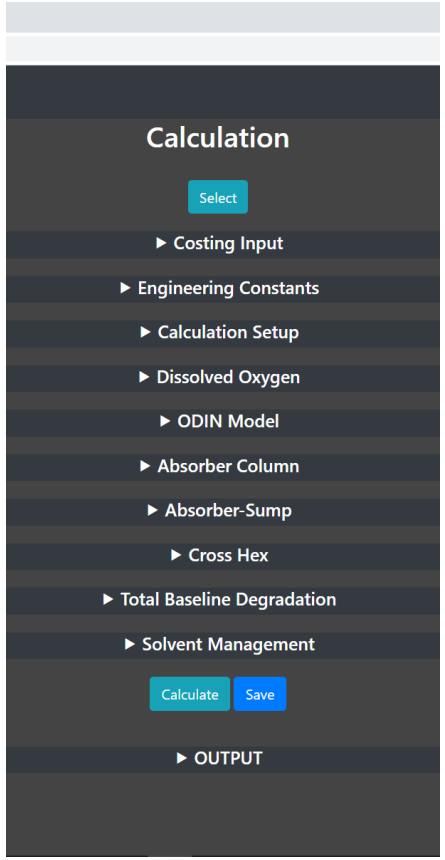
Total Plant Cost
Electricity, N_2, consumables

User manual input

→ OOM CAPEX and Annual OPEX

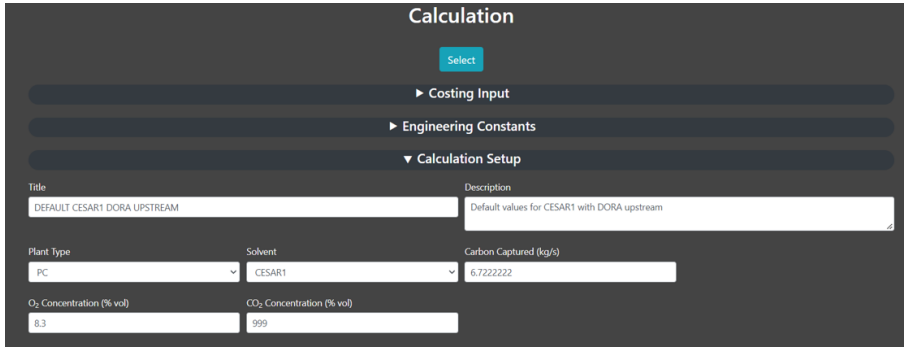
€/tCO_2

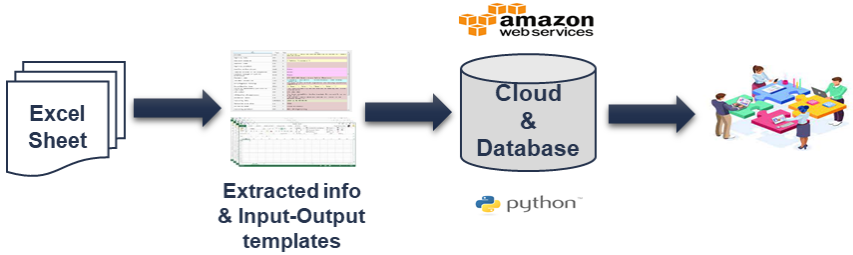
Techno-economic evaluation - software tool



	Option	1	2	3	4	5
	Baseline					
Prevent	DORA D/S					
	DORA U/S					
	N2 Sparge					
Correct	Bleed and Feed					
	Reclaim					
	Ion Ex					

