



Catalytic Process Intensification Routes for the Production of Synthetic Fuels and Energy Vectors

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Inès Esma Achouri, Eng., MSc., Ph.D.

CRC - Process intensification for advanced catalysts and sustainable energy

USherbrooke.ca/Recherche

CORFU 2022



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Université de
Sherbrooke

Overview

- ▶ Université de Sherbrooke
- ▶ Research group
- ▶ Process intensification
- ▶ Nonthermal plasma assisted reactions
- ▶ New approaches for CO₂ Hydrogenation
- ▶ Clean biofuels production from contaminated biomass
- ▶ Conclusion



UDS in numbers



3 CAMPUS: THE
MAIN CAMPUS IS
SITUATED AT
SHERBROOKE



8 FACULTIES



HOST TO MORE
THAN 33,000
STUDENTS



22 RESEARCH
CENTRES



78 RESEARCH
CHAIRS

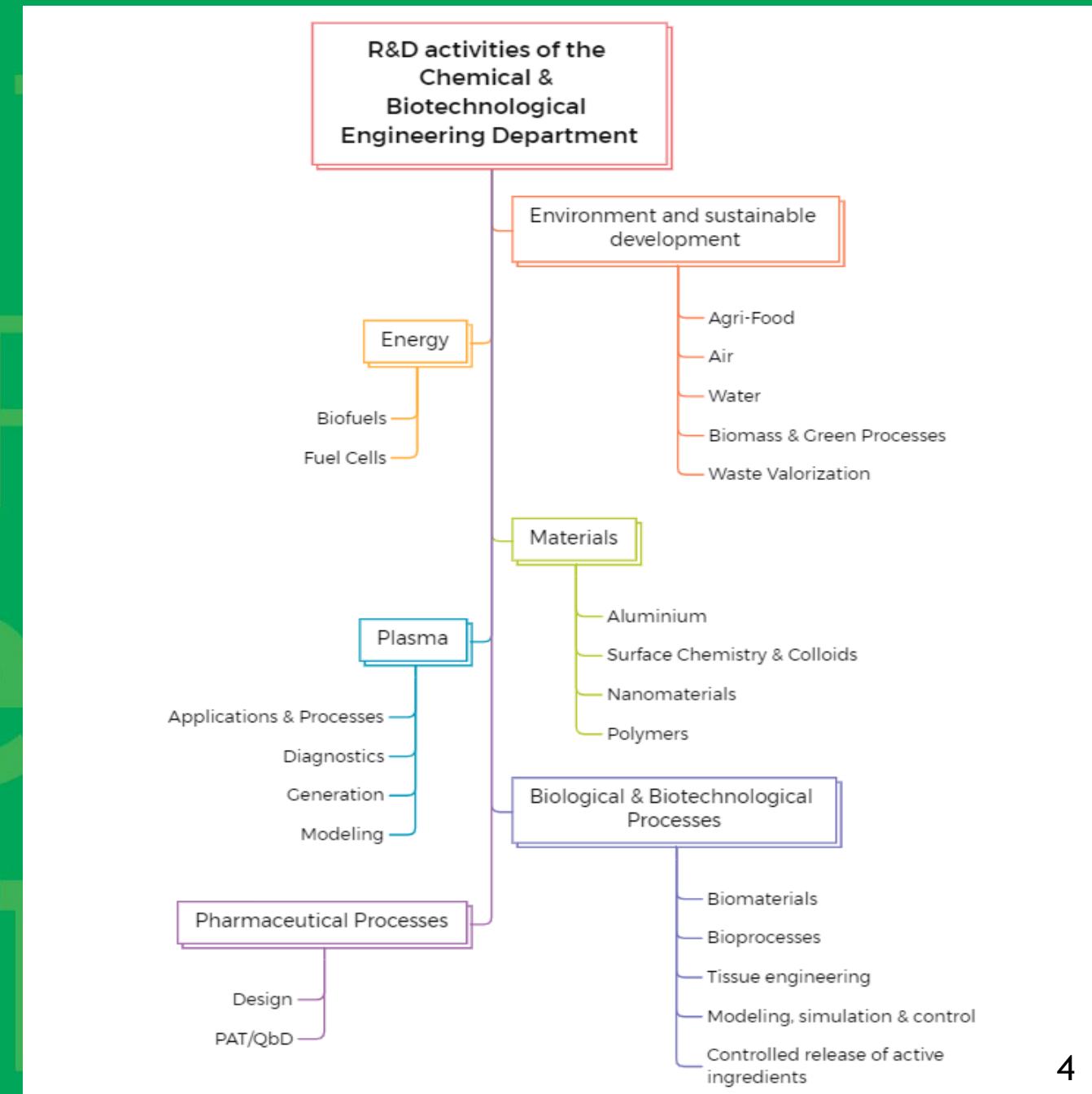


6 RESEARCH
INSTITUTES



Université de Sherbrooke

– R&D Chem. Eng.

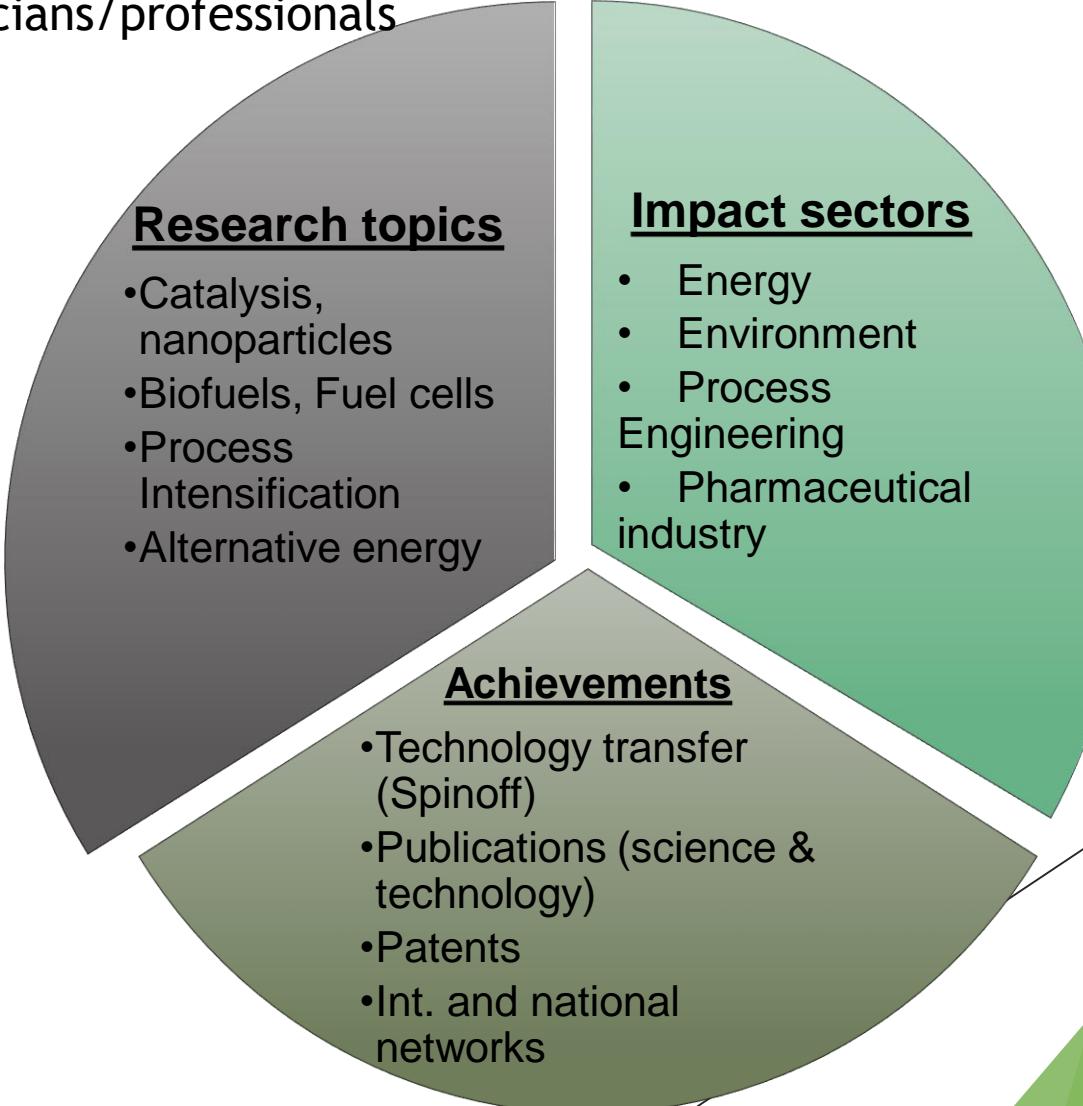




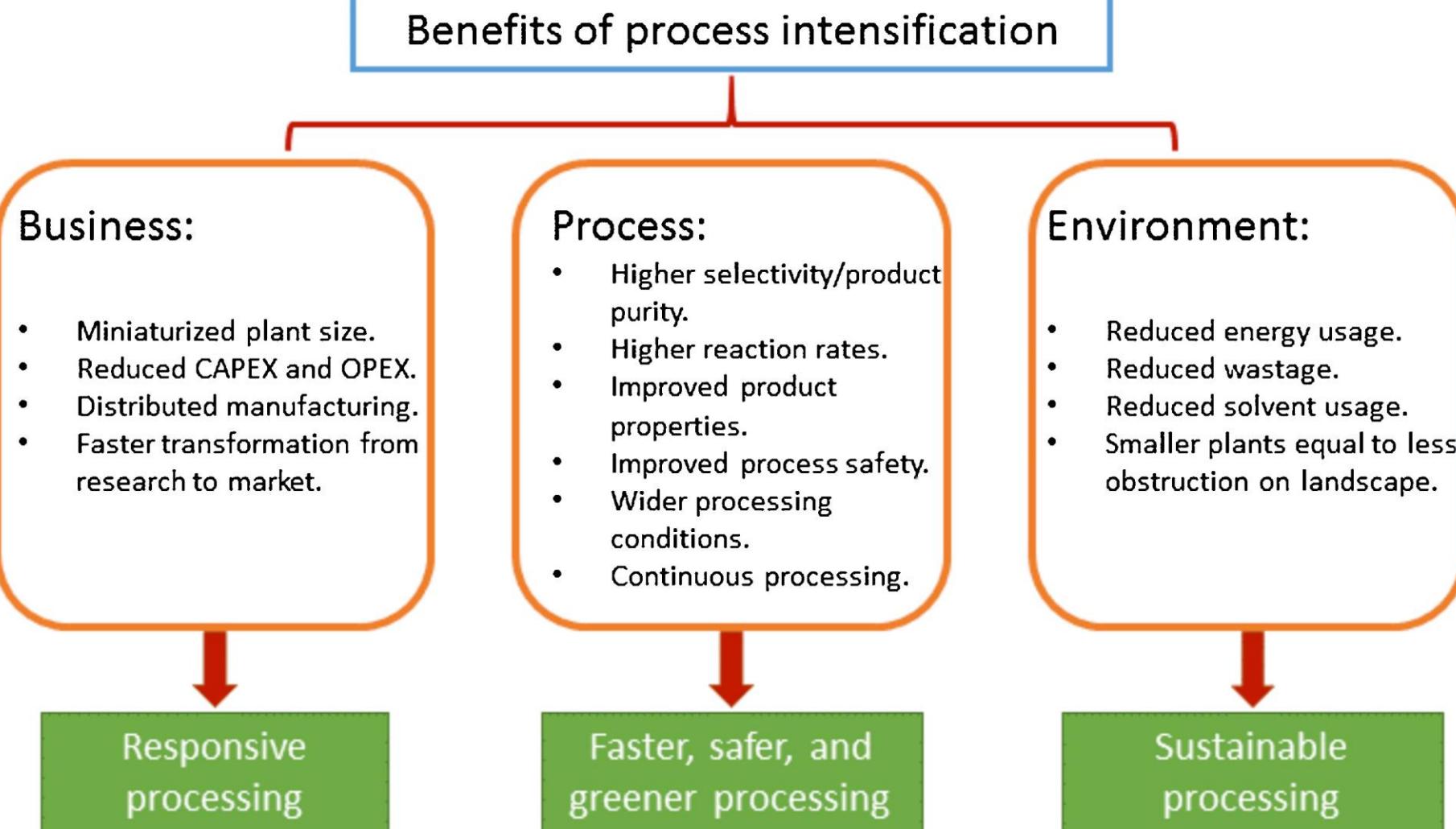
Université de Sherbrooke

Group of Research on technologies and processes (GRTP)

