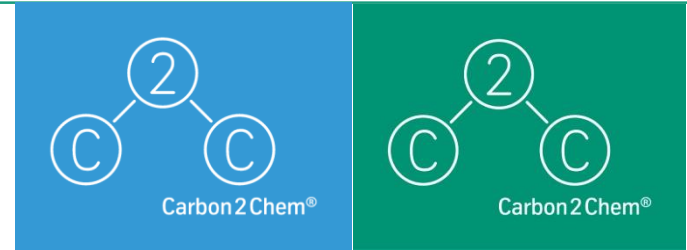


INDUSTRIAL SCALE-UP IN CARBON2CHEM® – DESIGN OF TEST CAMPAIGNS WITH THE DEMONSTRATION PLANT

Tim Schulzke, Fraunhofer UMSICHT

Dr. Matthias Krüger, thyssenkrupp Industrial Solutions

3rd Carbon2Chem® Conference, October 27th-28th, 2020

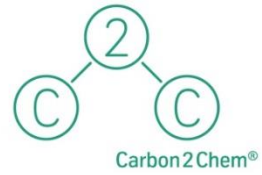
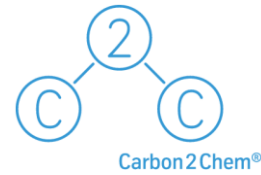


GEFÖRDERT VOM



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AGENDA



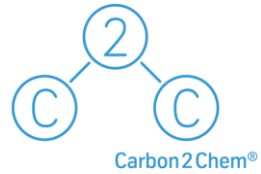
Part 1: Matthias Krüger, tkIS

- From laboratory to large scale plants: Problem definition and challenges during scale-up
- Summary of criteria for this project: towards the vision of a large scale Carbon2Chem® plant

Part 2: Tim Schulzke, Fraunhofer UMSICHT

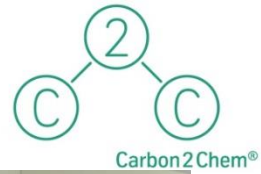
- Results from experimental facilities with clean (bottle) gases and real steel mill gases (Phase 1)
- Design of test campaigns with demonstration plant

Slides of Dr. Matthias Krüger



For a personal copy of Dr. Krüger's slides (3-11) please contact:
Dr. Matthias Krüger, thyssenkrupp Industrial Solutions, Dortmund
matthias.krueger@thyssenkrupp.com

Experimental facilities I

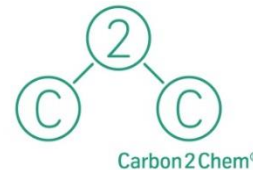


LCTS – Lab Catalyst Test Set-up

- Maximum operating conditions
 $T = 400\text{ }^{\circ}\text{C}$, $p = 100\text{ bar}$
- Reactor
 \varnothing_i 9.7 mm; length 270 mm
- Catalyst key indicators
ground commercial tablets
sieve fraction 250 – 500 μm
amount: up to 5 g
- Gas Feed
4 feed gases (CO , CO_2 , H_2 , N_2)
 $\sim 6 - 60\text{ Nm}^3/(\text{kg}_{\text{cat}}\text{ h})$



Experimental facilities II

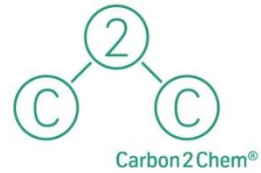


Pilot plant

- Maximum operating conditions
 $T = 350\text{ }^{\circ}\text{C}$, $p = 100\text{ bar}$
- Reactor
 \varnothing_i 19 mm; length 1,100 mm
- Catalyst key indicators
ground commercial tablets
sieve fraction 1 – 3 mm
amount: 20 – 100 g
- Gas Feed
5 feed gases (CO , CO_2 , H_2 , N_2 , Ar)
 $\sim 5 - 10\text{ Nm}^3/(\text{kg}_{\text{cat}}\text{ h})$



Experimental facilities III

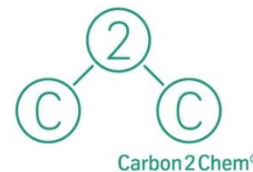


Demonstration plant

- Maximum operating conditions
 $T = 270\text{ }^{\circ}\text{C}$, $p = 89\text{ bar}$
- Reactor
 \varnothing_i 34.3 mm; Length $\approx 6,000\text{ mm}$
- Catalyst key indicators
commercial tablets $\varnothing 6 \times 4\text{ mm}$
amount: $\approx 6.5\text{ kg}$
- Gas Feed
4 feed gases (CO , CO_2 , H_2 , N_2)
 $\sim 5\text{ Nm}^3/(\text{kg}_{\text{cat}}\text{ h})$



