Laboratory for Analysis and Synthesis of Chemical Systems

Université de Liège

Study of 2-ethanolamine degradation

UTCCS 1

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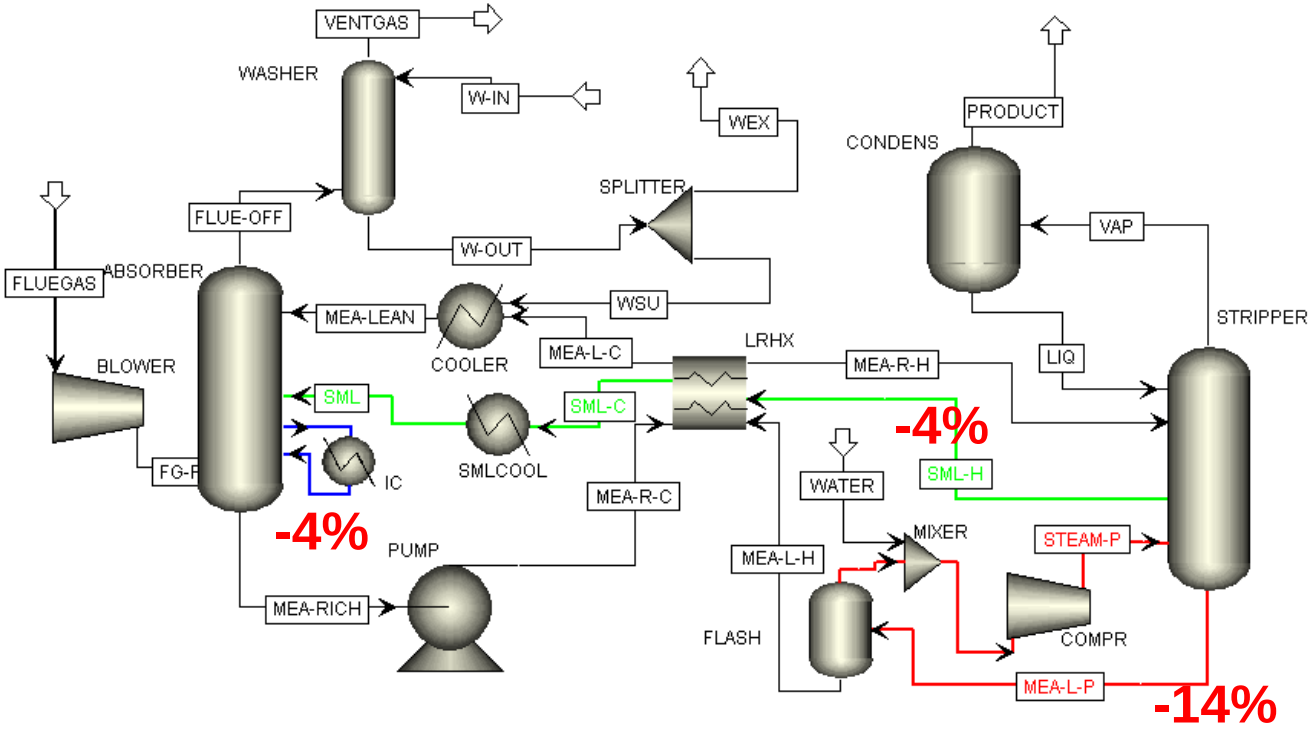
1. Introduction

1. Introduction

Original topic: Simulation and optimal conception of the post-combustion CO_2 capture process



However, simulation does not consider some important parameters!