- 4 professors
- 20 graduate students
- 3 technicians/professionals

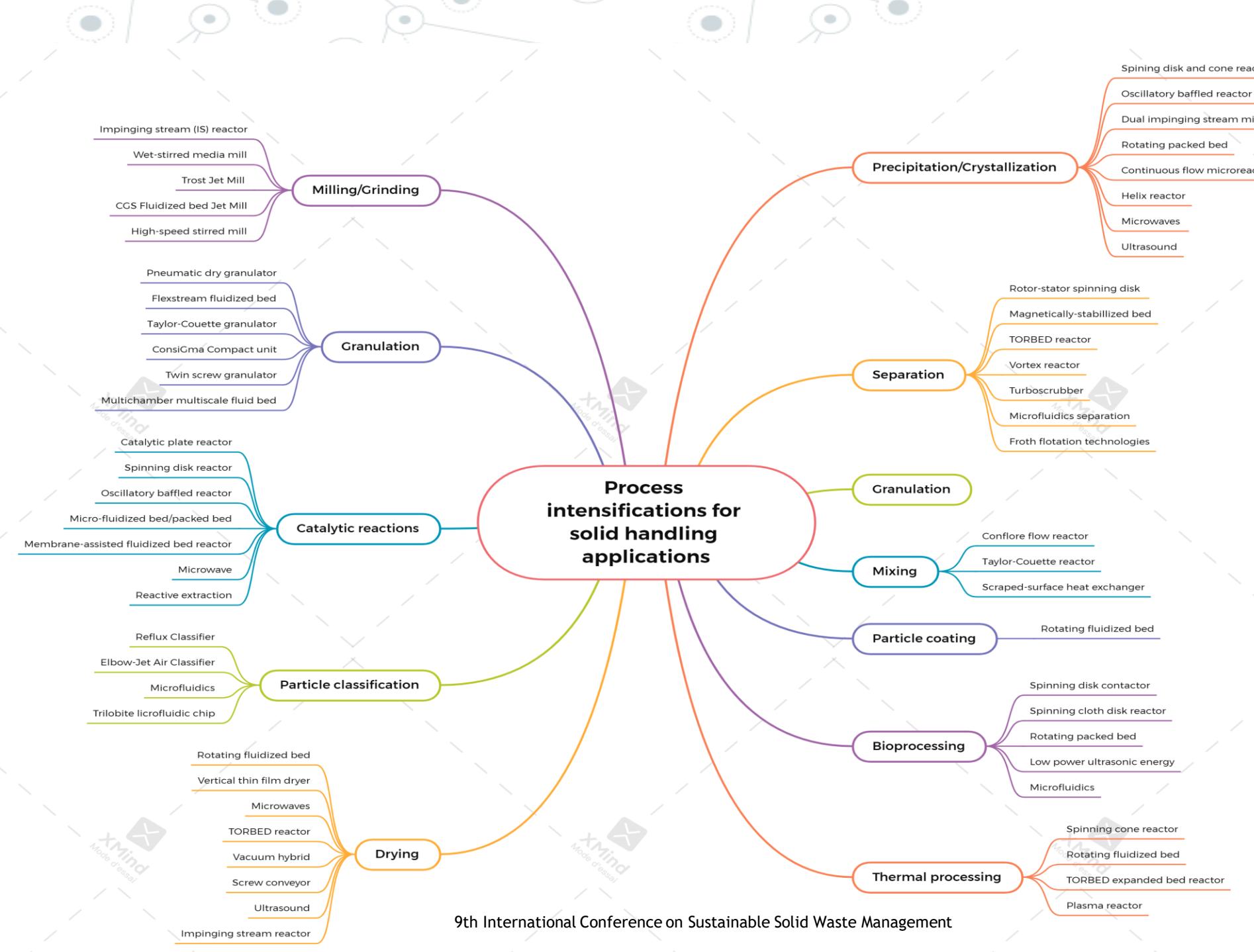


Process intensification



PI for solid handling

7



Targeted projects



CFI / FCI

Nonthermal plasma
assissted reactions



New approaches for CO₂
Hydrogenation



Clean biofuels production from
contaminated biomass



New Frontiers in Research Fund
Fonds Nouvelles frontières en recherche



CFI / FCI

Non-thermal plasma assisted reactions

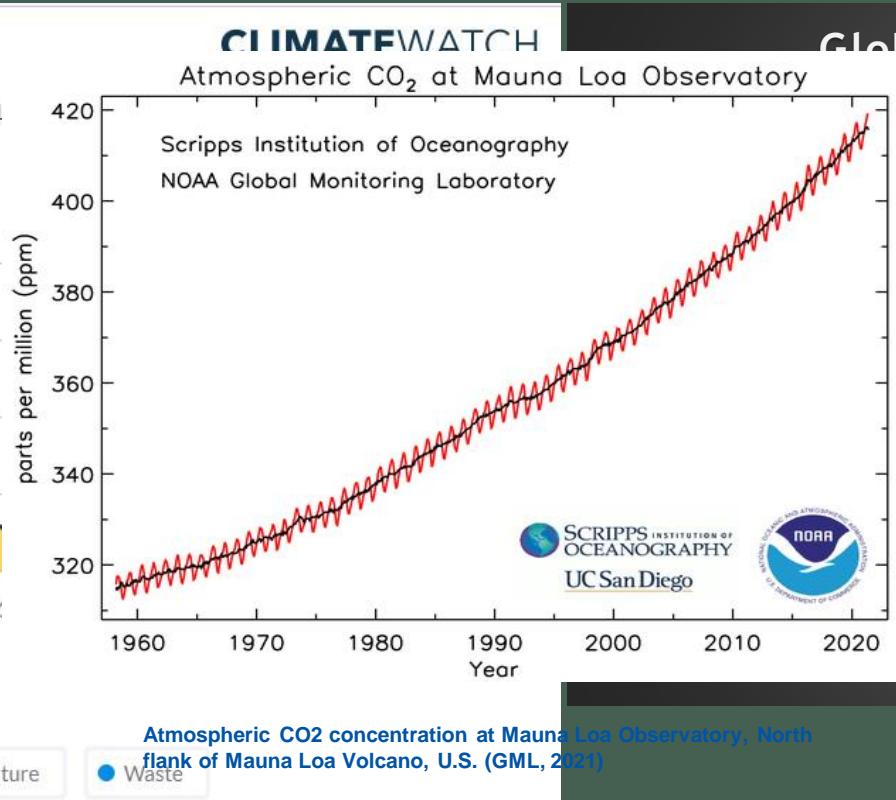
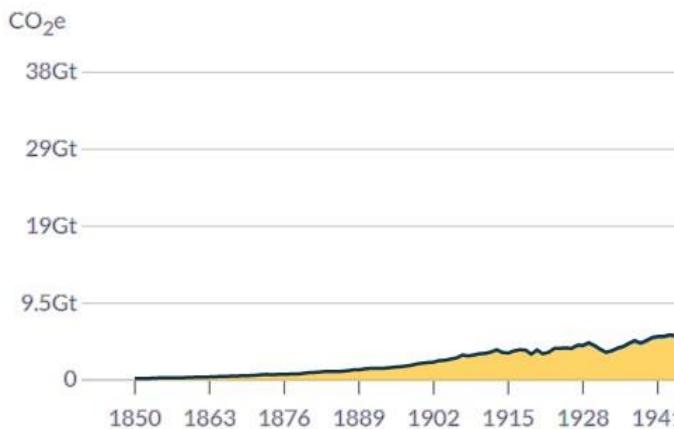
Project 1: Nonthermal plasma catalysis Methane dry reforming



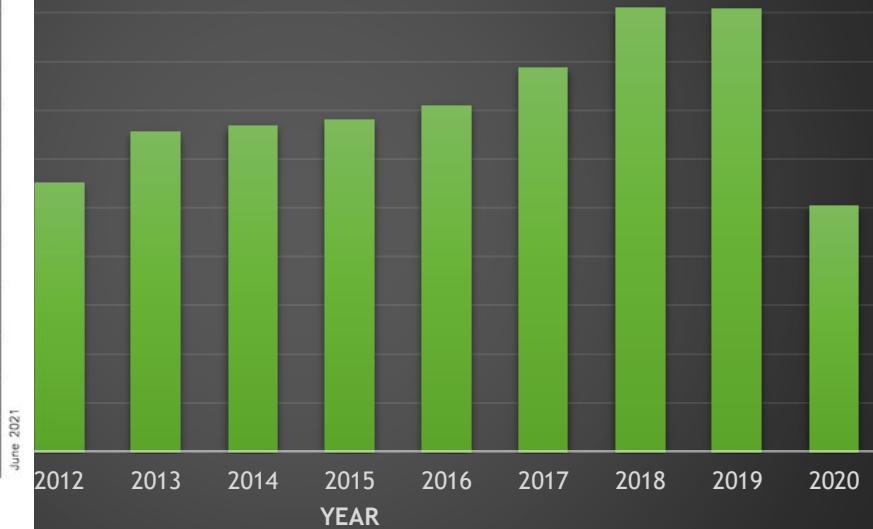
Global GHG emissions

Historical GHG emissions

Data source: PIK; Location: World; Sectors/Subsectors: Total excluding LULUCF Sectors.



Global Carbon Dioxide Emissions



<https://www.wri.org/insights/history-carbon-dioxide-emissions>, dated access: 09/05/2022

- Due to the Covid-19 pandemic, we experienced the largest falls in both energy demand and carbon emissions since World War II. However, The carbon budget is running out: CO₂ emissions have increased in every year since the Paris COP in 2015, except in 2020. Delaying decisive action to reduce emissions sustainably could lead to significant economic and social costs. (BP r., 2022)

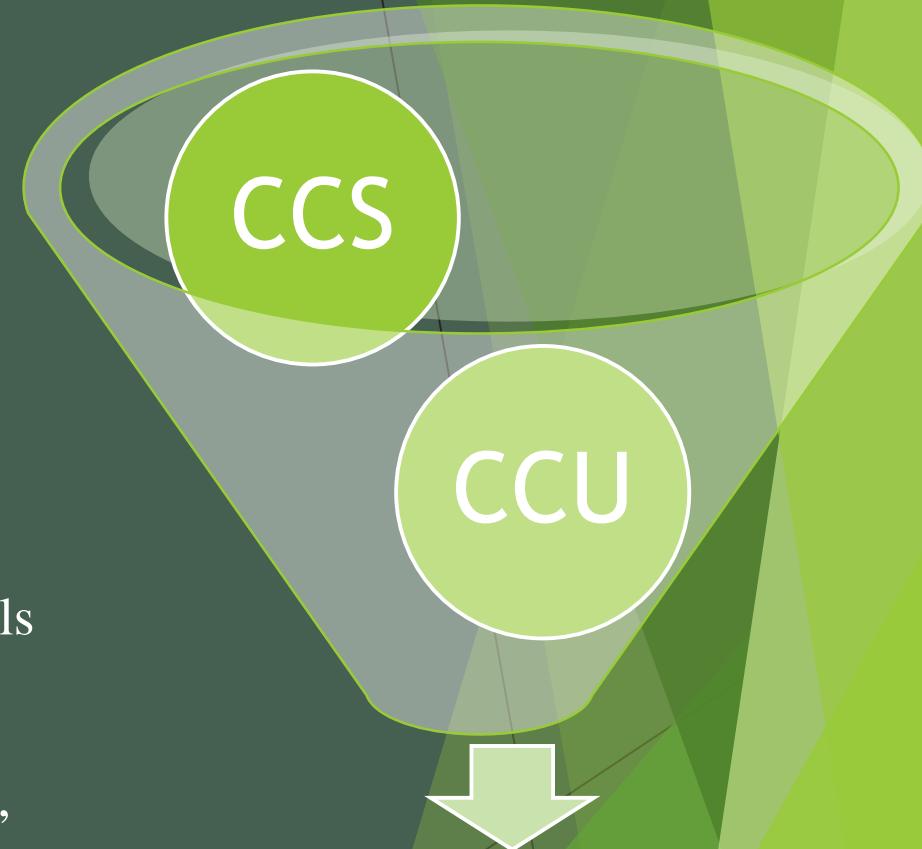
Routes to CO₂ treatment

➤ Carbon Capture & Storage (CCS):

- reported as an attractive solution for the temporary storage of large volumes of CO₂ (Gao, J. Y., 2011)
- has the potential risk of leakage (Li W. W., 2018)

➤ Carbon Capture & Utilization (CCU):

- consider CO₂ as a source to produce value-added chemicals and fuels (de La Fuente, 2016)
- there are several routes to utilize CO₂ such as beverage carbonation, food packaging, water desalination, EOR (70-80Mton of CO₂ is consumed per year), biomass gasification, construction and building materials, and renewable energy systems (for instance, CO₂ based geothermal energy systems)



Chemical treatment of CO₂

CONVENTIONAL METHANE DRY REFORMING REACTIONS:



247 kJ mol⁻¹

(1)

Side reactions:



75 kJ mol⁻¹

(2)



-171 kJ mol⁻¹

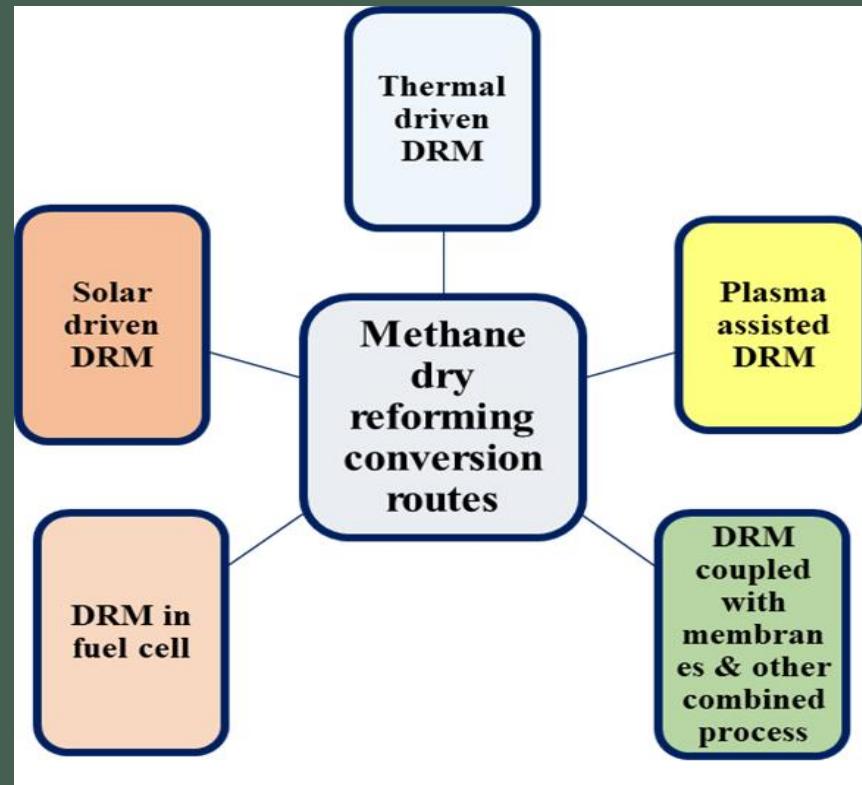
(3)

Problems:

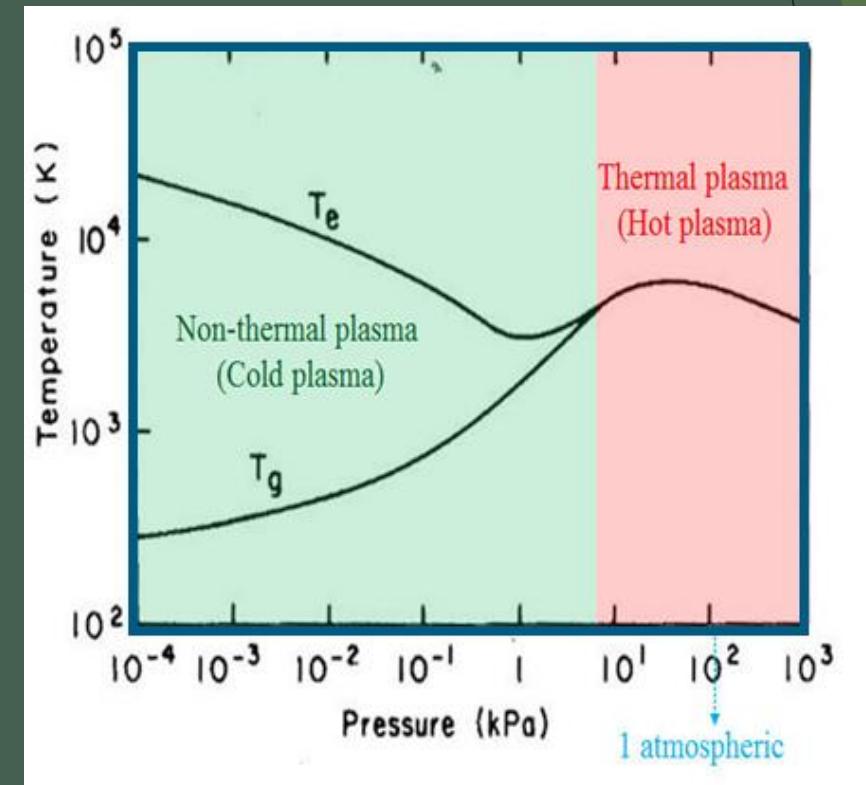
- 1) Intensive carbon deposition (Large Ni size)
- 2) Active metal sintering (weak interaction)
- 3) High temperature process

