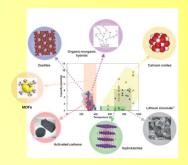
# SINTEF strength in performing CLC

# MATERIALS – selection and optimization

Experienced with most types of materials; e.g. oxides, carbonates, sulfides, sulfates, zeolites, MOF etc.



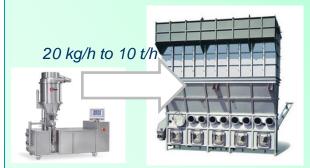
#### **Our expertise**

- Prediction based on;
  - Thermodynamics
  - DFT modelling
  - Material properties
  - Stability (chemical CO<sub>2</sub>, H<sub>2</sub>S, H2O, CO ..., at high pressure)
- Char. of process properties;
   HPTG, TG-DTA, sorbent
   isotherms, TPX type
   measurements etc.
- Char. using SEM/EDS, TEM, XPS, XRD etc.

#### **MANUFACTURING**

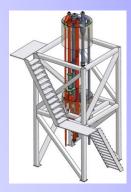


- Different type of powder synthesis
- Combustion
- Sol-gel, co-precipitation
- Hydrothermal
- Solid state reactions
- Spray pyrolysis
- Flame spray pyrolysis
- Different types of granulations
- Agglomeration
- Spray granulation
- Spray coating
- (Infiltration)
- Pre-industrial production methods



# Reactor testing

- Laboratory with 30 bar infrastructures for most gases (including H<sub>2</sub>S lab)
- ■Test several process conditions; TSA, PSA, CSA, VSA, redox, etc.
- ■Different process layouts; fixed bed, fluidized bed, moving bed etc.
- Small and medium rig for testing materials3kW and 150kW rig





# Several design & fuel

- ENCAP (NG and Coal) 2005 (EU FP6)
  - SINTEF developed CaMn<sub>1-x</sub>M<sub>x</sub>O<sub>3</sub> (M=Ti, Fe, Cr), TNO fixed bed, IFP rotating fixed bed, Chalmers fluidized and bubbling bed
- ÉCLAIR / ACCLAIM (Alstom) (RFCS)
  - Demonstration of 1MW demo plant CLC fluidized bed, coal fired
- DemoClock (EU FP7)
  - Demonstration of 500kW demo CLC fixed bed, syngas from gasification of coal
- SUCCESS up-scaling of CMT material production (EU FP7)
- Mineral Scout (FENCO-Net (M-ERA(RCN)))
- Negative CO2 (Bio-CLC) (Nordic Energy Research)
- ITS-CLC (IFP,TNO,SINTEF co-operation)
- COMPOSITE fixed bed (RCN CLIMIT funded)
- BIGCCS / BIGCLC. (RCN CLIMIT funded)
  - SINTEF rotating fixed bed
  - SINTEF double circulating fluidized bed