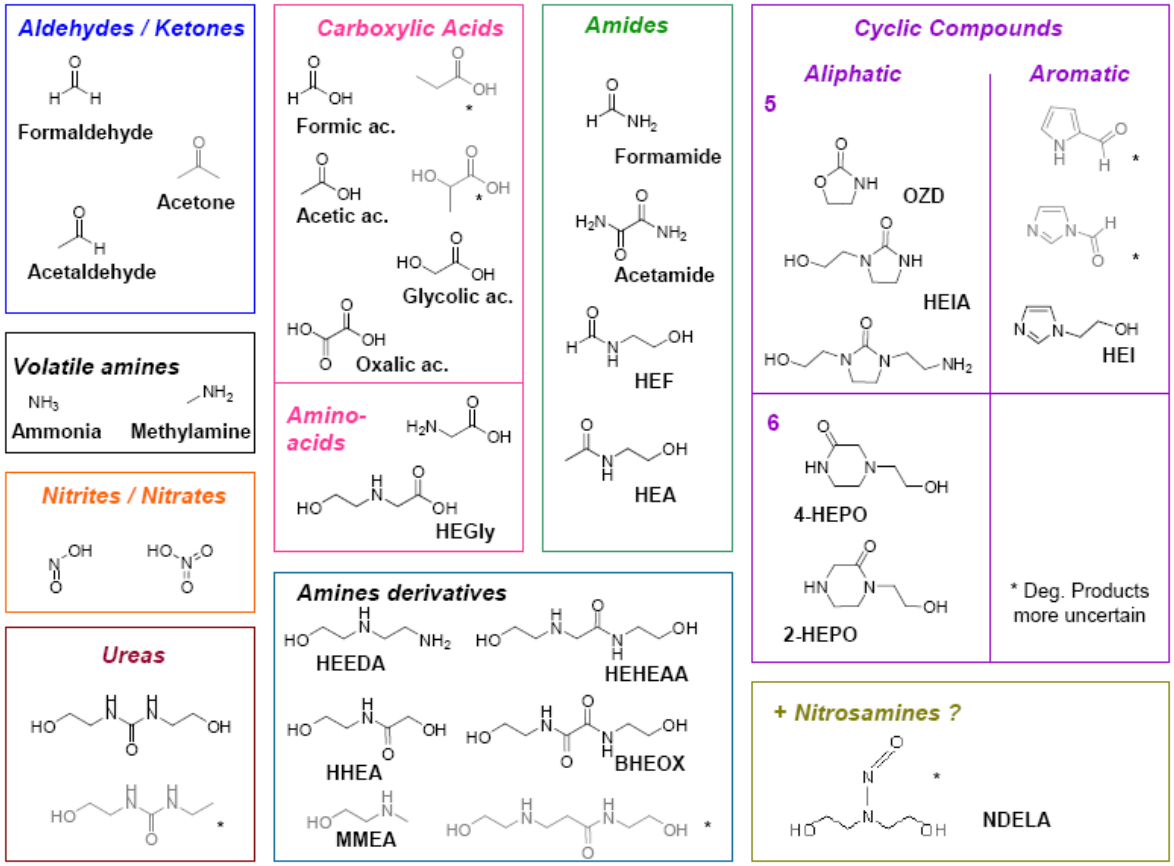
1. Introduction

- Focus set on solvent degradation
  - Process operating costs:
    - *Solvent replacement: up to 22% of the CO_2 capture OPEX[1]!*
    - *Removal and disposal of toxic degradation products*
  - Process performance:
    - *Decrease of the solvent loading capacity*
    - *Increase of viscosity*
    - *Foaming, fouling*
  - Capital costs
    - *Corrosion*

[1] Abu Zahra M., 2009. Carbon dioxide capture from flue gas, PhD Thesis, TU Delft, The Netherlands.

1. Introduction

- 3 types of degradation mecanisms: temperature, O_2, CO_2[2]



[2]Lepaumier H., Picq D., Carrette P.L., 2009. Degradation study of new solvents for CO_2 capture in post-combustion. Energy Procedia 1, 893-900.