Case Matrix

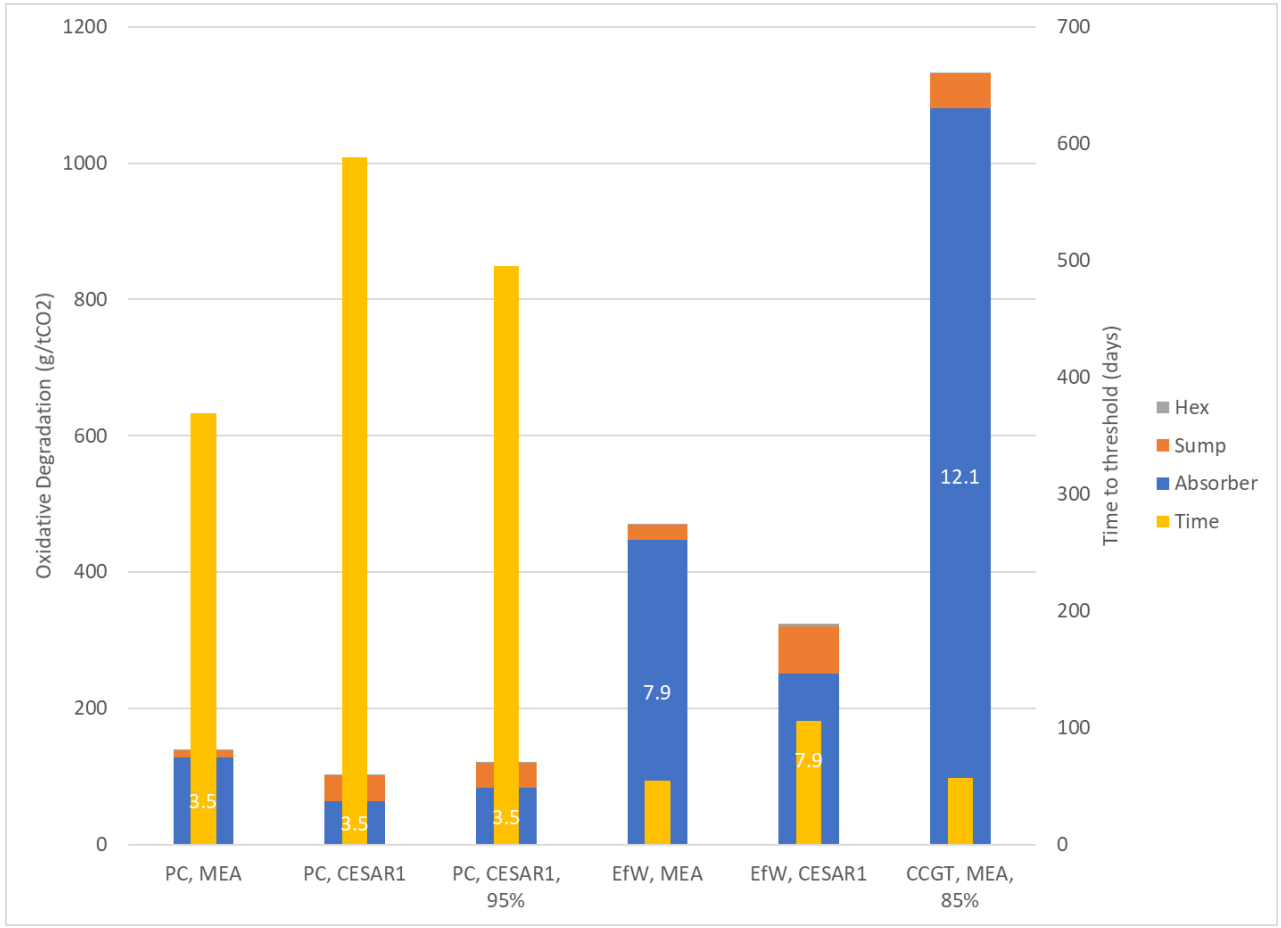




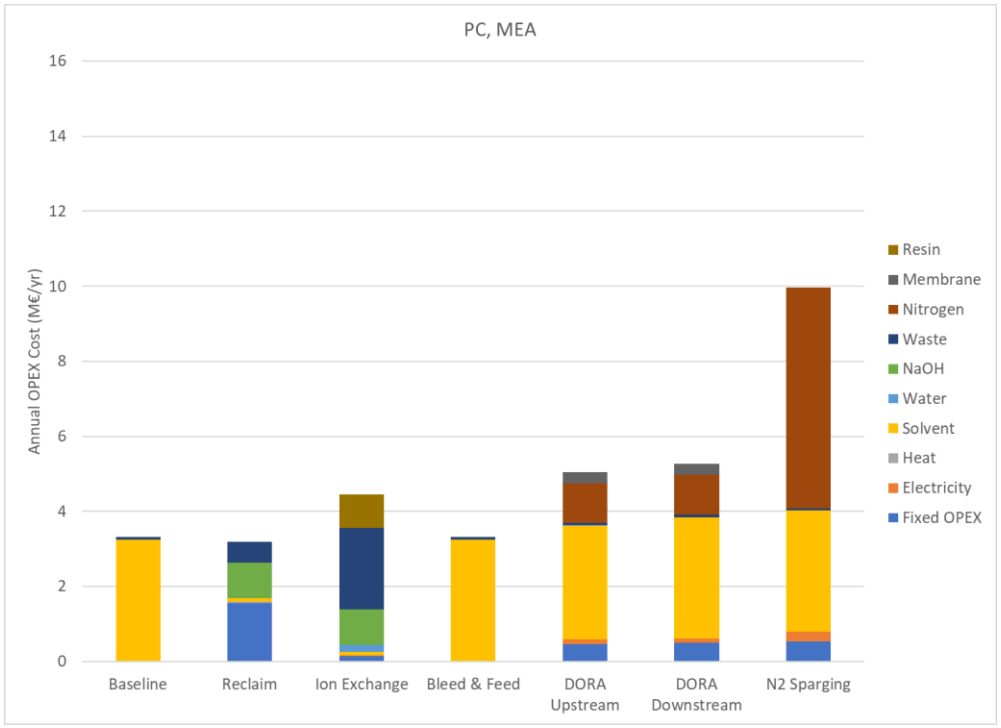
- Flexible code allows standardisation, version control and secure storage
- Cloud-based tool to be made available as a web application
- Ability to set up, run and save new calculations to a database

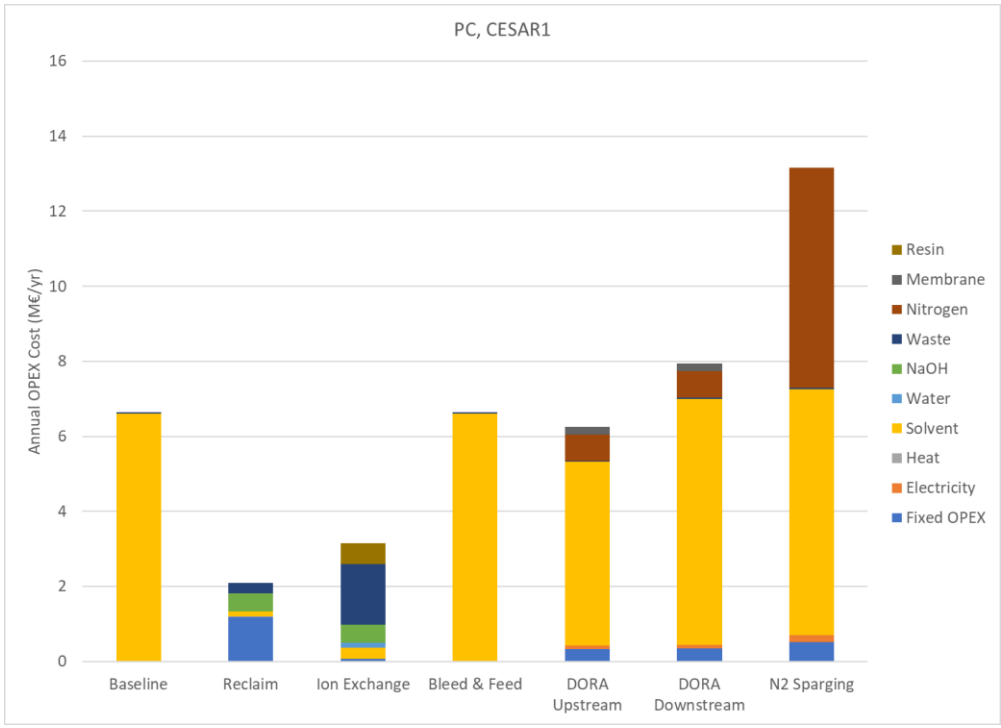
Techno-economic evaluation - results

- DNM output was calibrated to RWE pilot plant for each solvent = start of exponential degradation for MEA
- Clear impact of oxygen, temperature and residence time on total rates between plant
- Total degrade rate lower for CESAR1
- Greater proportion of degradation in absorber sump with CESAR1 solvent
- Solvent life 12+ mths for PC but 2-3 mths for EfW and CCGT