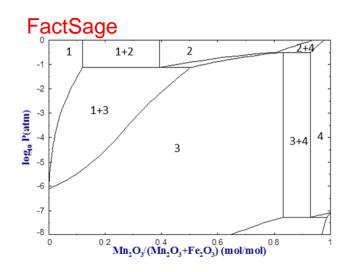




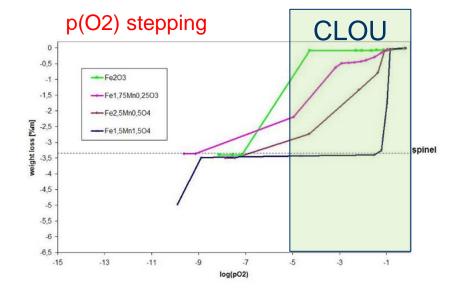


# **COAL: Fe-Mn SYSTEM (from ÉCLAIR)**

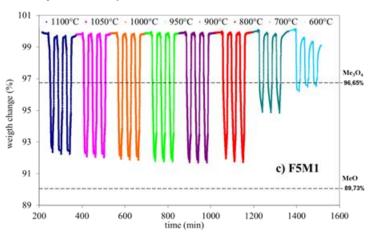
## **Tools for selecting material:**



1: Hematite
2: Bixbyite
3: Spinel
4: Hausmannite
5: Manganese
6: Halite

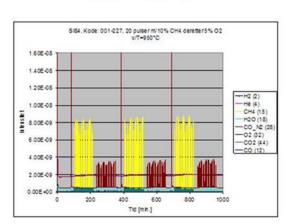


### Cyclic experiments

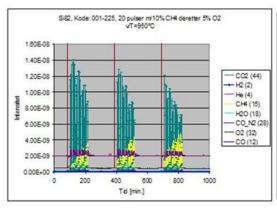


# Fixed bed -TPX

 $Fe_{1.75}Mn_{0.25}O_3$ 



## $Fe_{1.5}Mn_{1.5}O_4$









# 230478 - "Minerals for Sustainable COst and energy efficient chemical looping combUstion Technology"

## Scope

- Improve the novel Chemical Looping Combustion (CLC) technology for power production with CO2 capture, by finding better oxygen carrier materials with high and fast oxygen release giving full combustion. (Avoiding penalty from extra oxygen elsewise needed from air separation unit (ASU))
- Find and get hold of 10 new minerals from mining industry, including some from industrial waste.
- Test and evaluate them against criteria's needed for coal based Chemical Looping Combustion.
- Down select 2-3 for further testing under rig operation



NTUA – National Technical University of Athens







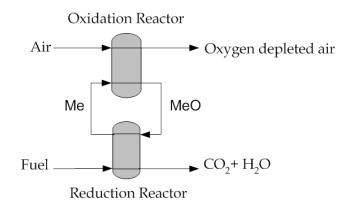




#### MINERAL SCOUT



#### **Chemical Looping Combustion**

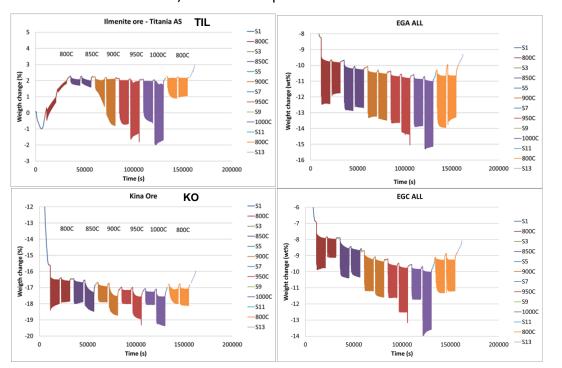




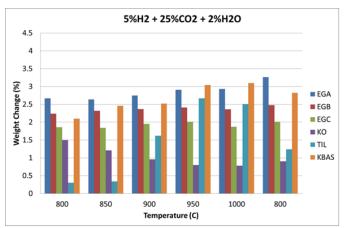


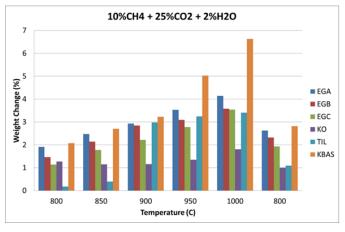
# Task conducted at SINTEF the first period

- Pre-testing by TG
  - 800, 850, 900, 950, 1000, 800C
  - At each temperature
    - 20 cycles between air and 5%H2, 25% CO2, 2%H2O step 2min
    - 20 cycles between air and 10%CH4, 25% CO2, 2%H2O step 2 min