DRM conversion routes

plasma deviation from the kinetic equilibrium ($T_e \gg T_h$) where the electron temperature is sufficiently high compared to the heavy species with a high degree of non-thermodynamic equilibrium is classified as non-thermal plasma

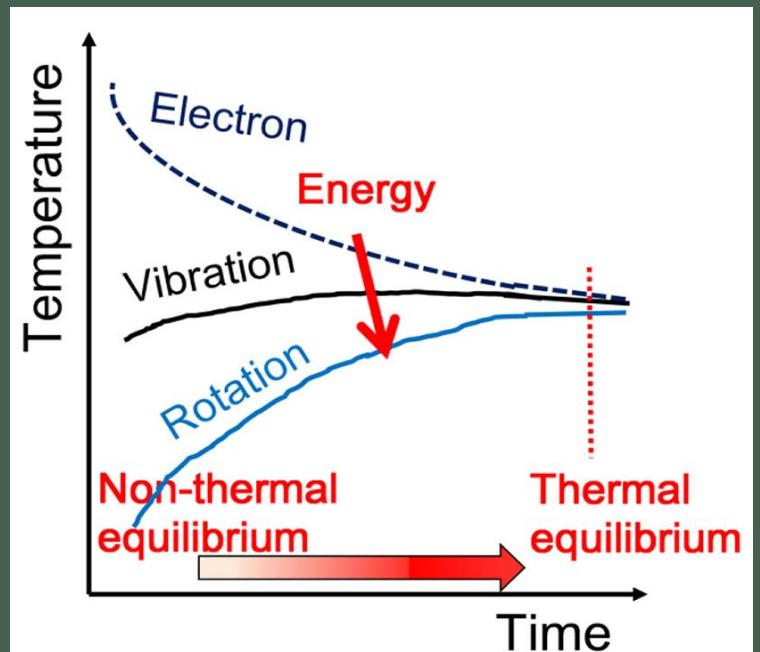


Current Dry reforming routes in cold plasma



Electron impact energy transfer in cold plasma

$$T_e \gg T_v > T_r \approx T_i \approx T_o$$



T_v is the temperature of vibrationally excited molecules, T_r is the rotational degree of freedom, T_i is ion temperature, and T_o describes the gas temperature

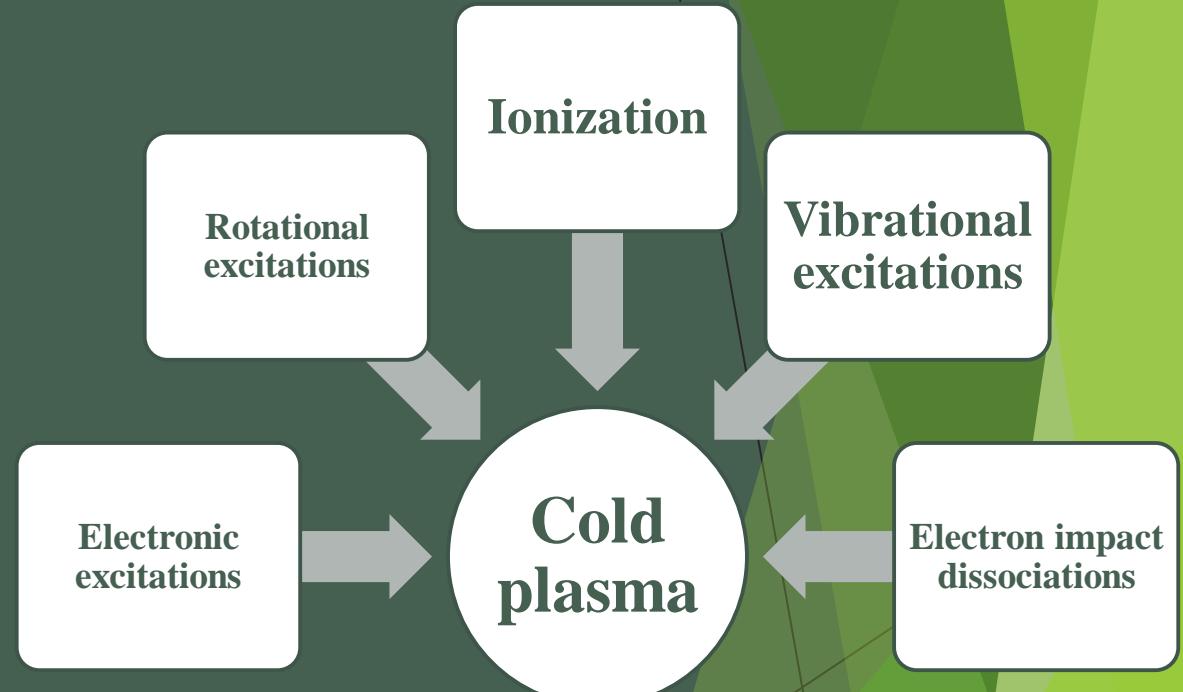
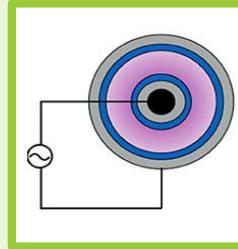
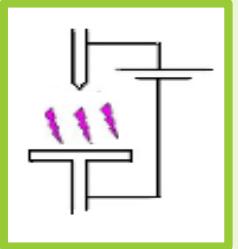
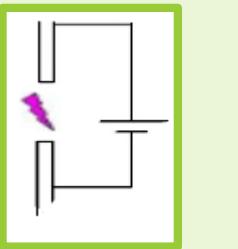
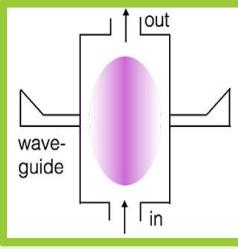
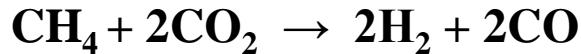


Table 1. Use of different cold plasma discharge in DRM

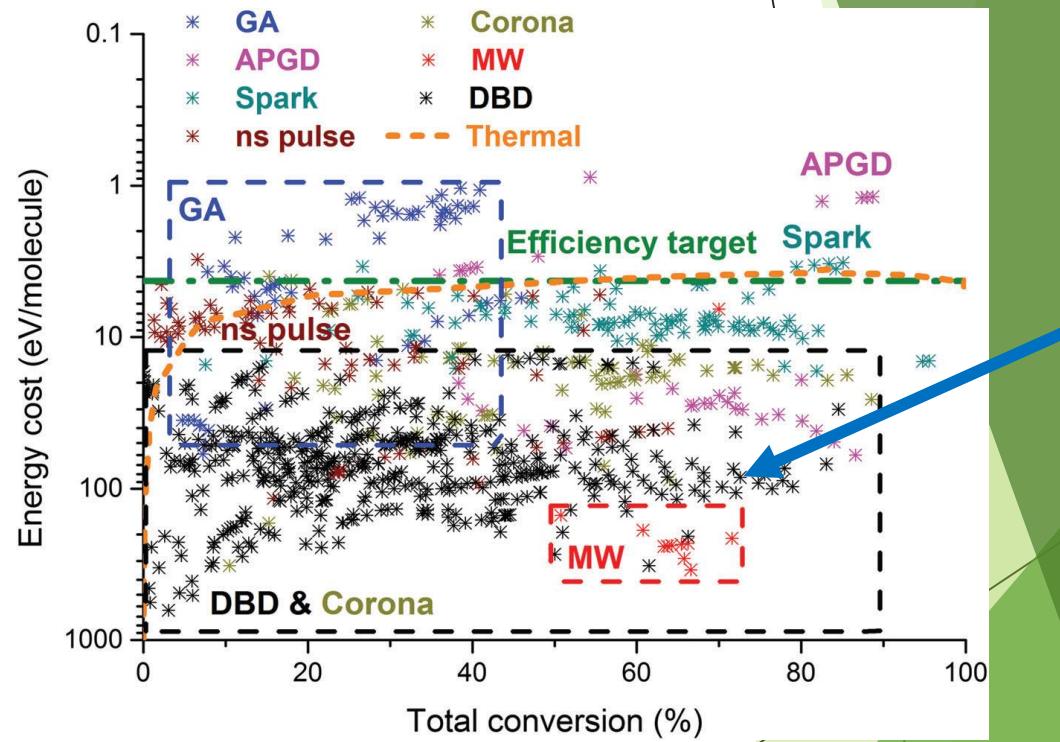
Reactor type	DBD	CD	SD	MW
Scheme				
Electron energy [eV]	1 - 30	≈ 5	-	1-2
Electron density [cm^{-3}]	$10^{12} - 10^{15}$	$10^9 - 10^{13}$	$10^{14} - 10^{15}$	10^{13}
Current [A]	1 - 50	$\approx 10-5$	20 - 30	-
Gas Temperature [K]	300 - 500	≈ 400	400 - 1000	100-2000
Break down voltage [kV]	5 - 25	10 - 50	5 - 15	-

Evaluation of cold plasma discharge in DRM



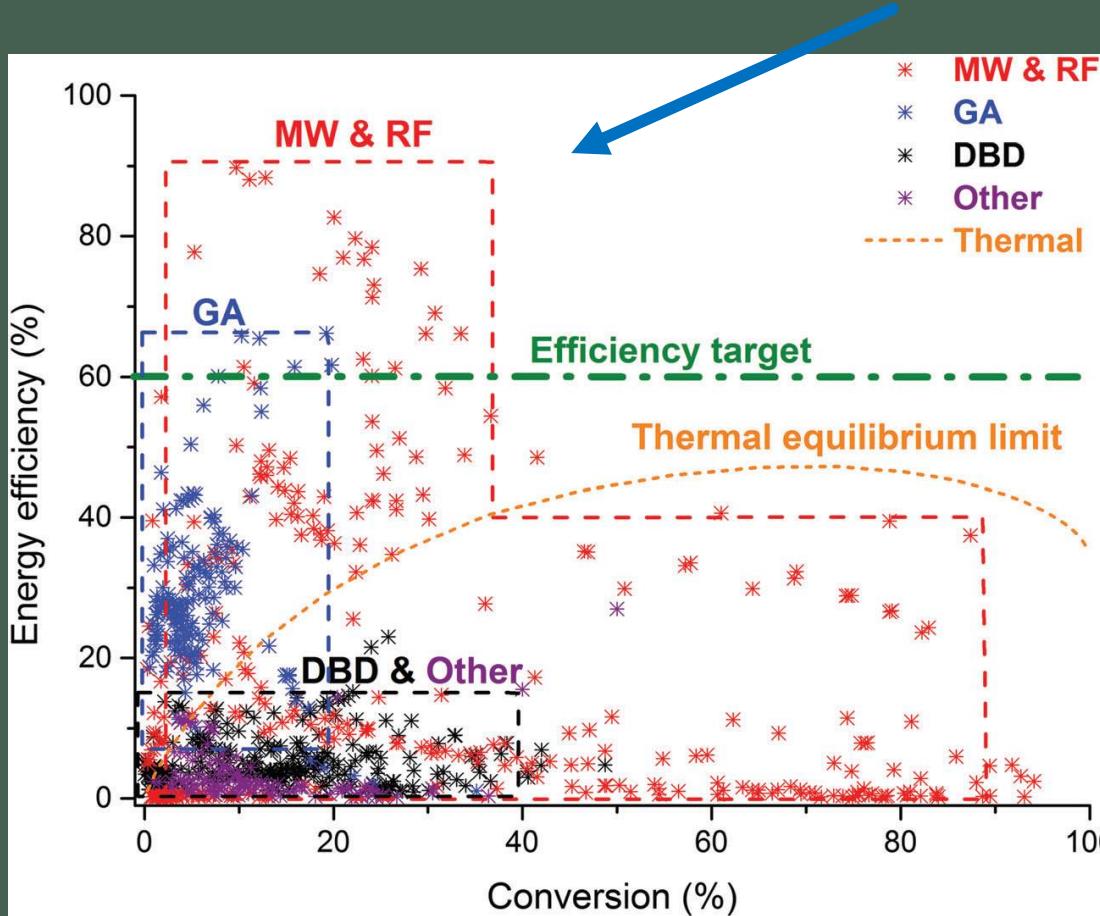
247 kJ mol⁻¹ ~ 2.57 eV

It is worth mentioning that for a 100% energy-efficient dry reforming reaction, considering all the products, including gas and liquids, the energy cost per molecule conversion is 2.57 eV, equal to the reaction's standard enthalpy (247.3 kJ/mol)