2. Degradation Test Rig

2.1 Objectives

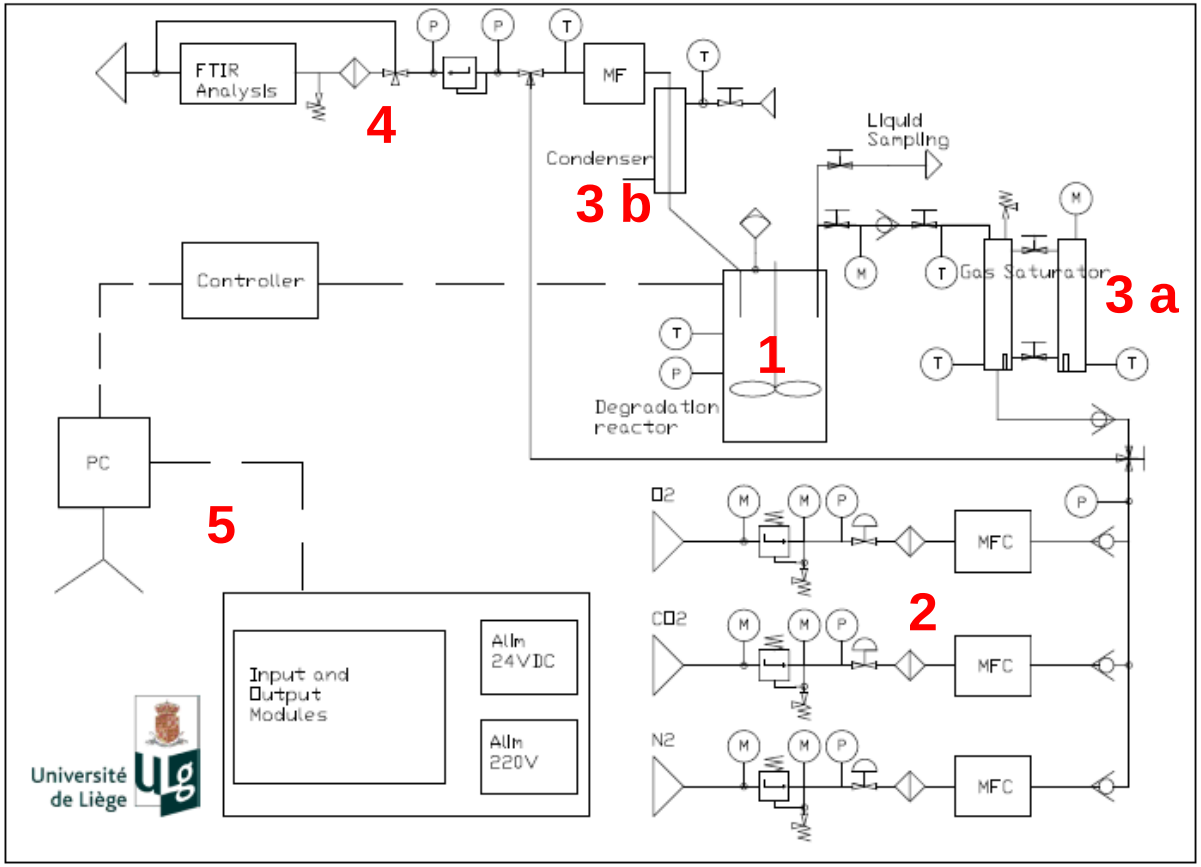
- Detailed screening of solvent degradation
- Study of the impact of operating conditions:
  temperature, gas composition and flow rate, …
- Study of the effect of additives: *degradation inhibitors, metals*

=> Need for accelerated degradation conditions

- *High temperature (up to 140°C), high pressure (up to 25 bar)*
- *Enhanced gas-liquid contact*
- *Varying gas flow rate and composition*

2.2 Degradation Test Rig

1. Reactor
2. Gas supply
3. Water balance
4. Gas flow
5. Control panel



2.2 Degradation Test Rig

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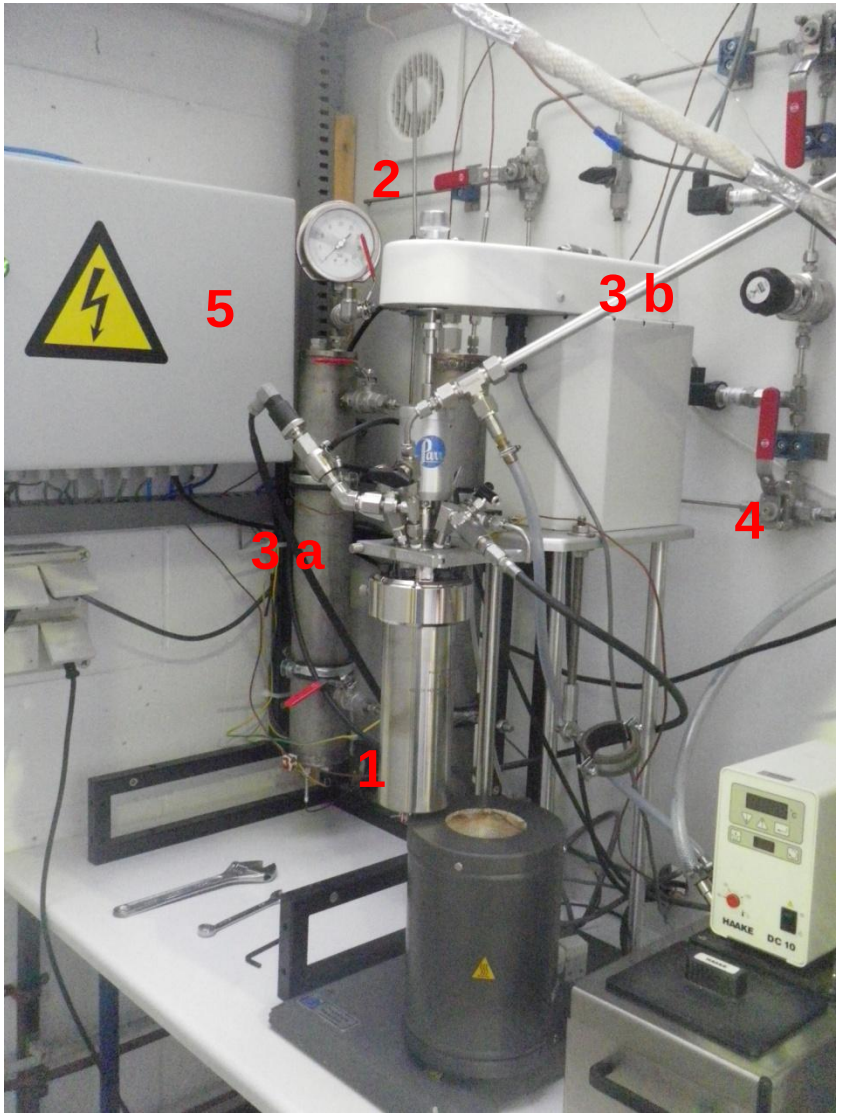