#### Oxygen carrier capacity and reactivity







# **Summary pre-test**

## Elwaleed samples:

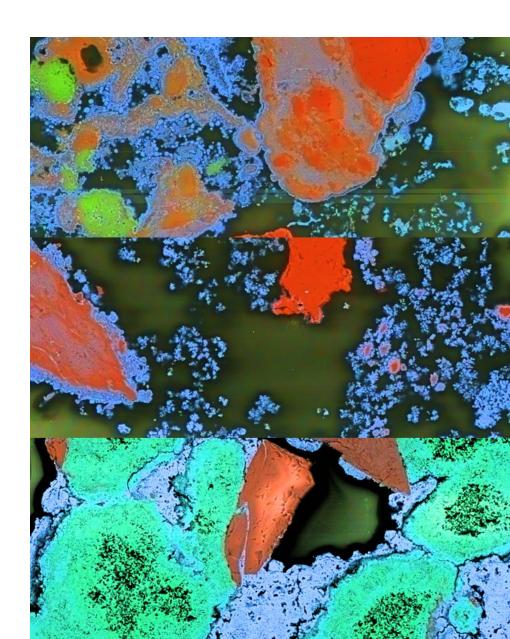
- Inhomogeneous!
- Fe and Mn not much intermixed, even after testing.
- Works directly without pretreatment.
- High reactivity and oxygen carrier capacity

#### Krivbas

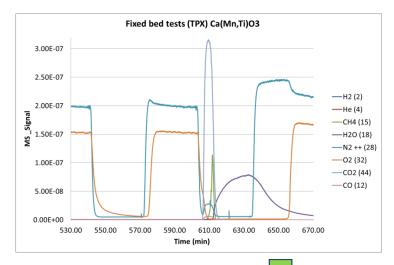
- Nearly pure Fe-oxide .
- Gets highly porous upon testing.
- Strength might be an issue.
- Very high capacity and good reactivity

#### Titania ilmenite

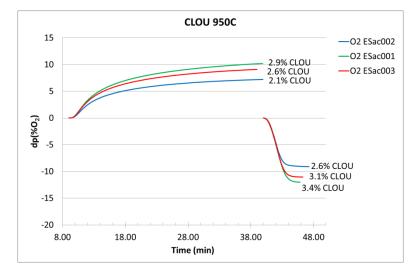
- Nearly pure ilmenite.
- Fe diffusion to the surface of the grains upon testing.
- Need activation.
- Capacity medium

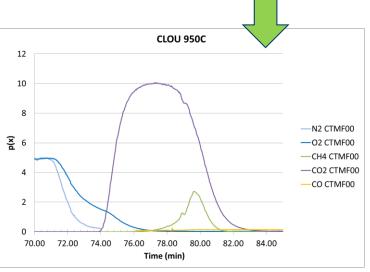


# NG: Ca(Mn,Ti,Fe)O3 system (from BIGCCS/SUCCESS) Fixed bed (TPX) test for CLOU and reactivity evaluation

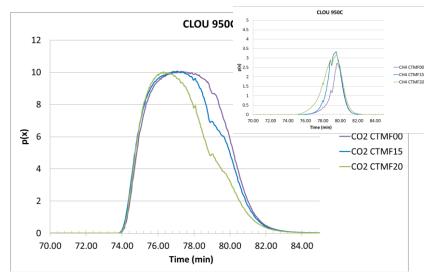
















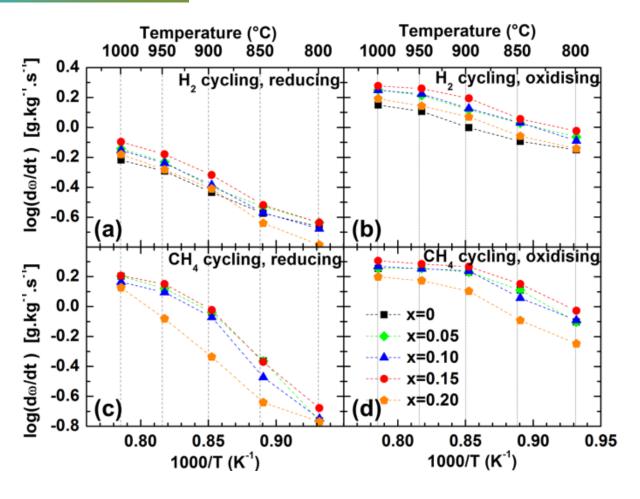










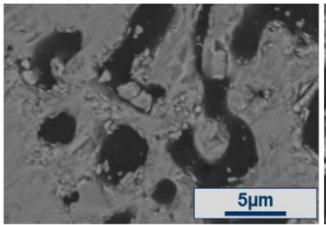






CMT - fresh:

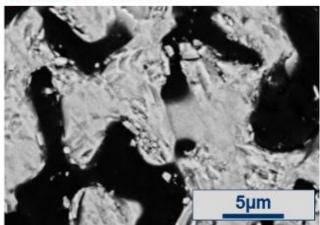
h: CMT – after 240 cycles:

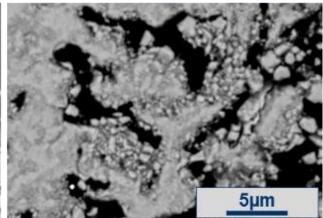


\_\_<u>5μm</u>\_\_\_\_

CMTF15 - fresh:

CMTF15 - after 240 cycles:

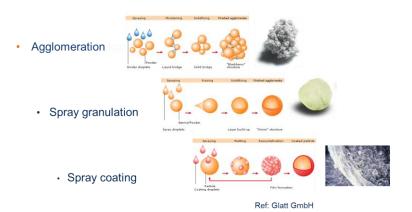




# Powder morphology for up-scaling to industrial scale

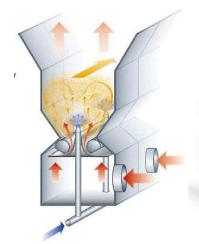
#### Glatt ProCell unit installed at SINTEF in 2012

Agglomeration, spray granulation, spray coating











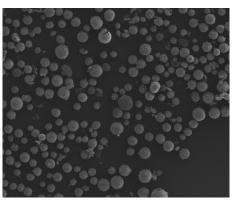


# Buchi B-290 Spray Dryer

# ProCept Spray Dryer









# GEA Mobile Minor Spray Dryer

# Naberthem furnace







# One promising evaluated route

- CaMn<sub>1-x-y</sub>Ti<sub>x</sub>Fe<sub>y</sub>O<sub>3</sub> (CMTF) using cheap, non toxic, raw materials
  - CaCO<sub>3</sub>, Colormax (Mn<sub>3</sub>O<sub>4</sub>), TiO<sub>2</sub>/FeTiO<sub>3</sub>
- Production method not selected (Spray drying or spray granulation)
- Must be produced in tons with necessary properties
  - 100-300 µm, spherical, porous, sufficient mechanical strength

## **Crushing and screening**



## **Granulation / coating**



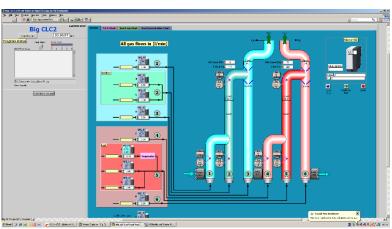
## **Spray drying**





# 3kW rig developed for hot attrition testing of materials.







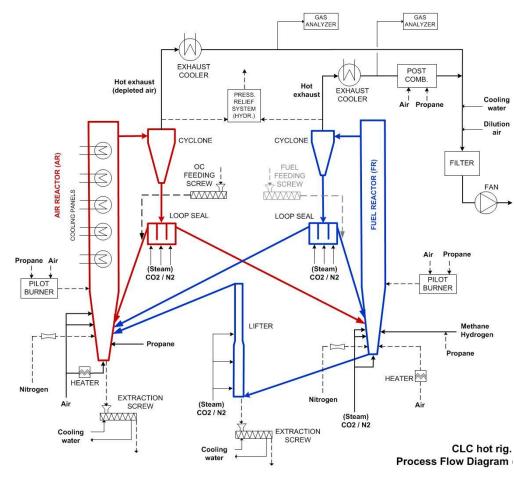




# 150 kW CLC rig at SINTEF

- Double loop CFB reactor system
- Reactor height: 6 m
- Reactor diameters: AR 230 mm, FR 154 mm