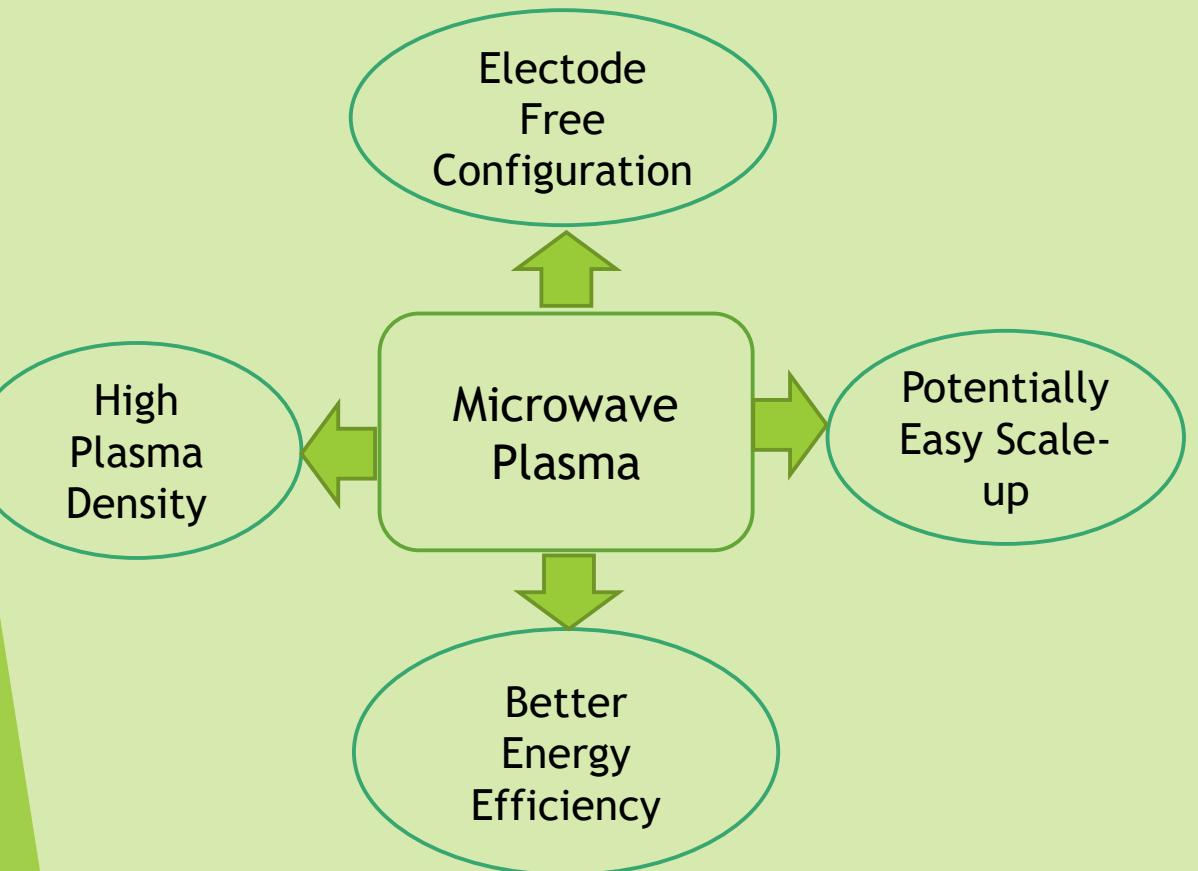
Performance/collection of data of energy cost vs total conversion under different plasma discharge in DRM

Evaluation of cold plasma discharge in CO₂

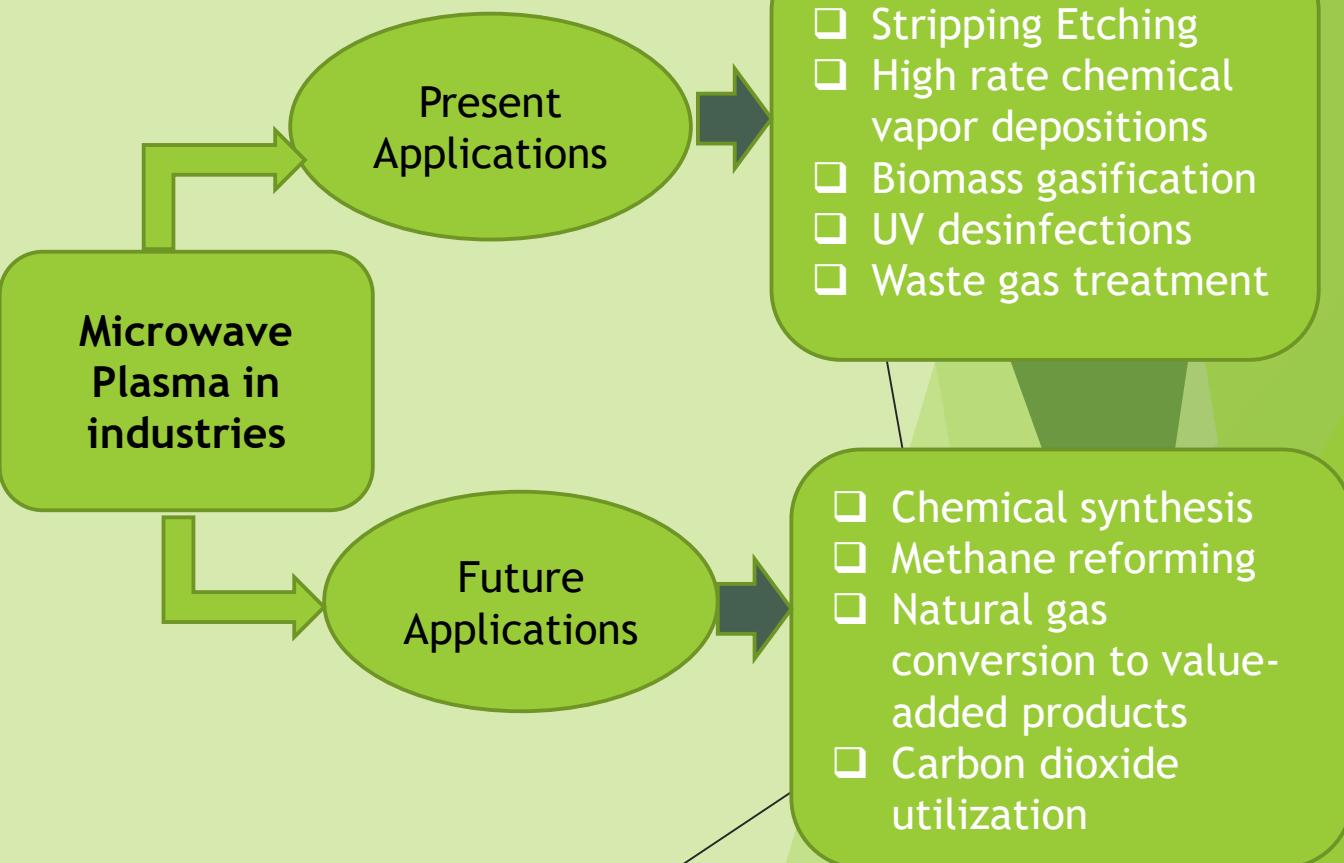


Performance/collection of data of energy efficiency vs conversion under different plasma discharge in CO₂ splitting

Advantages of MW plasma



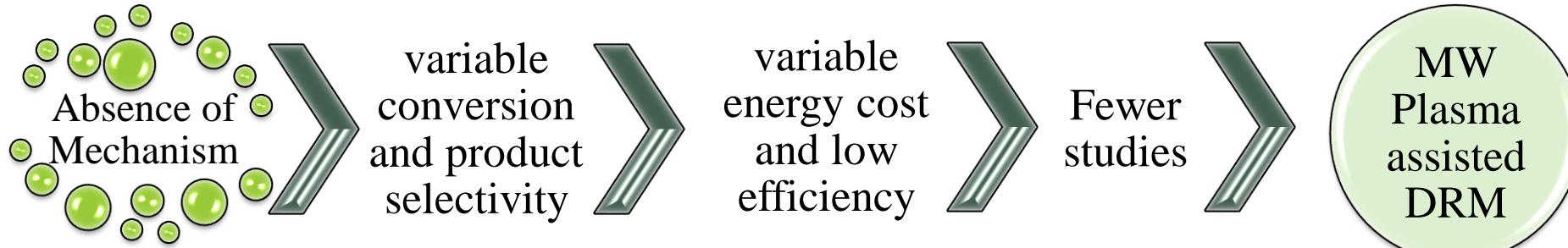
Current and future applications of MW plasma



Microwave plasma dry reforming

Plasma	Power (W)	Frequency	Pressure	Year	CH ₄ /CO ₂	Conversion		Selectivity (%)	Ref.
						CO ₂	CH ₄		
MW	6 kW	2.45 GHz	Atmospheric pressure	2017	1:1	68.4 %	96.8 %	H ₂ Production rate g/H = 240 And H ₂ :CO = 0.9:1.1	[20]
MW	6.5 kW	915 MHz	Atmospheric pressure	2019	2:3	-	86.5 %	H ₂ (73.3)	[21]
MW	2kW	2.45 GHz	Atmospheric pressure	2021	1:1	90.7 %	96.4 %	H ₂ (97.3) CO(85.7)	[22]
MW	700	-	Atmospheric pressure	2019	1:2	19 %	83 %	H ₂ (99) CO (19)	[23]
MW	700	-	Atmospheric pressure	2018	1:2	44.40 %	84.91%	H ₂ (51.31) CO (61.17)	[24]
MW	1.0 kW	2.45 GHz	atmospheric pressure	2010	-	-	91.6%	H ₂ (92.7)	[25]