2.3 Analytical Methods

- Liquid phase:
  - **HPLC** (High Pressure Liquid Chromatography): *MEA quantification*
  - **GC-FID** (Gas Chromatography): *identification & quantification of the degradation products*
  - **IC** (Ionic Chromatography): *quantification of organic anions*
  - **AAS** (Atomic Absorption Spectroscopy) and **CE** (Capillar Electrophoresis): *quantification of inorganic ions*
  - **Karl-Fischer** Titration: *water quantification*

- Gas phase:
  - **FTIR** (Fourier Transform Infrared Spectroscopy):

    NH_3 and MEA quantification

3. Solvent degradation

3.1 Results Summary

- First test-campaign with MEA

	Operating conditions												
Name	Experiment Start	Experiment end	Length [Days]	Parameter tested	T [°C]	P_{tot} [bar]	P_{O2} [bar]	P_{CO2} [bar]	P_{N2} [bar]	Gas flow [mln/min]	Solvent [wt% MEA]	Mass balance [%]	Problems
Experiment 1	19/02/2011	5/03/2011	14	Base case	120	4	0.2	3	0.8	80	30.00	not recorded	-
Experiment 2	24/03/2011	5/04/2011	12	Exp. Length/strong cond.	140	20	1	15	4	200	30.00	-3.33	Gas exhaust stopped due to crystal formation in the condenser, pressure up to 25 bar
Experiment 3	11/04/2011	25/04/2011	14	Temperature	120	20	1	15	4	200	30.01	10.07	-
Experiment 4	10/05/2011	19/05/2011	9	Pressure (N_2)	140	20	0.2	3	16.8	500	30.05	-1.43	Foaming, temperature sensor defectuous => heating stopped automatically
Experiment 5	27/05/2011	10/06/2011	14	Repetability	120	4	0.2	3	0.8	80	30.01	-62.33	Crystal formation in the condenser, pressure up to 20 bar for a few hours, mass losses
Experiment 6	1/07/2011	15/07/2011	14	Repetability	120	4	0.2	3	0.8	80	30.02	-47.60	Mass losses (150g)
Experiment 7	20/07/2011	3/08/2011	14	Batch	120	20	0.2	3	0.8	0	29.99	-0.33	Corrosion of the temperature sensor
Experiment 8	24/08/2011	31/08/2011	7	Temperature and gas flow	120	20	0	0	20	20	30.00	-2.33	-
Experiment 9	31/08/2011	9/09/2011	9	Temperature and gas flow	120	20	0	0	20	200	30.00	-3.70	Gas bottle empty (2 days), current shortage
Experiment 10	13/09/2011	27/09/2011	14	New base case	120	4	0.2	0.6	3.2	160	29.99	-11.30	Mass losses

3.1 Results Summary

➢ Experimental feedback of the 1st test campaign:

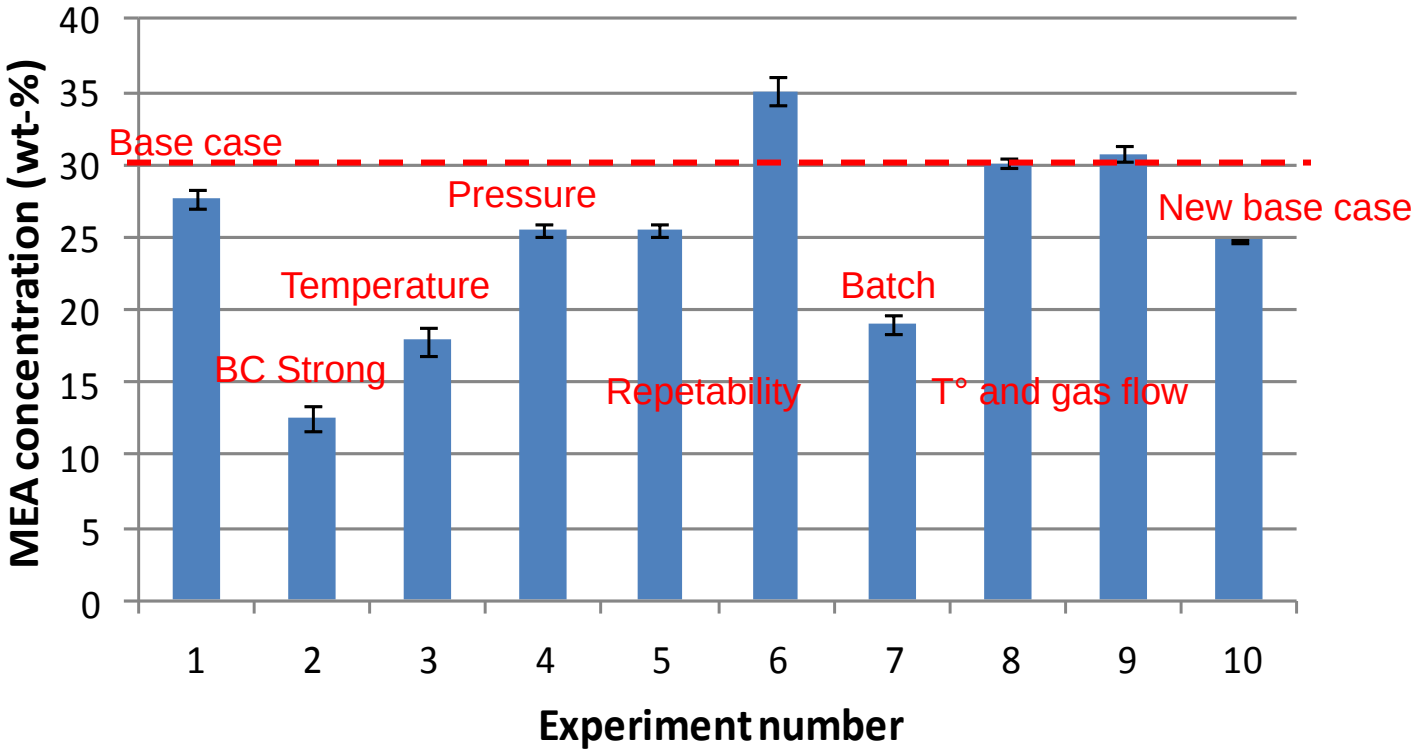
- Corrosion

- Crystal formation
- Mass balance regulation

- Agitation

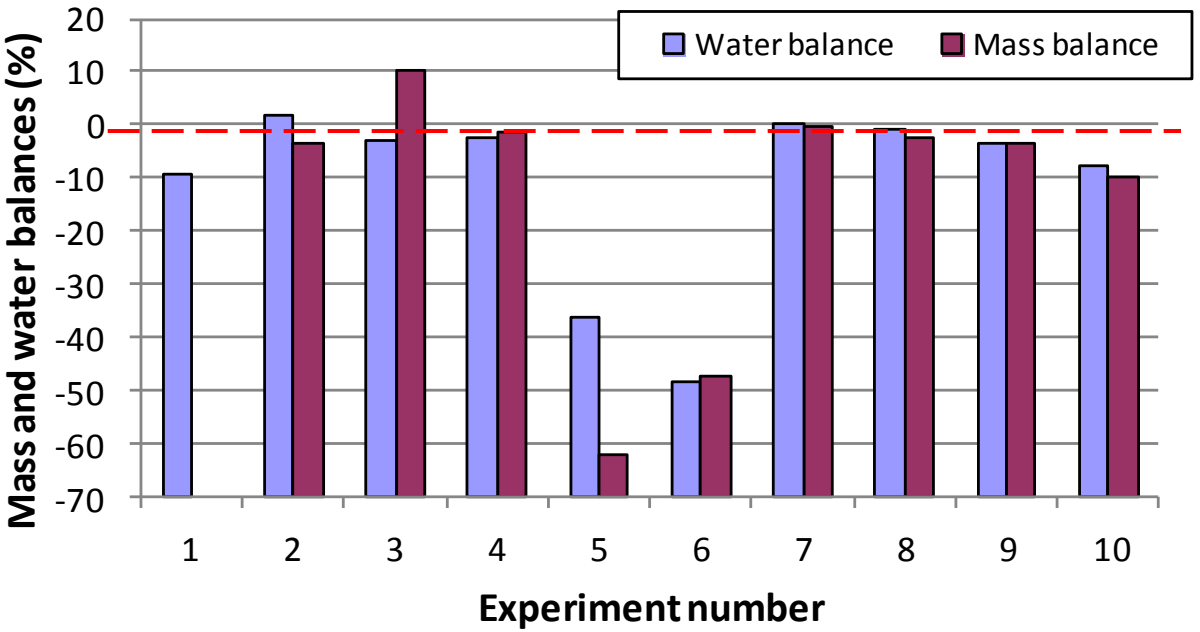
3.2 HPLC

➢ Quantification of MEA in degraded samples