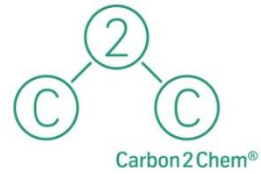
Experimental facilities



Commonalities and Differences

	LCTS	Pilot plant	Demonstration plant
T measurement	Reactor inlet + exit; 1 point in catalyst bed	16 points along reactor axis	36 points along reactor axis
T control	Electrical trace heating * Oil cooling jacket		Boiling-water cooling jacket
Plant set-up	Once through * Gas recycle		Gas recycle
Catalyst	Commercial methanol catalyst		
Catalyst size	Fine powder	Fragments	Commercial tablets
Online analysis, sample point	Reactor outlet		Condenser outlet
Online analysis	GC-MS/FID/TCD	GC-FID/TCD	TCD (H ₂) NDIR (CO, CO ₂ , CH ₄ , C ₂ H ₄)
Reaction regime	Primarily kinetic	Primarily kinetic; additionally equilibrium	equilibrium
Object of investigation	Catalyst performance; reactor model validation		Plant performance; plant model validation

Results from Phase 1



Benchmark tests

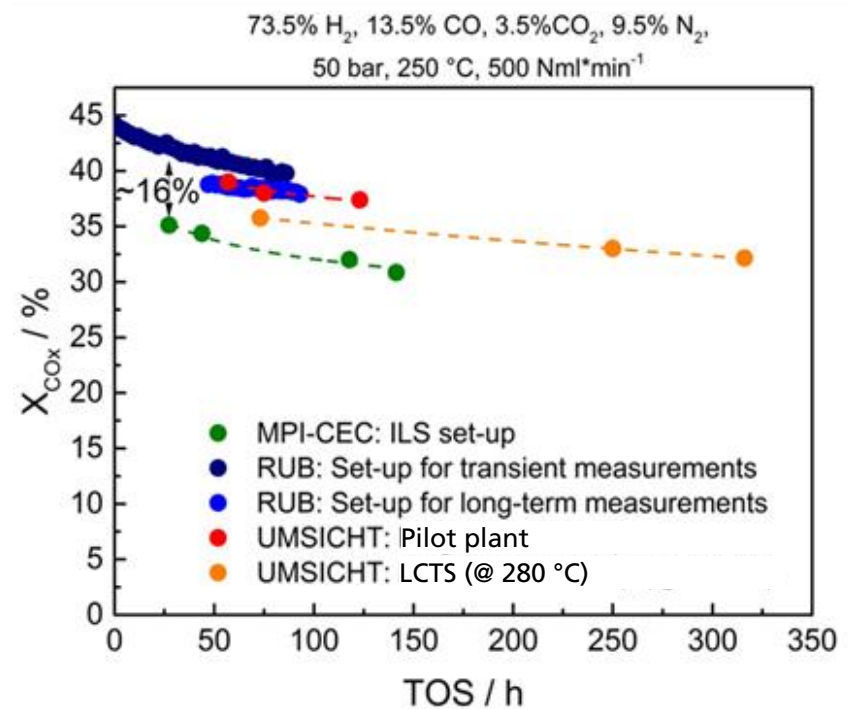
Comparison of different test set-ups operated by different consortium partners