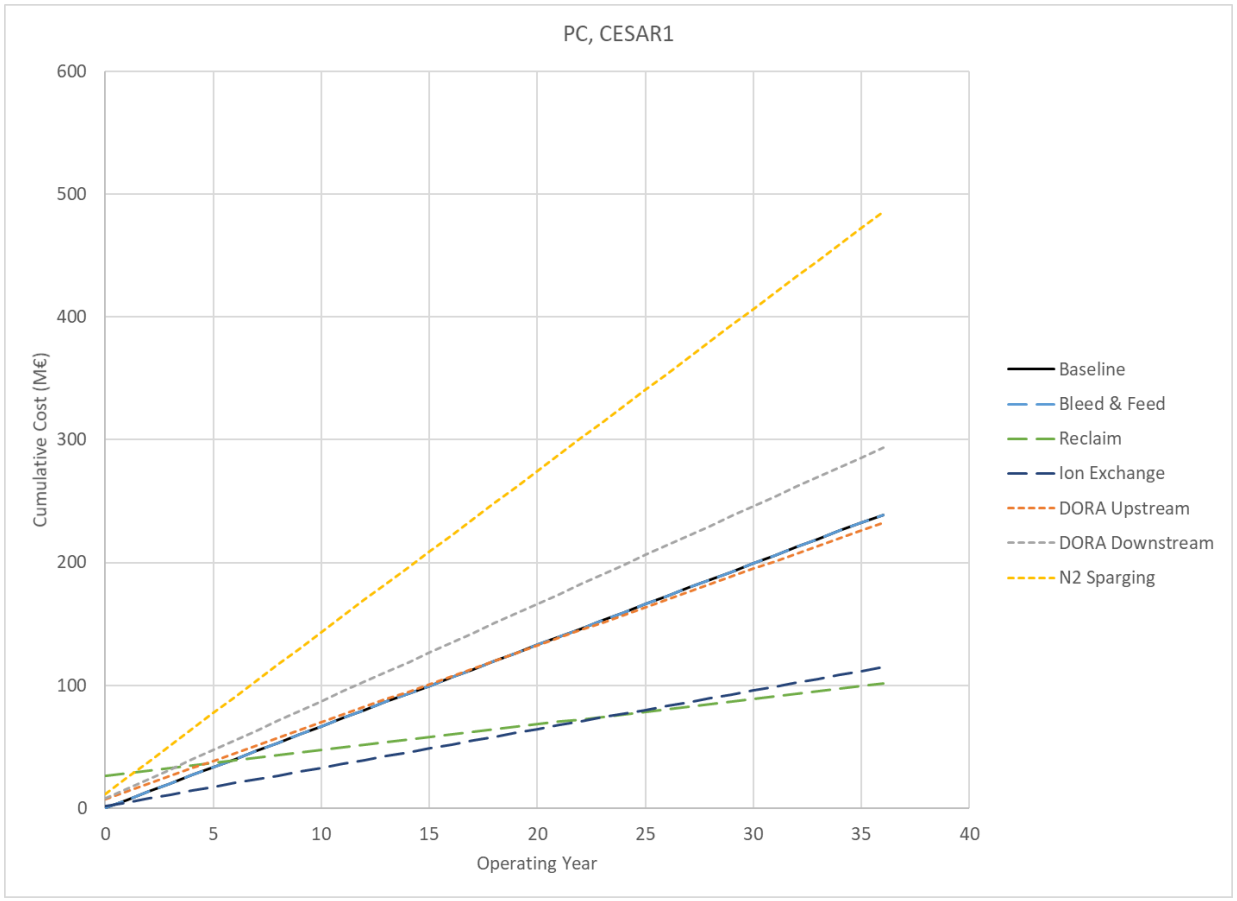
Techno-economic evaluation - results





- Lowest variable OPEX, but highest CAPEX, for thermal reclaim
- Higher solvent related OPEX, but lower CAPEX, for CESAR1 (2/3 inventory versus 4x cost)
- Nitrogen costs currently significant for novel preventative techniques

Techno-economic evaluation - results



- For PC and EfW with MEA, no cost advantage of any technique. Payback of thermal reclaim for CCGT after 12 yrs
- For PC and EfW with CESAR1, thermal reclaim or ion exchange cost effective
- DORA (upstream) shows potential for advanced solvent if economies of scale can be realised
- Reduction in oxidation not sufficient to justify combination of technique types
- With increasing capture rate there are incremental savings for techniques using less solvent

Acknowledgements

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Thank you for listening

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