Challenges and Problems in MW assisted plasma DRM



The gaps and areas to be improved in MW plasma assisted DRM

Reactor configurations with *in situ* Characterization

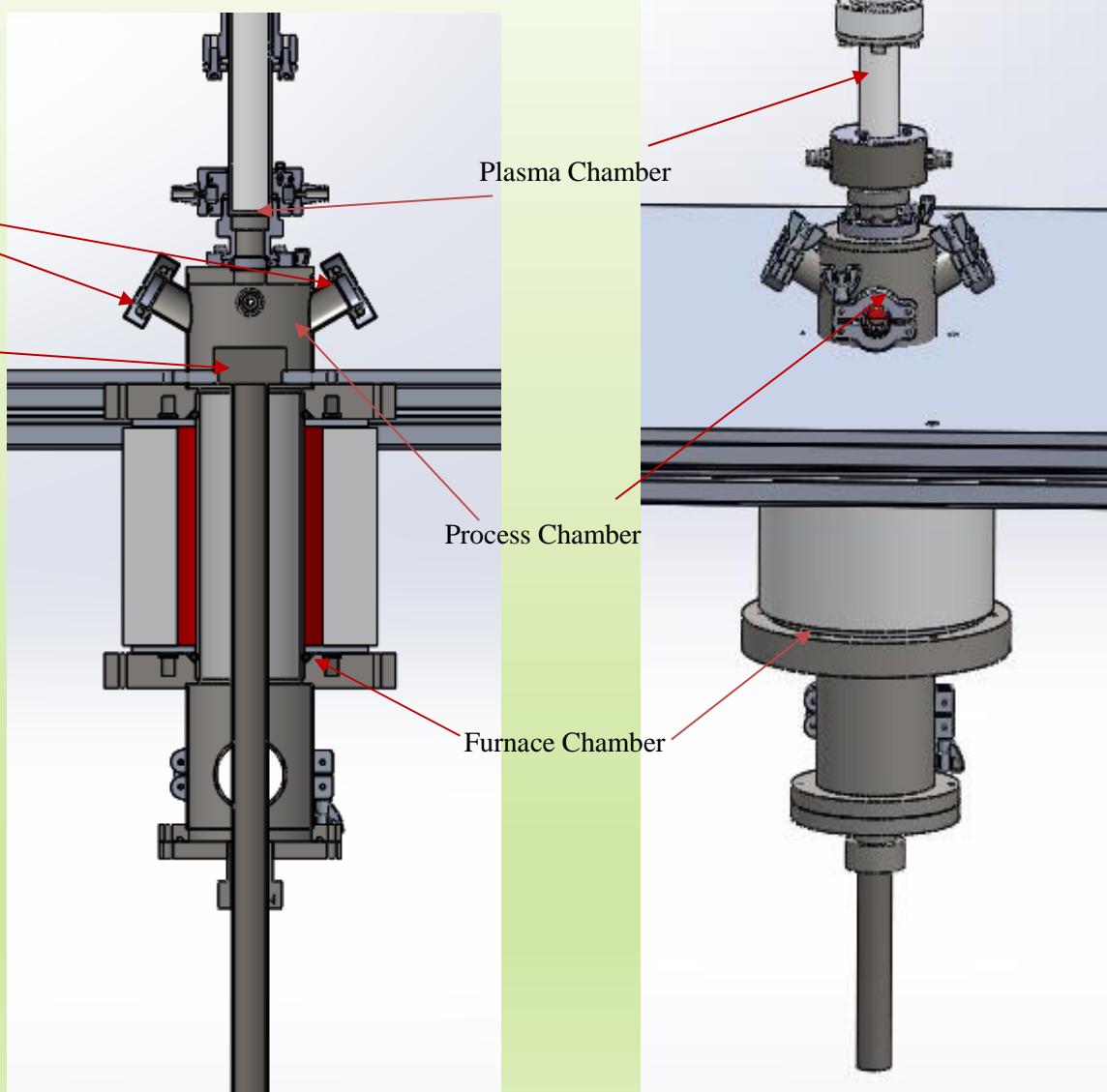
Diagnostic Ports

Catalyst Stage

Plasma Chamber

Process Chamber

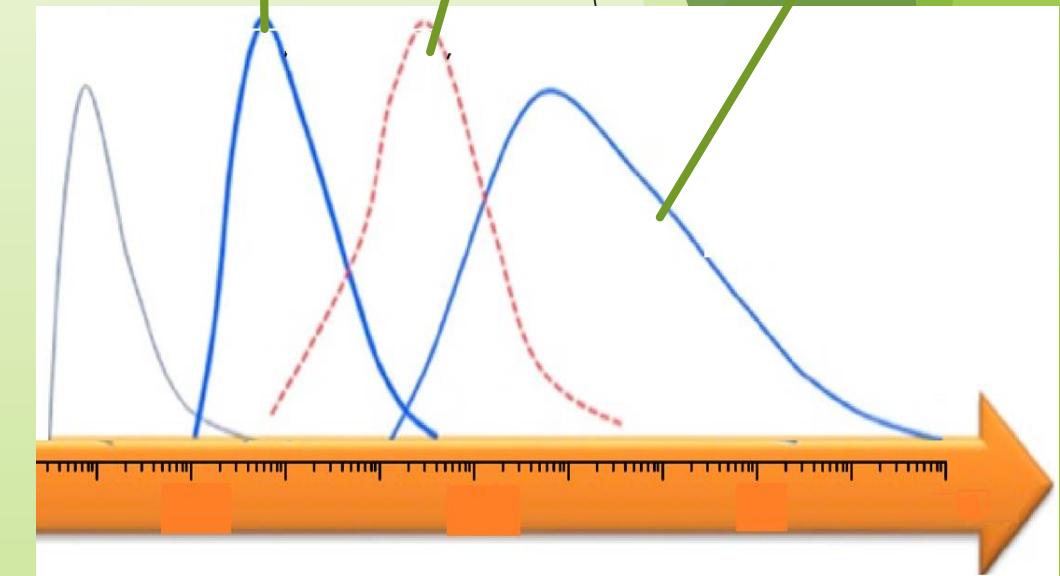
Furnace Chamber



Functional Group

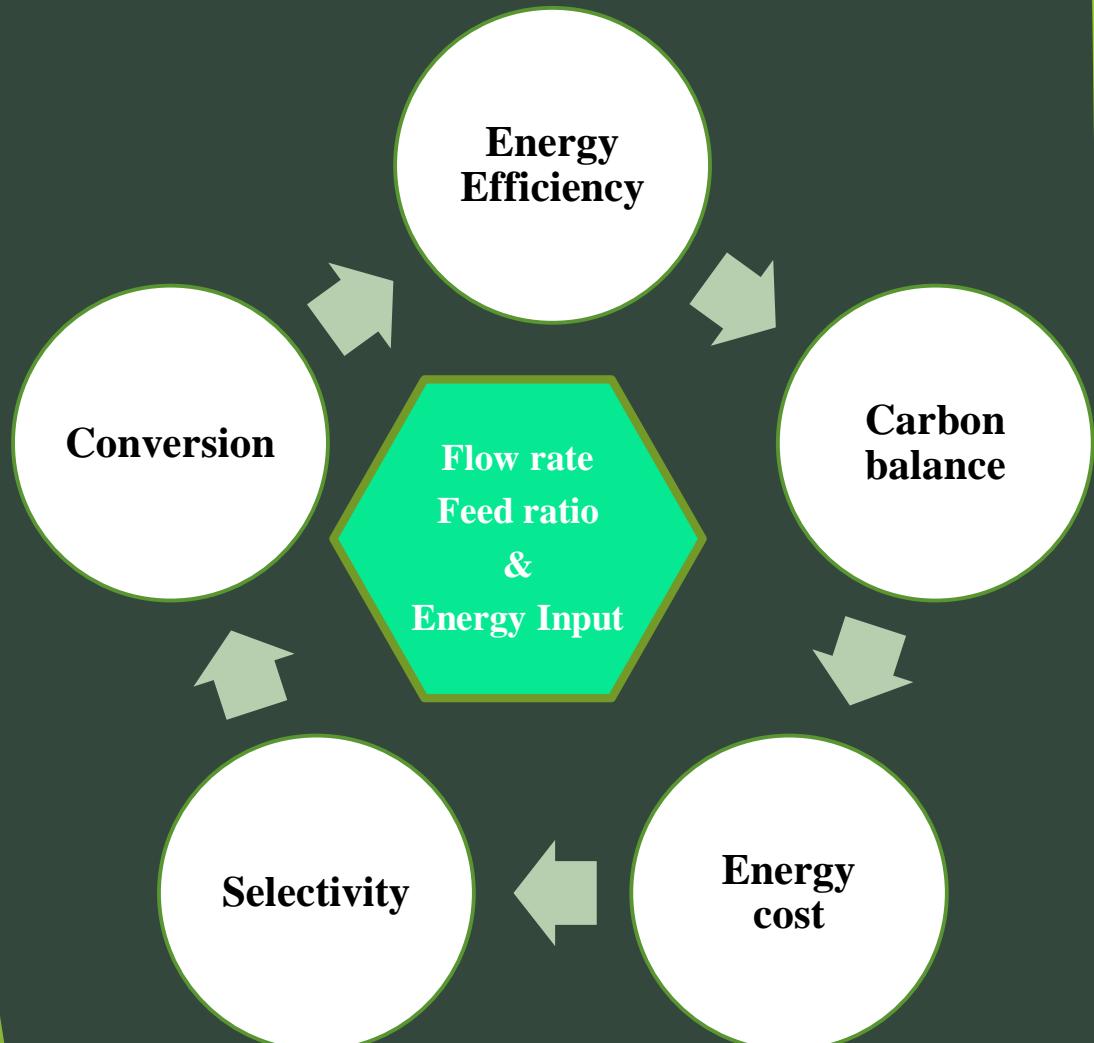
Mass:Q ratio
MW info

Elemental composition



Elements needed to advance the field of plasma catalysis.

Correlations Process Parameters



Effect of flow rate , feed ratio and energy input on the reaction metrics

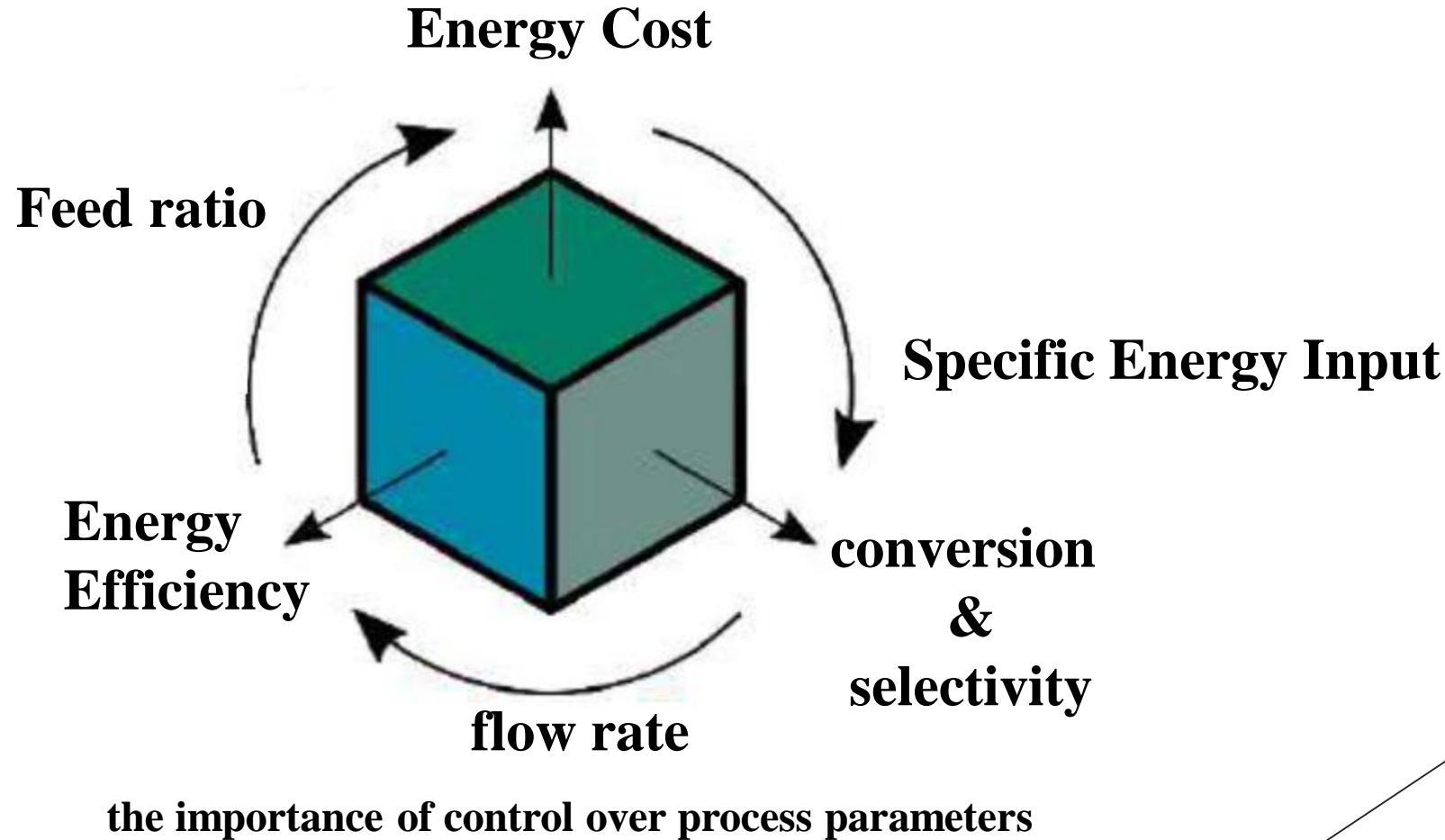
Specific energy input (SEI)
the ratio of plasma power to the gas flow rate

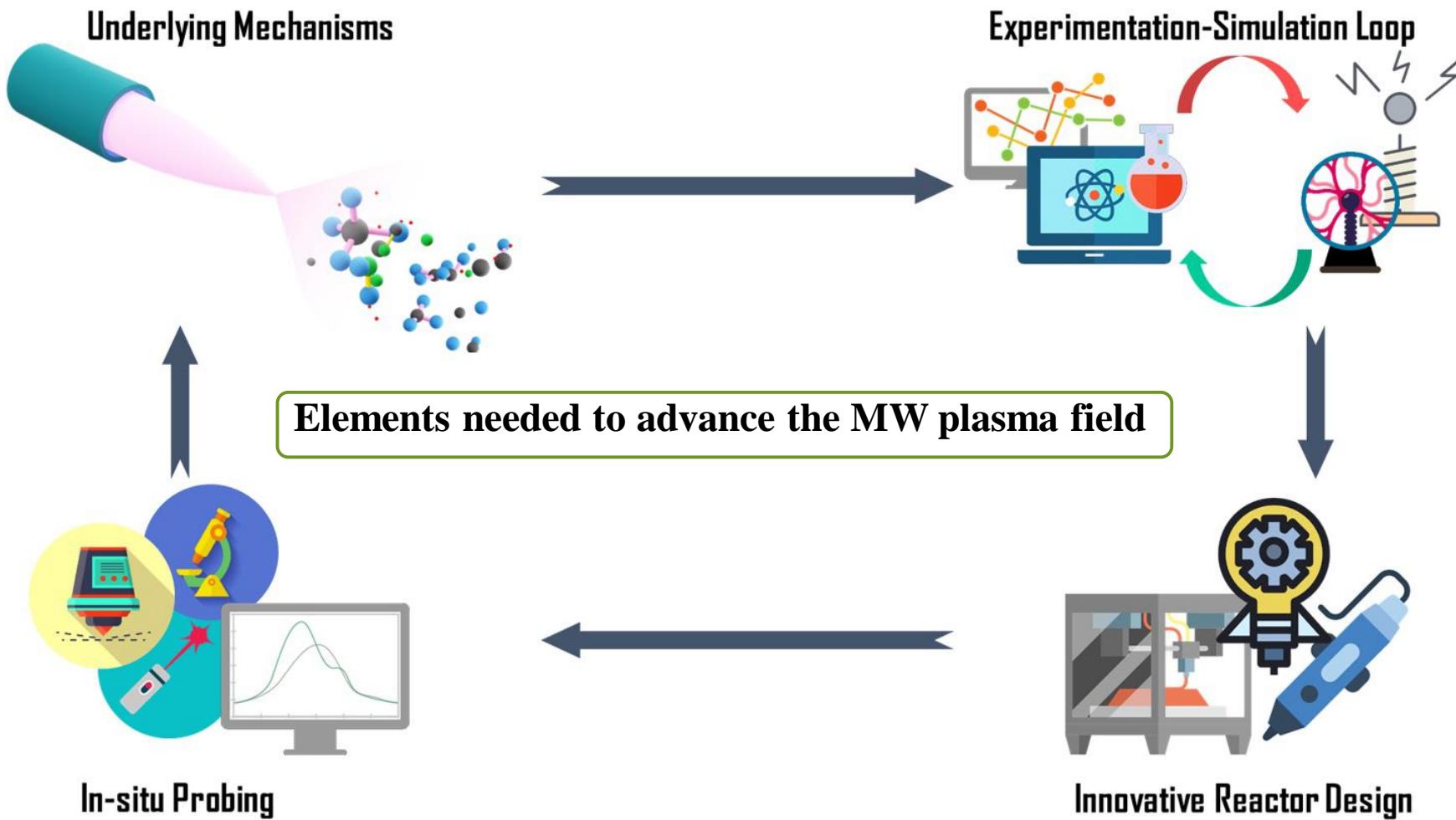
$$\text{Energy cost} = \frac{\text{SEI}}{X_{total}(\%)} \quad \text{Equation 1}$$

$$\text{Energy efficiency (\%)} = \frac{\Delta H_R \left(\frac{\text{kJ}}{\text{mol}} \right) \times X_{total}(\%)}{\text{SEI}} \quad \text{Equation 2}$$

$$\text{SEI} = \frac{\text{Plasma power (kW)}}{[CO+H2]_{produced} (l/min)} \quad \text{Equation 3}$$

Optimization of Process Parameters





Main objectives for drying reforming:

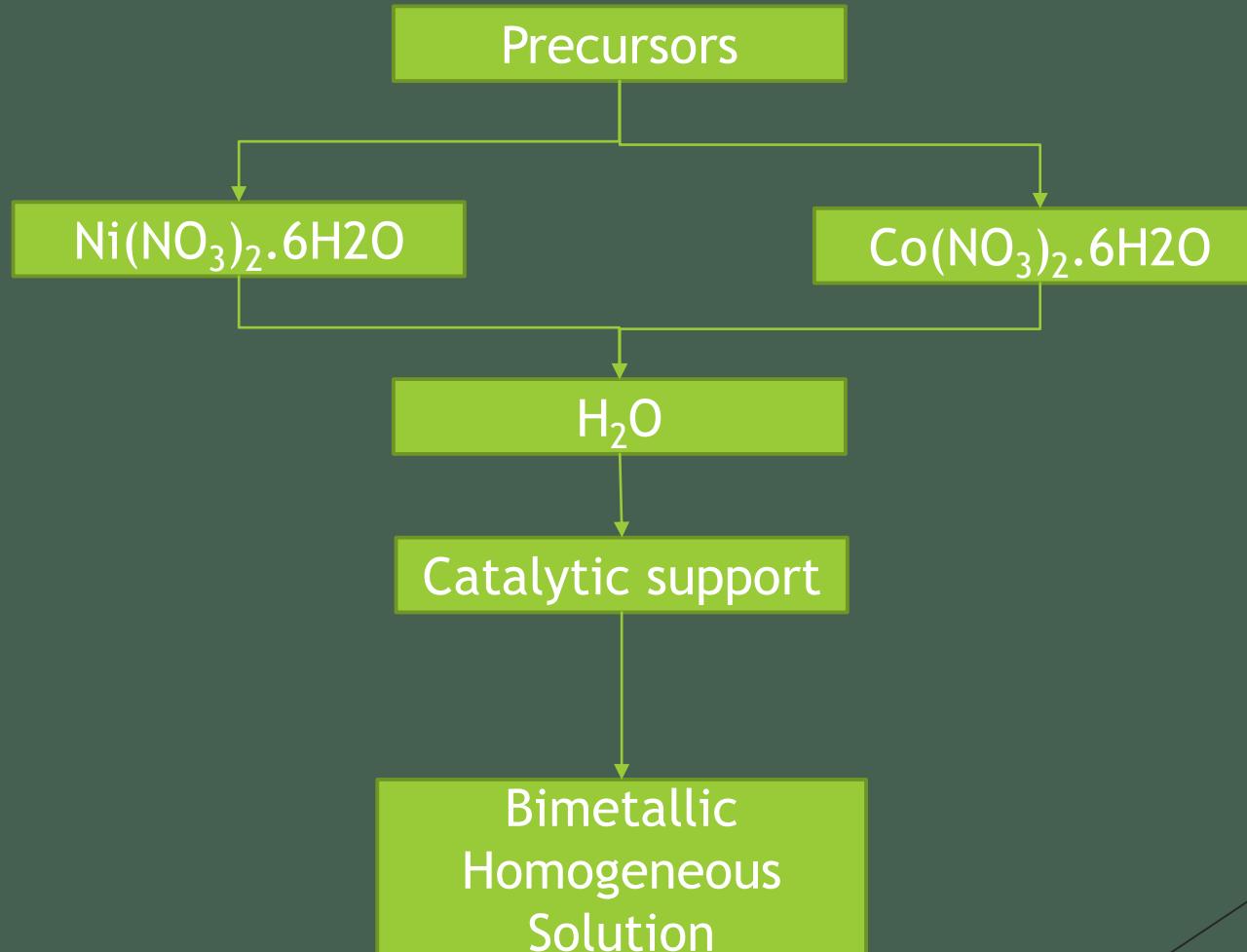
- ▶ Production of hydrogen together with carbon monoxide
- ▶ The use of catalysts mainly based on Nickel only or with other metals on several supports.
- ▶ Analysis and characterization of catalysts by different characterization techniques (XRD, SEM, BET ...)