⇒ good overall consistency

Definition: Carbon Oxide Ratio

$$\text{COR} = \text{CO} / (\text{CO} + \text{CO}_2)$$

Benchmark: $\text{COR} = 13.5 / 17 = 0.79$

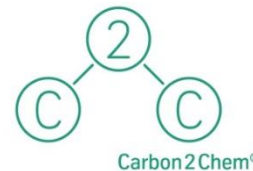


Results from Phase 1

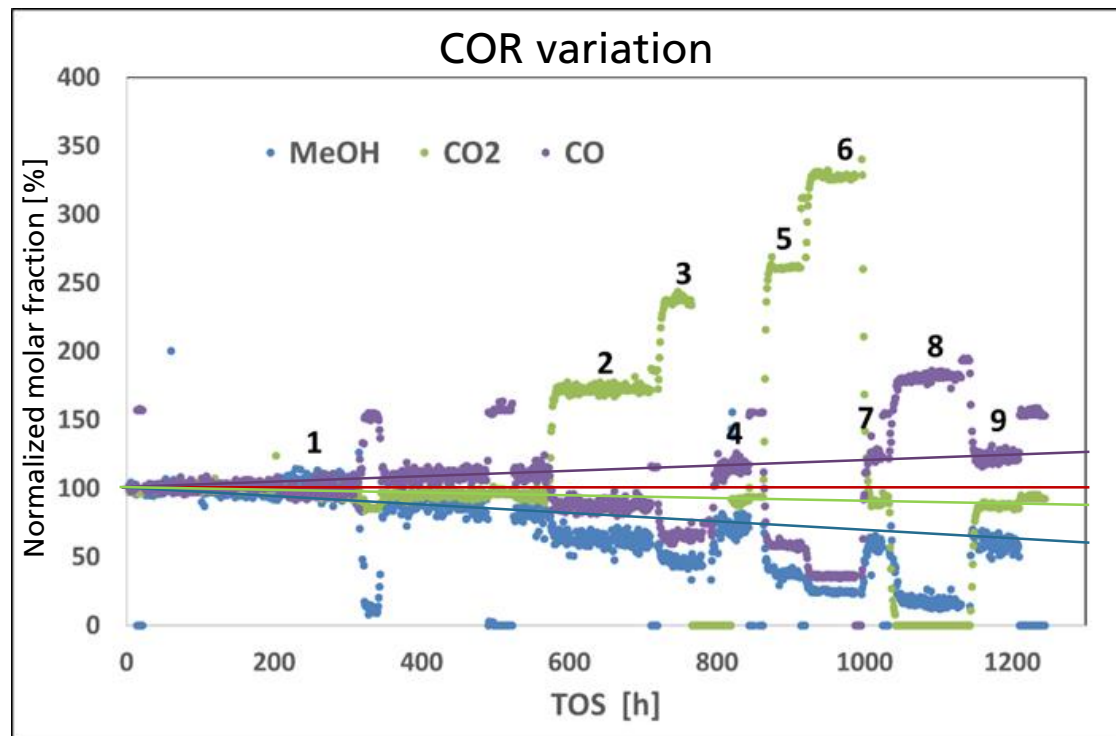
LCTS, bottle gases

73.5 % H₂, 9.5 % N₂

280 °C, 50 bar



Step	CO ₂ [Vol. %]	CO [Vol. %]	COR
1	3.5	13.5	0.79
2	7	10	0.59
3	10.5	6.5	0.38
4	3.5	13.5	0.79
5	12	5	0.29
6	17	0	0
7	3.5	13.5	0.79
8	0	17	1
9	3.5	13.5	0.79



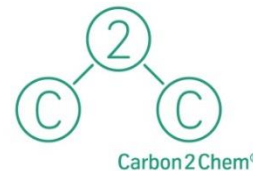
Results from Phase 1

LCTS, cleaned blast furnace gas, balance hydrogen from bottles

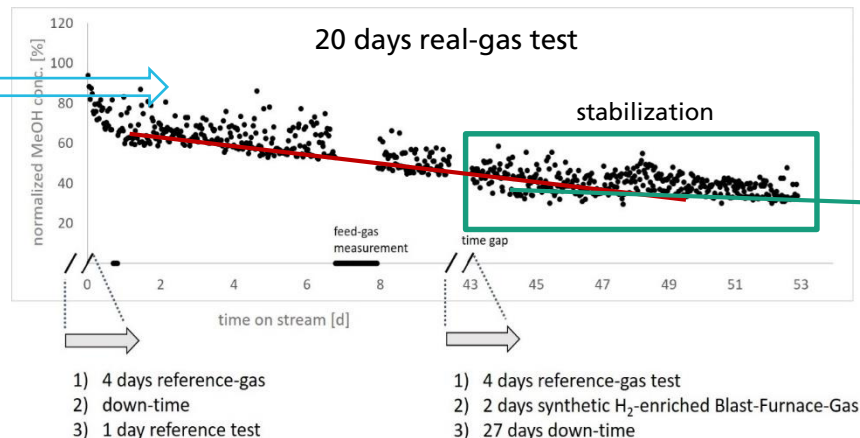
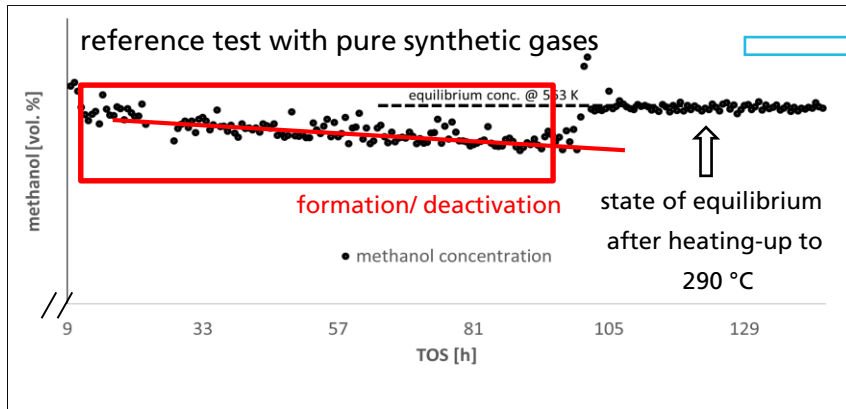
250 °C, 84 bar