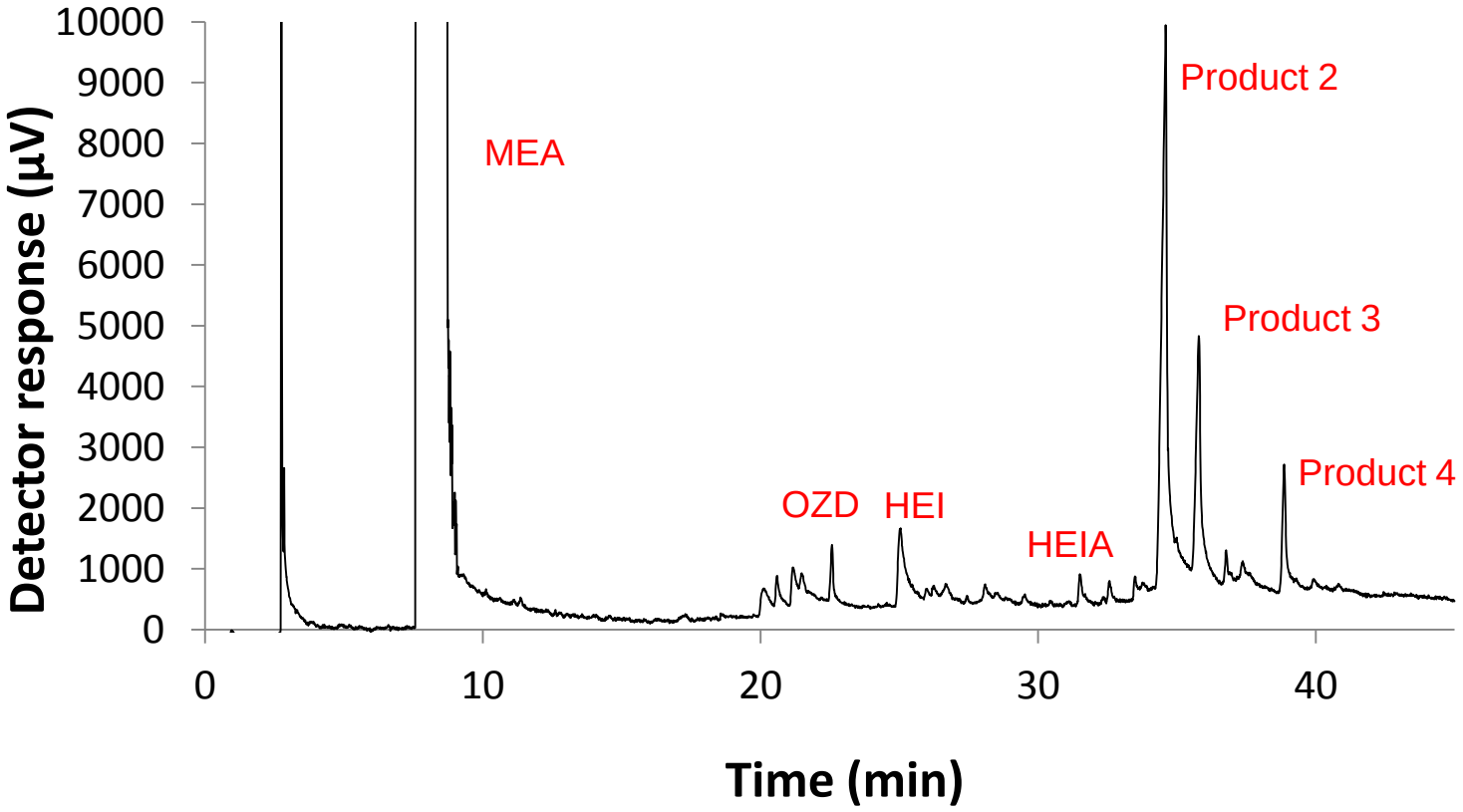
3.3 Karl Fischer Titration

- Quantification of water in degraded amine sample
- Good correspondance with mass balance results!