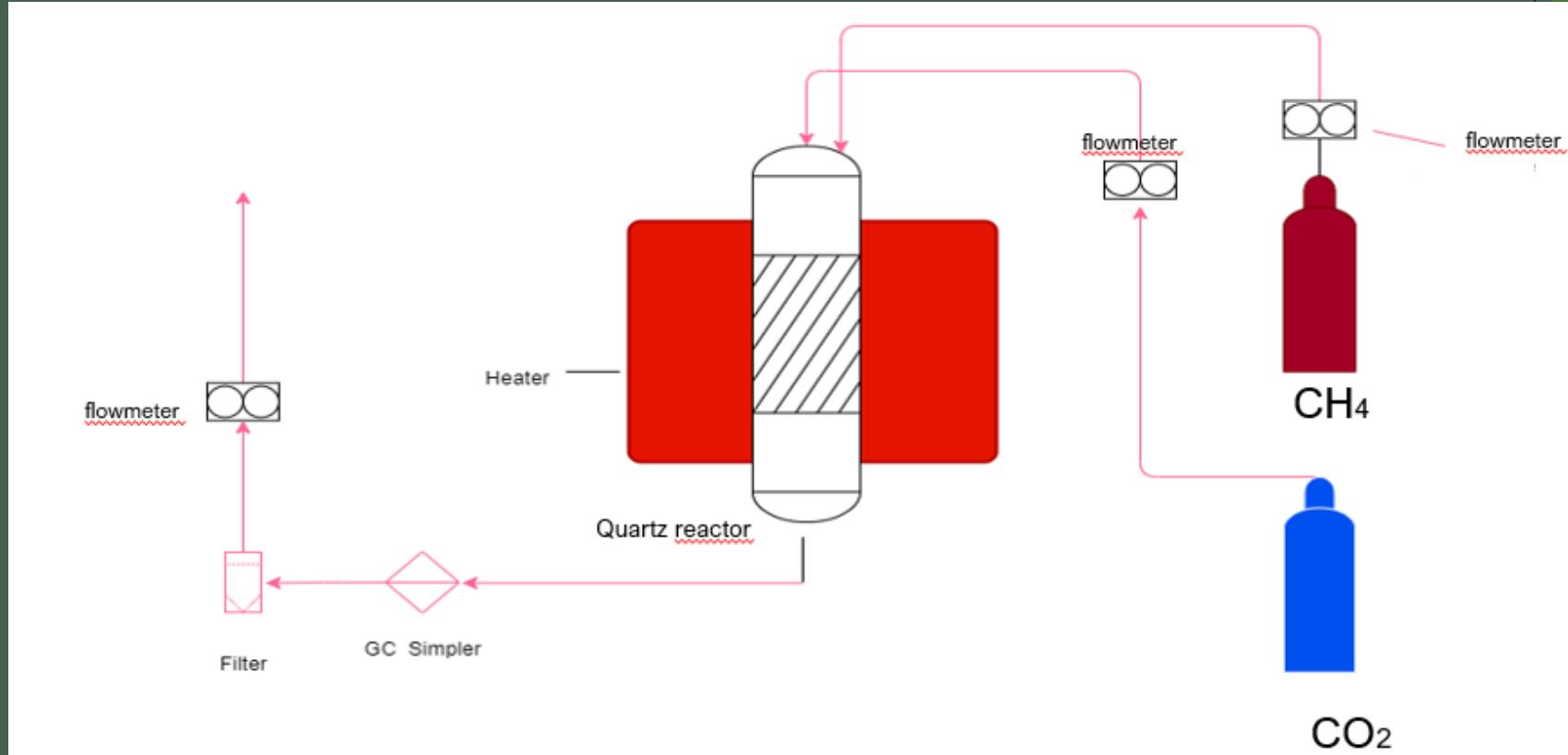


Experimental section

Synthesis of catalysts

Catalyst preparation by the wetness incipient impregnation method





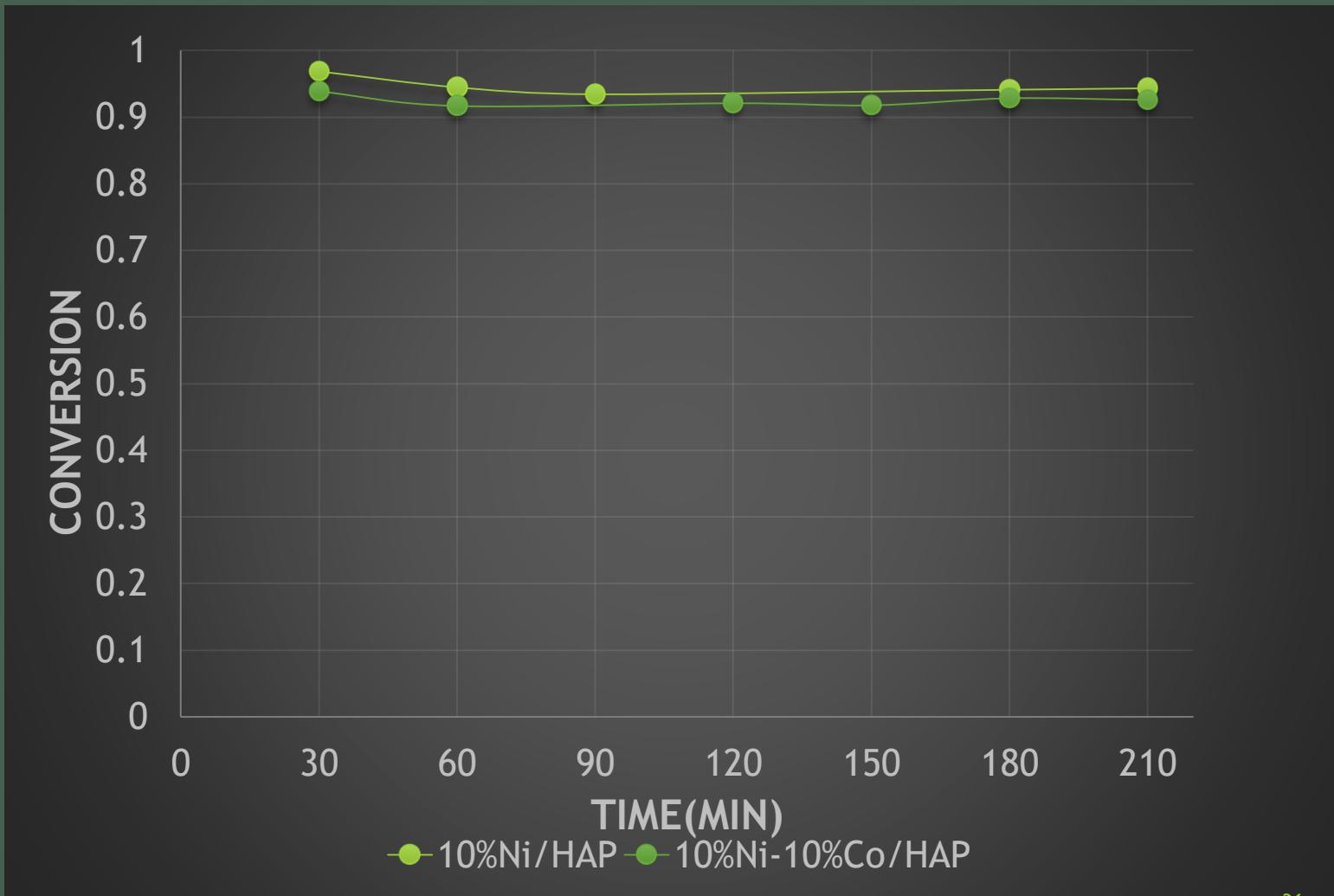
Experimental set-up for the dry reforming reaction

Experimental conditions:

- ▶ Temperature: 800 °C
- ▶ Pressure: atmospheric
- ▶ A ratio of $\text{CH}_4/\text{CO}_2=1$
- ▶ GHSV is chosen (1000) ($\text{ml.h}^{-1}.\text{g}^{-1}_{\text{cat}}$).

Conversion of CH_4

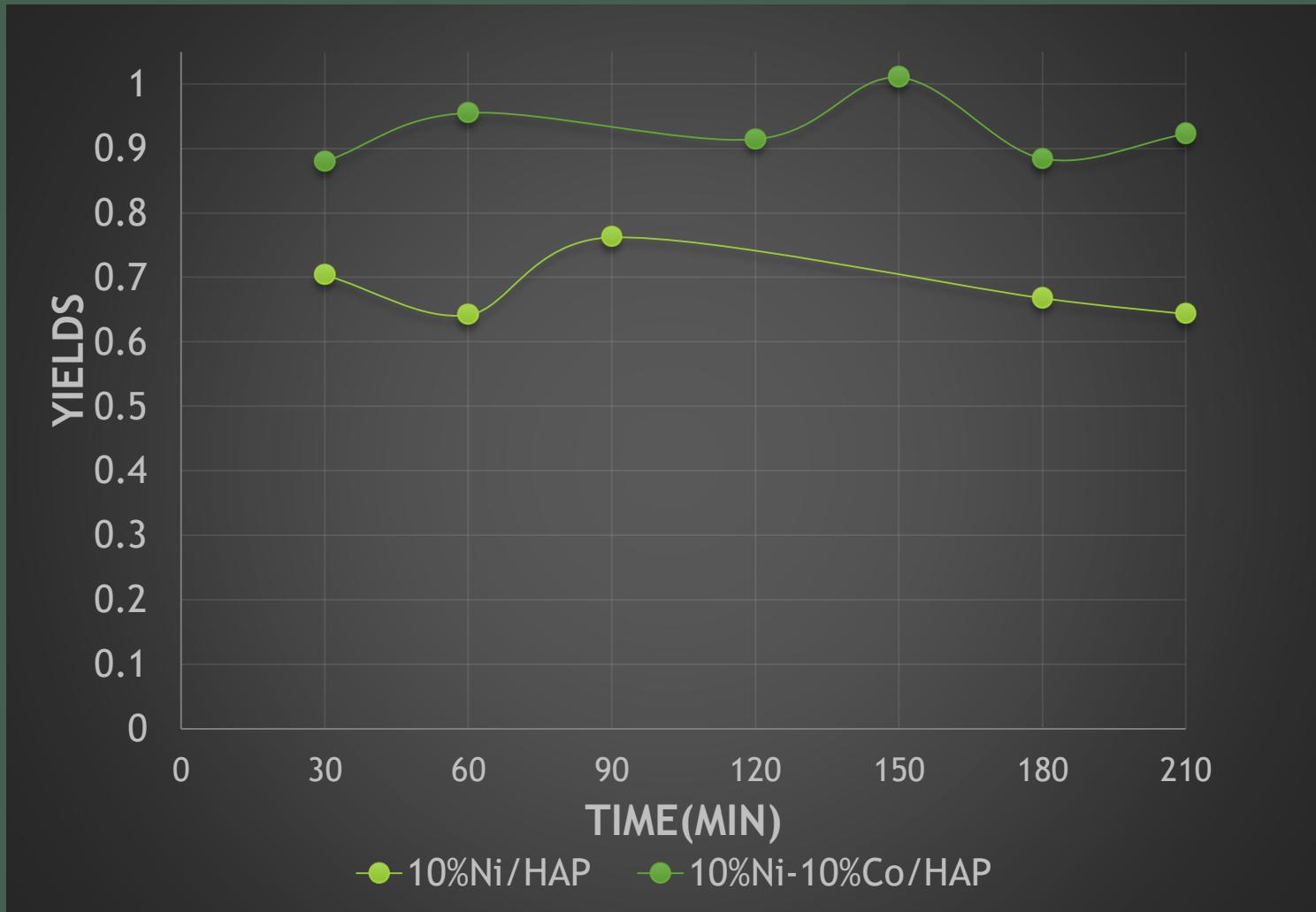
Conversions of 10%Ni, 10%Ni-10%Co/HAP



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Yields

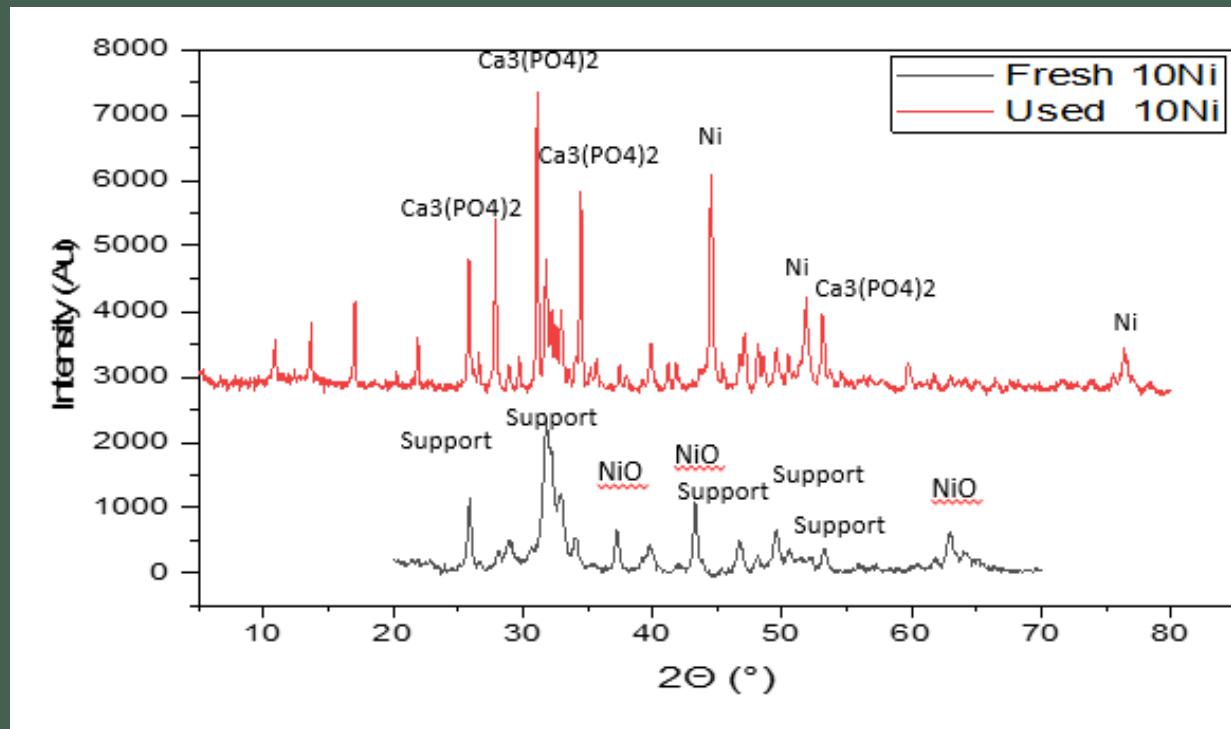
Hydrogen yields of 10%Ni, 10%Ni-10%Co/HAP