Cleaned BFG:

≈ 25 % CO
≈ 23 % CO₂
≈ 4 % H₂
≈ 48 % N₂



325 Nml/min BFG } SN ≈ 2.4
425 Nml/min H₂ }

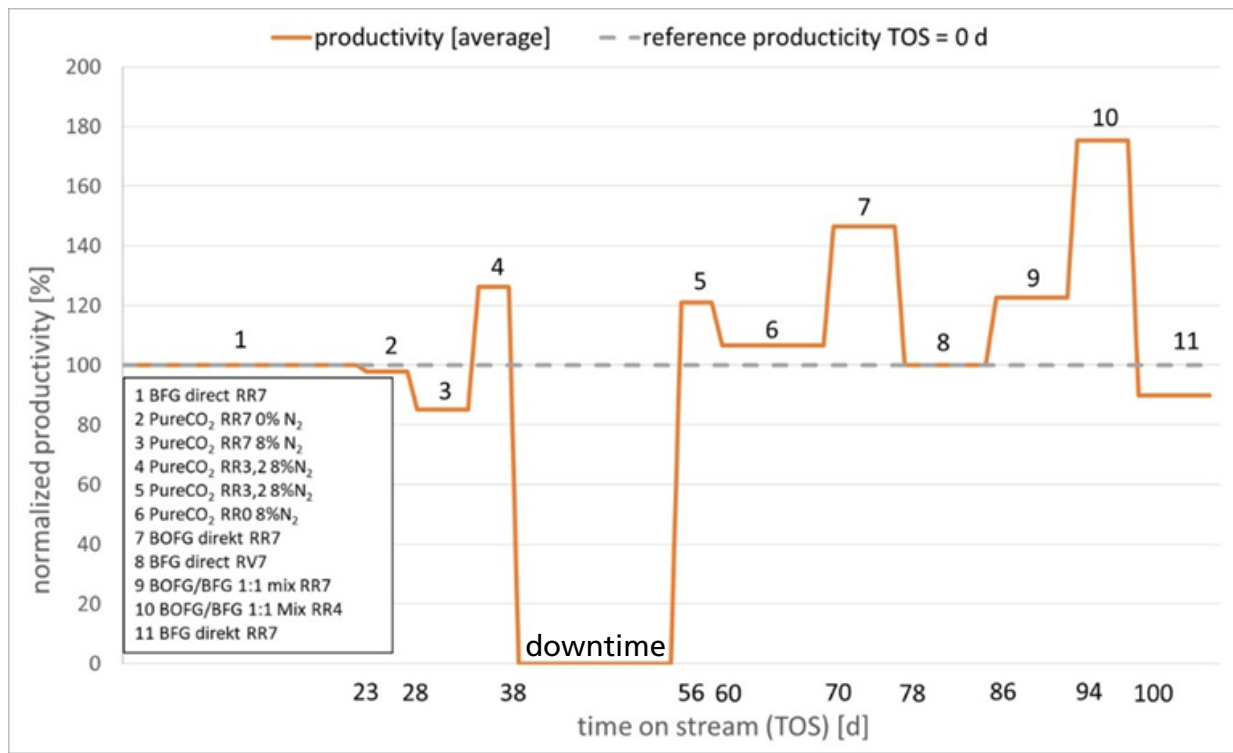
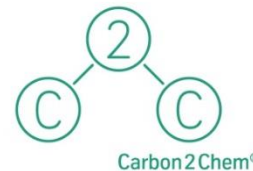