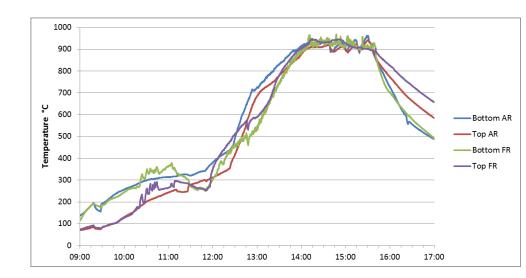
# 150 kW CLC rig at SINTEF

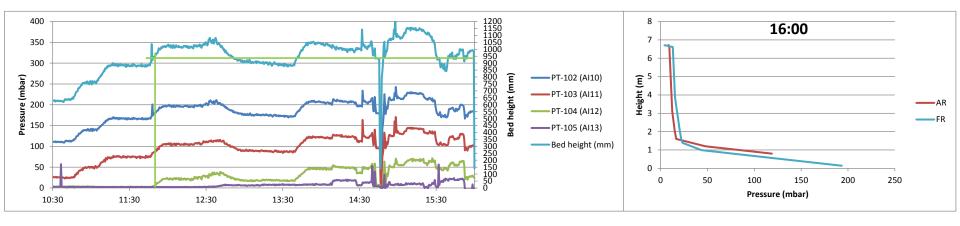
### Heat-up of reactor system

 Required temperatures for CLC is being reached in both reactors (>900°C)

## Next step:

- Transfer to CLC mode using
  - ilmenite and hydrogen
  - or CMTF and NG











# Negative CO<sub>2</sub>

Negative CO<sub>2</sub> Emissions with Chemical-Looping Combustion of Biomass

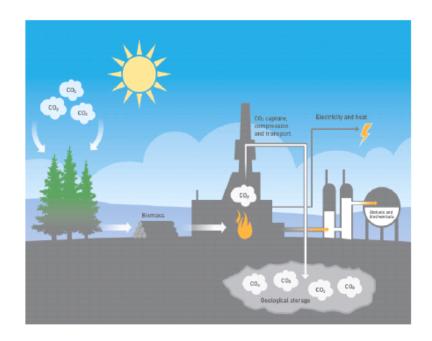
- Negative CO<sub>2</sub> is a multi-partner and crossdisciplinary project funded by Nordic Energy Research that runs from November 2015 to October 2019.
- The research topic is CO2 capture during biomass combustion by CLC.
- Aiming at demo
- Budget 27 Mill NOK

#### The project partners are:

- Chalmers University of Technology
- The Bellona Foundation
- Sibelco Nordic AB
- SINTEF Energy Research
- SINTEF Materials and Chemistry
- VTT Technical Research Centre of Finland Ltd
- Åbo Akademi University

#### Advisory board associated with the project:

- Alstom Power AB
- Andritz Oy
- AKZO Nobel
- Elkem AS
- E.ON Sverige AB
- Fortum Oyj
- · Foster Wheeler Energia
- · Göteborgs Energi
- Titania A/S
- Arbaflame



Schematic description of BECCS (illustration by Doghouse.no/SINTEF).



# People working with CLC

Strength introduced by different groups in SINTEF and NTNU<sup>(\*)</sup> working together

- SINTEF MC Oslo (Material selection and characterization)
  - Yngve Larring, Mehdi Pishahang, Bjørnar Arstad, Richard Blom.
- SINTEF MC Trondheim (Material Fabrication)
  - Tommy Mokkelbost, Christian Schøning, Ingeborg Kaus
  - Shariar Amini, Schalk Cloete, John Morud, Abdelghafour Zaabout
- SINTEF ER Trondheim (Rig development and testing 150kW unit)
  - Nils Haugen, Inge Saanum, Øyvind Langørgen, Jørn Bakken
- NTNU Trondheim (Material optimization)
  - Kjell Wiik, Vincent Thoréton
  - (\*) NTNU: Norwegian University of Science and Technology