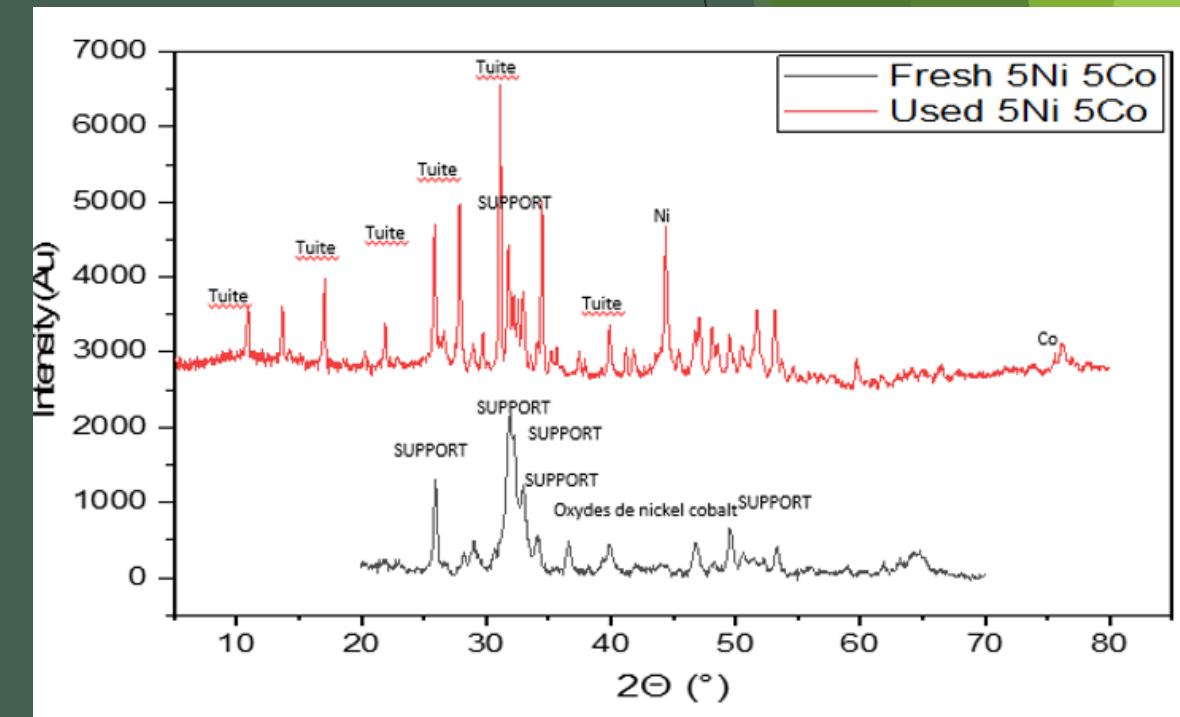
characterization of catalysts



XRD Characterization of fresh and used catalysts



XRD for 10%Ni/Hap



XRD for 10%Ni-10%Co/Hap

Project 2: CO₂ direct hydrogenation

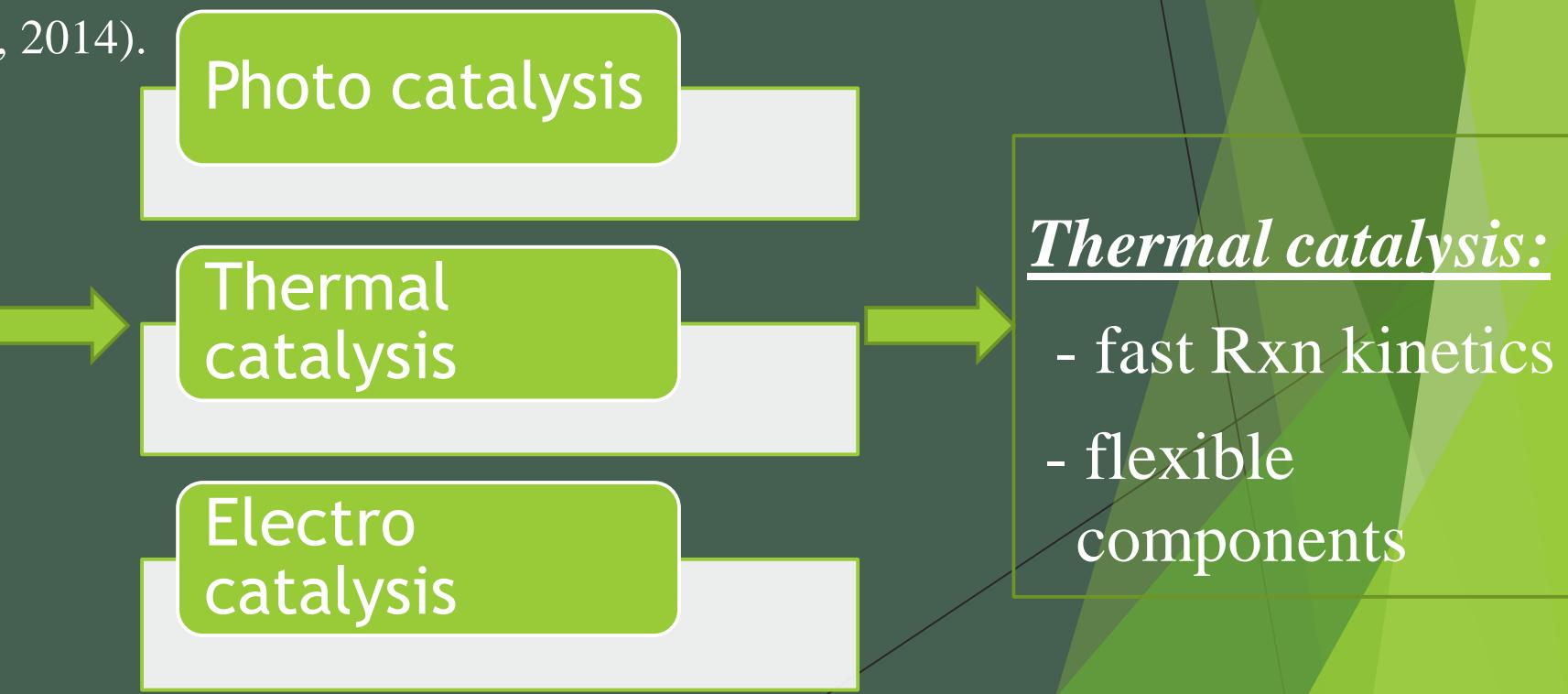
► PhD student: Farbod Farzi



Thermal catalysis of CO₂

- Considering the CO₂ limitations of being soluble in water and its thermodynamic stability, CO₂ conversion to commercial products is deemed to be highly energy intensive as thermal decomposition of pure CO₂ commences at 2500-3000 K (Kozák, 2014).

CO₂ reduction can be catalyzed via:



Thermal catalysis of CO₂ (Cont'd)

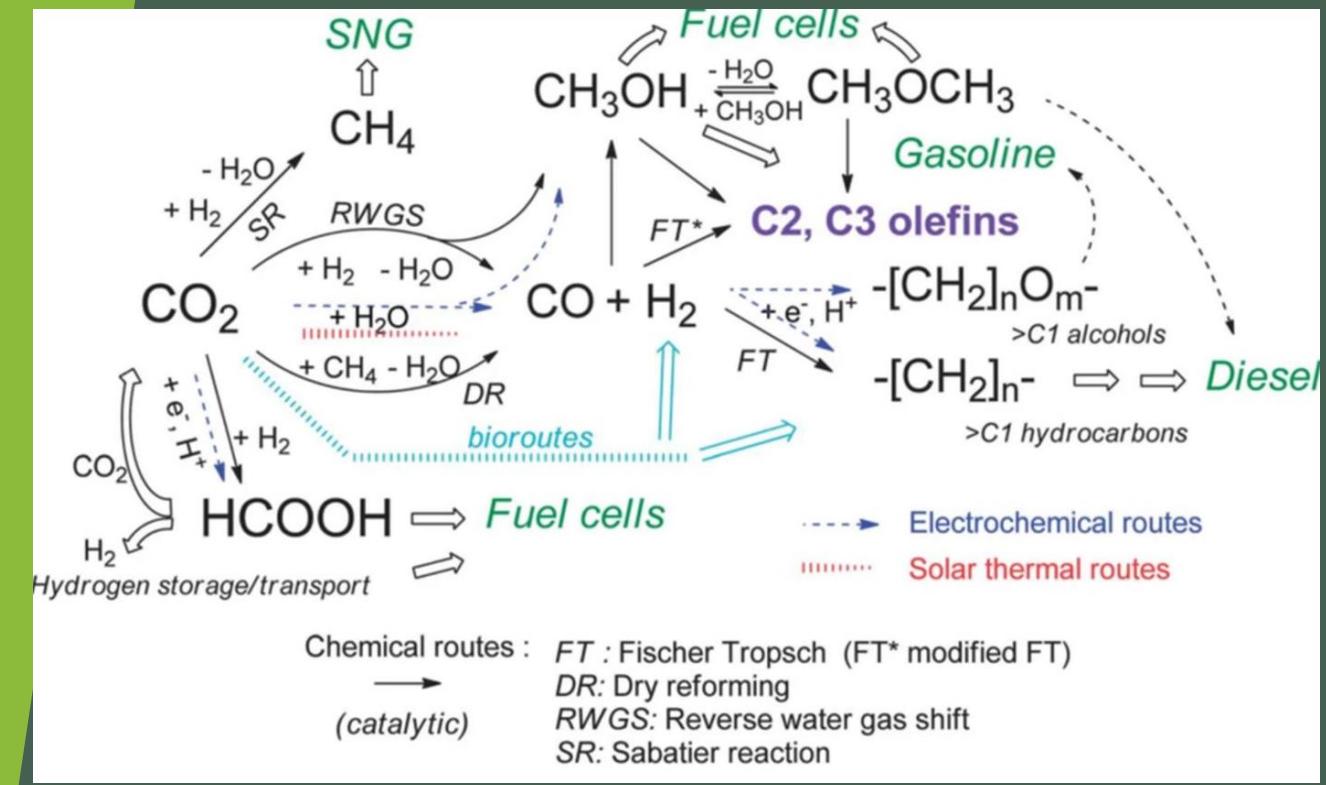
CO₂ is thermodynamically stable;

Activation and more importantly reduction of CO₂ is energy-intensive

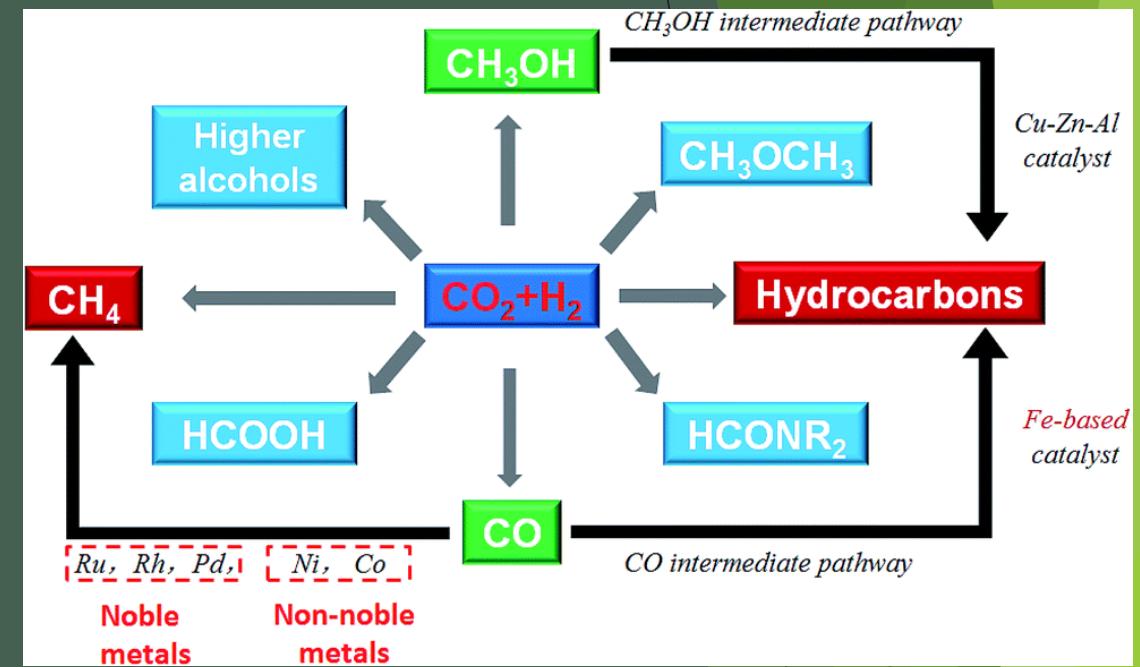


Addition of a higher Gibbs energy component such as H₂ will make the conversion more favorable from the thermodynamic point of view (Li W. W., 2018)

CO₂ hydrogenation products



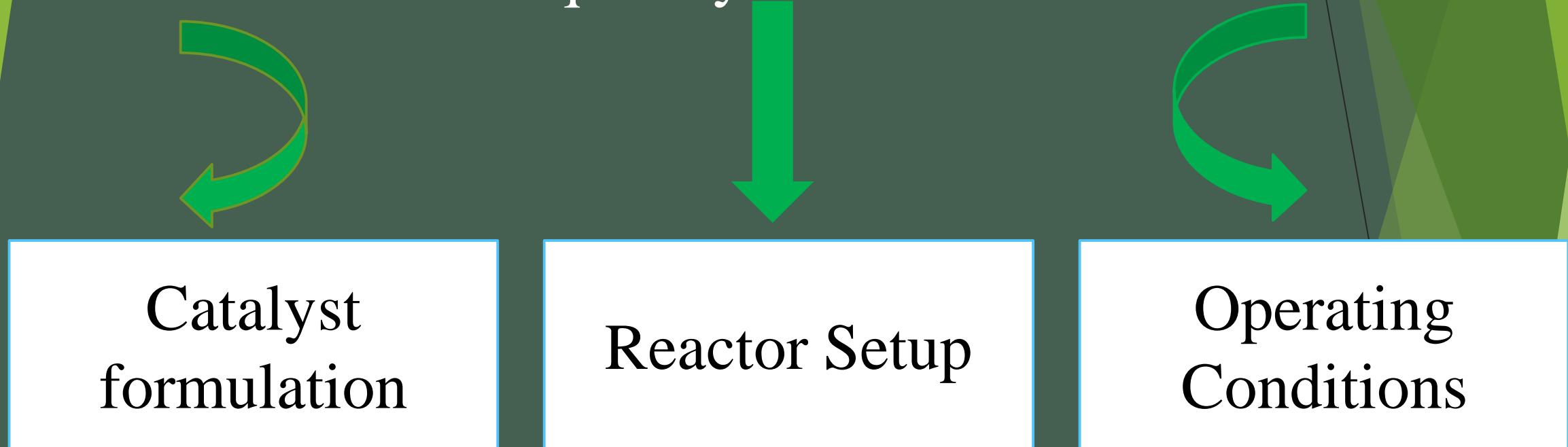
(Adamu, 2020, 10.1186/s42480-019-0026-4)



(Li, 2018, 10.1039/C7RA13546G)

Experimental section

Study of thermal catalysis of CO_2 hydrogenation reaction towards liquid hydrocarbons



Experimental section (Cont'd)

Catalyst Formulation

Active metal:

Monometallic:

- Fe
- Co
- Cu
- Mn
- Ni, ...