Results from Phase 1

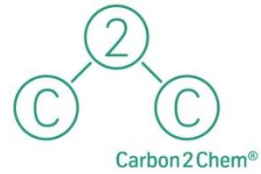
Pilot plant, bottle gases

Scenario comparison

SN \approx varying

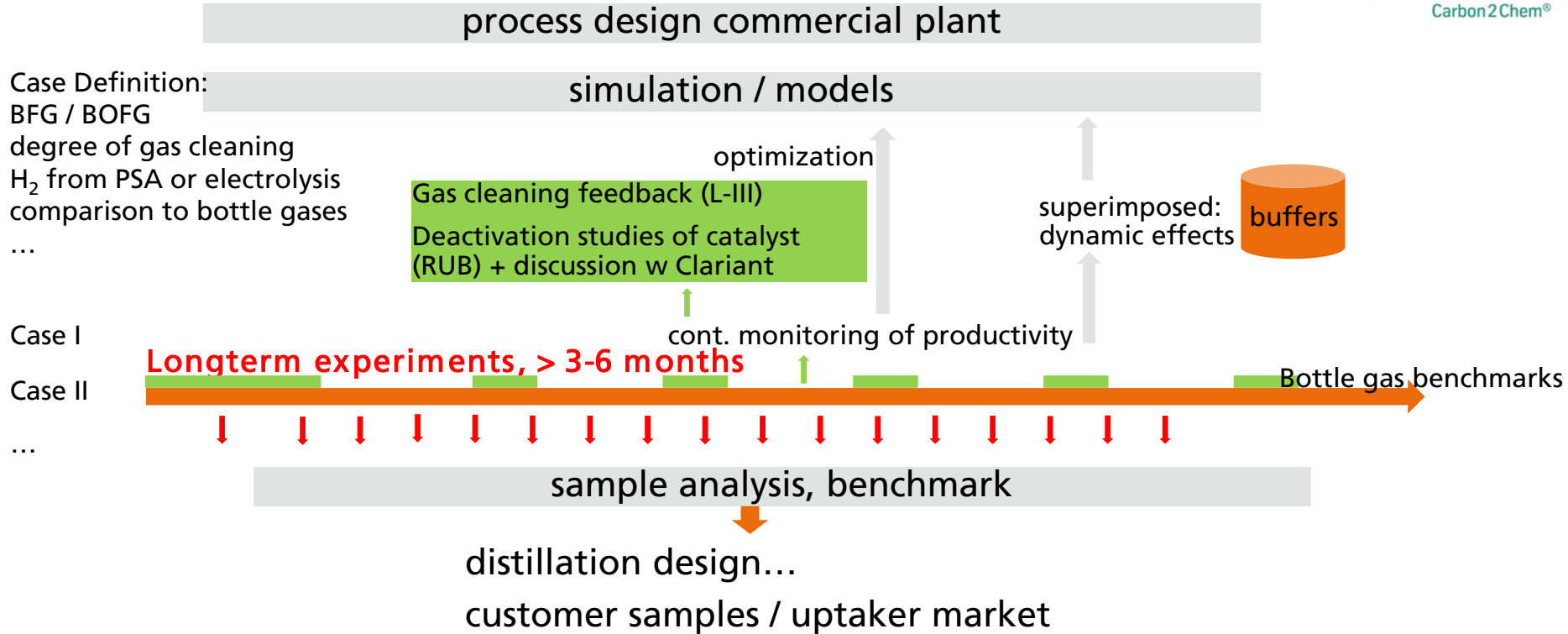
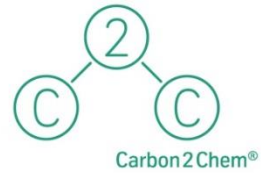


Demonstration plant modification

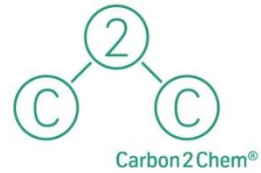


- Operation at UMSICHT in Oberhausen with
 - changing ratio of CO / CO₂ (originally designed for pure CO₂)
 - changing addition of N₂ to makeup gas
 - changing recycle ratio and makeup gas flow rate⇒ Determination of operating window
- Application for building permit at Carbon2Chem test facility in Duisburg
- Relocation of demonstration plant to Duisburg

Design of campaigns at steel mill



Summary



- Scale-up is a complex topic, not straight forward
- Carbon2Chem partners are experienced with successful scale-up
- First basis for scale-up is longterm stability of lab and pilot plants and experimentally validated reaction / reactor / plant models
- Stepwise transfer of results and campaigns to larger plants towards the vision of Carbon2Chem first-of-its-kind plant

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