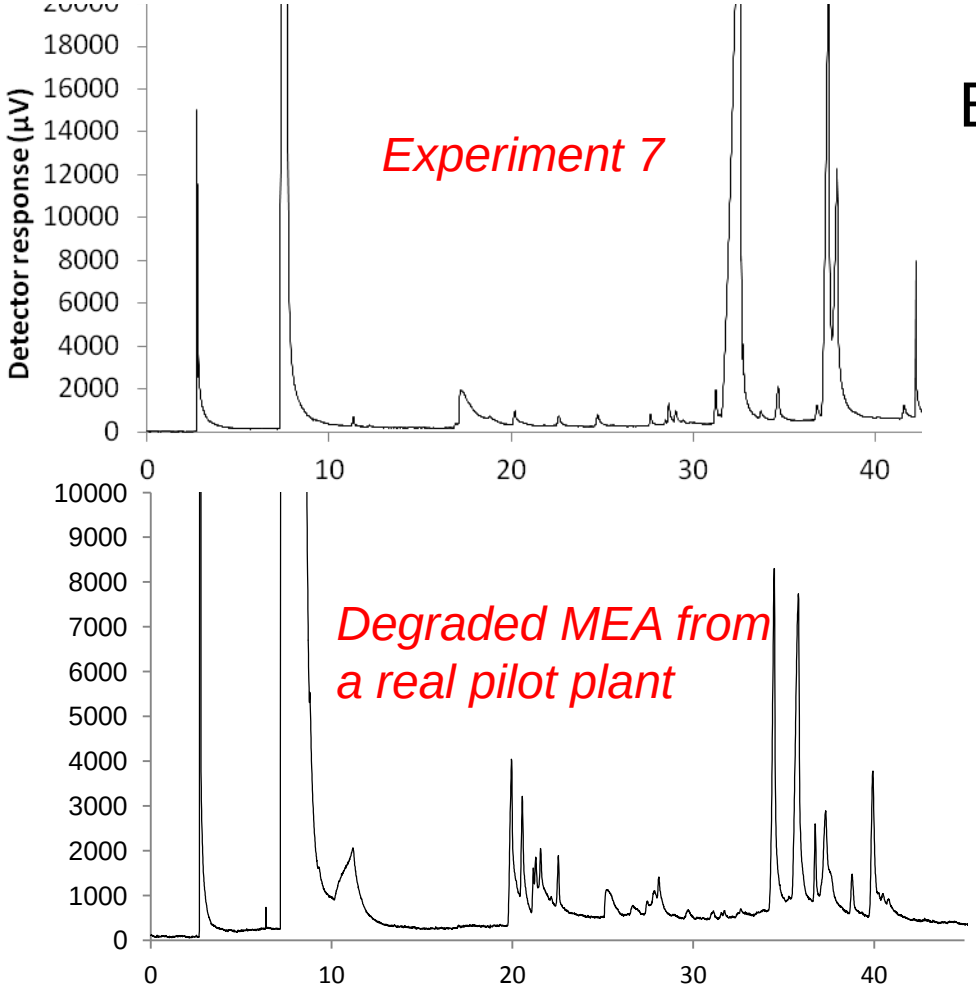
3.4 GC-FID

- Identification of degradation products



3.4 GC-FID

- Comparison with degraded MEA from a real pilot plant

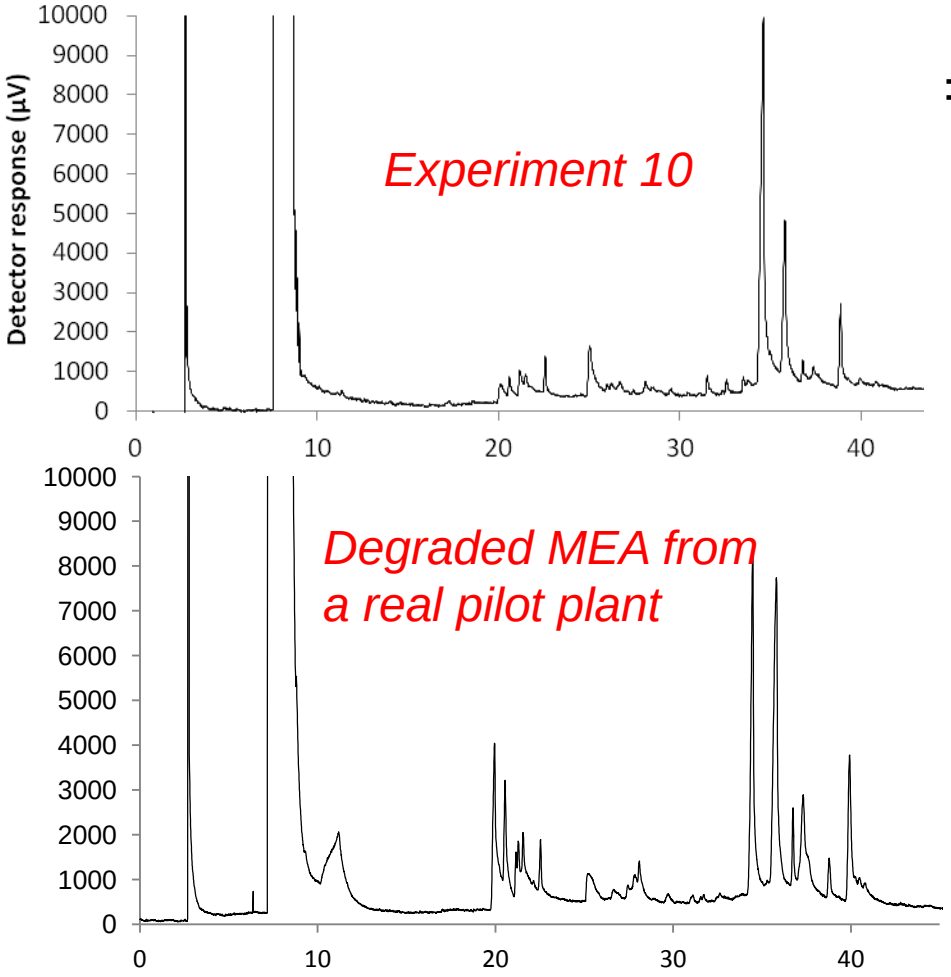


Experiment 7 in batch:

- 30 wt % MEA
- *120°C*
- *20 bar*
- *5%O_2, 75%CO_2, 20%N_2*
- *no gas flow*
- *2 weeks*

3.4 GC-FID

- Comparison with degraded MEA from a real pilot plant



=> Experiment 10 has been chosen as the new base case for next tests campaign:

- 30 wt % MEA
- *120°C*
- *4 bar*
- *5%O_2, 15%CO_2, 80%N_2*
- *160 mln/min gas flow*
- *2 weeks*

4. Conclusions and perspectives

4.1 Conclusions

- Experimental bench for the study of accelerated solvent degradation, with different analytical methods
- Detailed screening of MEA degradation is in progress
- Influence of temperature, gas flow rate, gas composition and pressure may already be observed
- The study of MEA degradation under accelerated conditions can be related to pilot scale results

  => *definition of a new base case*

4.2 Perspectives

- Second test campaign with MEA
  - influence of gas composition and temperature
  - influence of metals and degradation inhibitors.

- Construction of a simulation model for CO_2 capture including degradation results

- This model will be **validated** with pilot plant data

=> *Goal is to perform a **multi-objective optimisation** of the CO_2 capture process*

Acknowledgements

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F.R.I.A. – F.N.R.S

Thank you for your attention!