Bimetallic:

- Ni-Co
- Co-Fe, ...

Support:

- ✓ Al_2O_3 ,
- ✓ SiO_2 ,
- ✓ TiO_2 ,
- ✓ CeO_2 ,
- ✓ ZrO_2 ,
- ✓ Ce-ZrO₂,
- ✓ Zeolites (Hbeta, HZSM-5, SAPO-34, ...)
- ✓ $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ commonly known as Hydroxyapatite (HAP) was used as a catalyst support for Dry Reforming of Methane (DRM)

Our formulations:

- Cu/HAP
- Mn/HAP
- Ni-Co/HAP
- Co-Fe/HAP
- $\text{Ni/ZrO}_2\text{-HAP}$

Experimental section (Cont'd)

Catalyst Synthesize

Synthesize methods:

- precipitation
- impregnation (dry & wet)
- sol-gel
- thermal
- solvothermal
- solvent-free methods
- plasma decomposition
- reverse microemulsion

(Park & McFarland, 2009),
(Chew, et al., 2014),
(Lu, Fatah, & Khodakov, 2017),
(Jia, Zhang, Rui, Hu, & Liu, 2018)

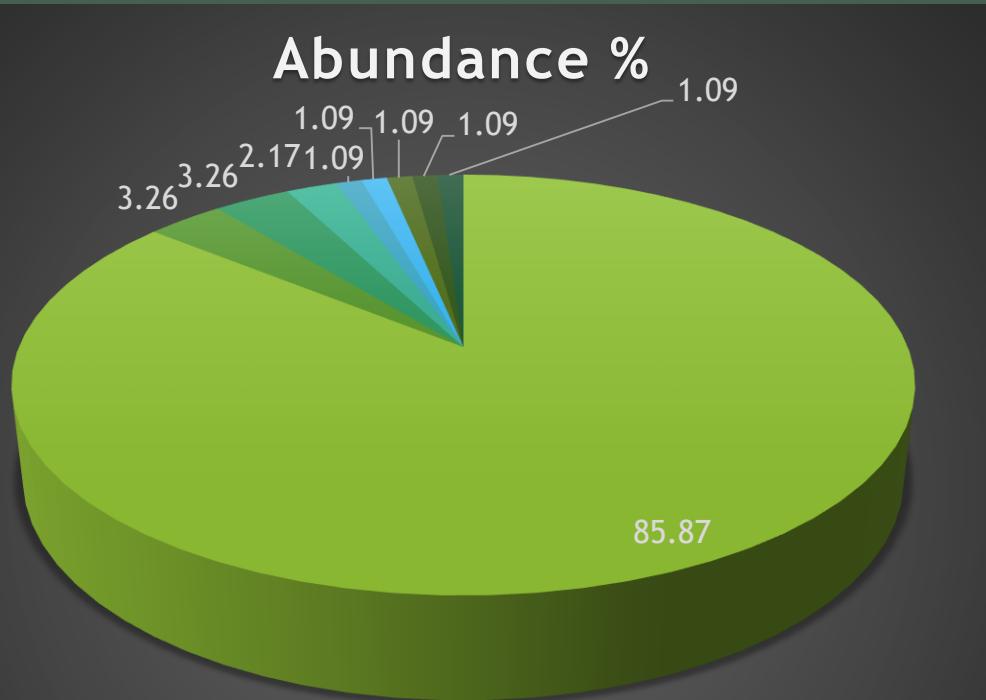
Our synthesize method:

- ✓ incipient wetness co-impregnation
- Simplicity of operation
- Controlled measures
- Proper way of fine distribution of metal over support shell
- Mixing & Solid handling solutions available for scale-up purposes

Post-synthesize steps:

- Drying at 105 °C, overnight
- Calcination at 500 °C under static air atmosphere for 2h

REACTOR CONFIGURATION

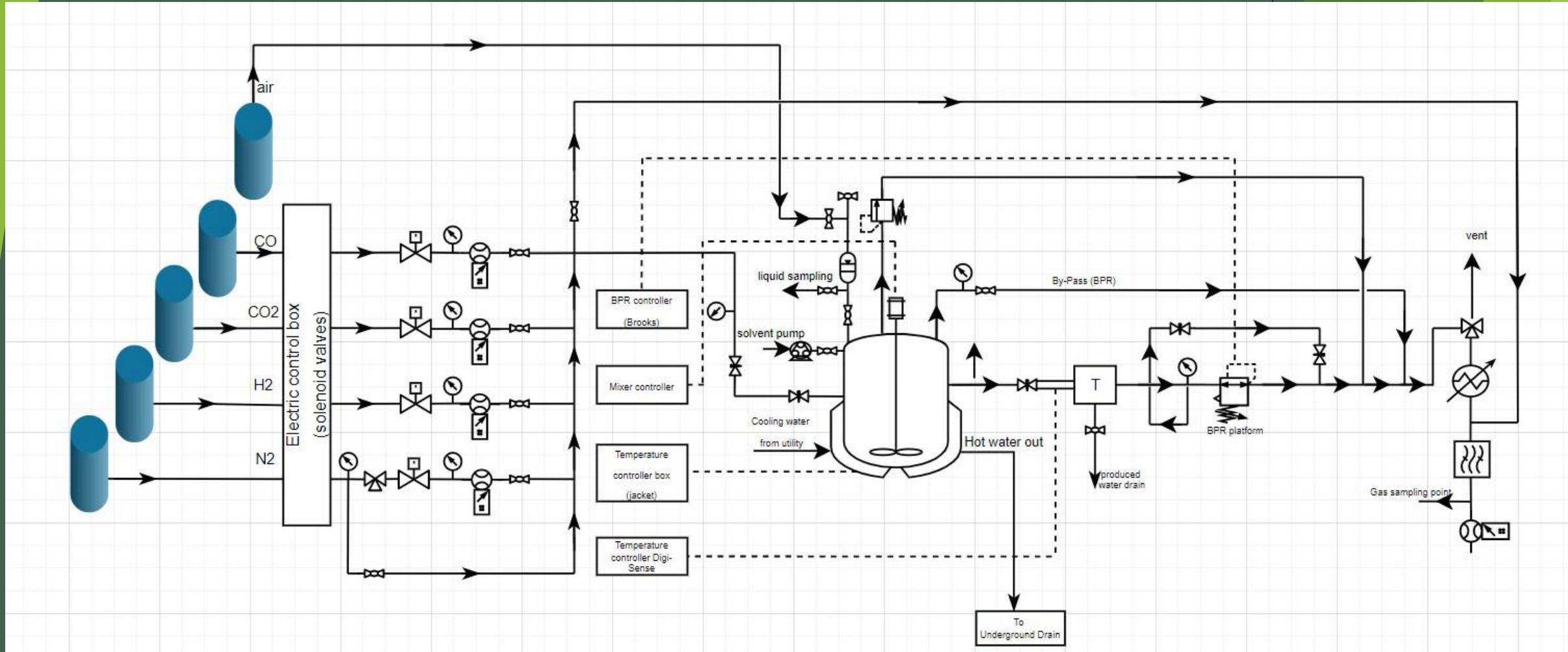


- Fixed Bed
- Slurry
- Parallel reactors
- Recycle
- Fischer-Porter
- Series
- Fluidized bed
- Parrallel fixed bed
- Two-stage

Among the 92 investigations available in the recent literature, 79 were performed in a fixed bed reactor, revealing a huge potential to study other reactor configurations.

Experimental section (Cont'd)

Reactor Set-up

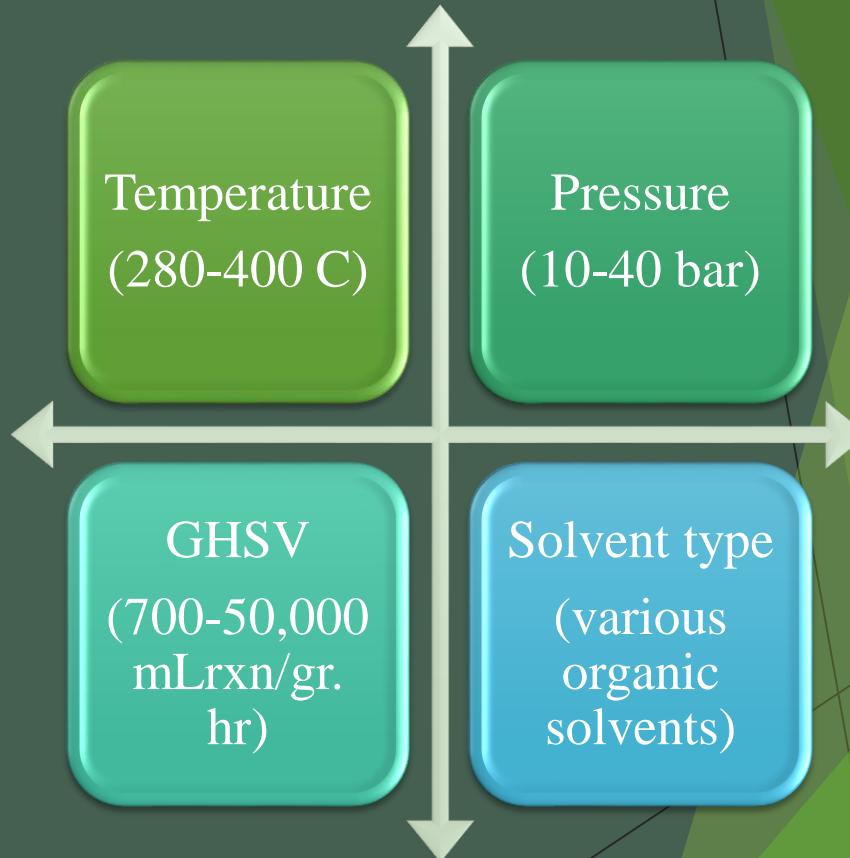


Experimental section (Cont'd)

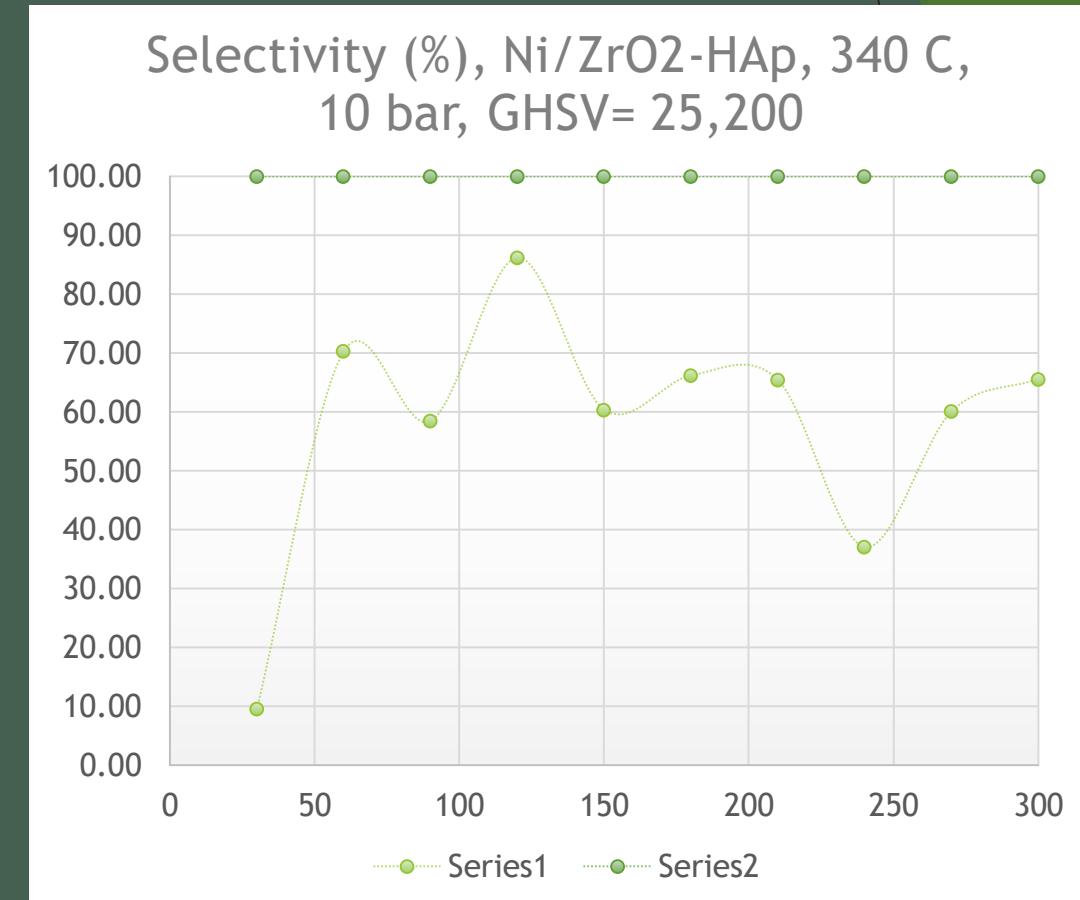
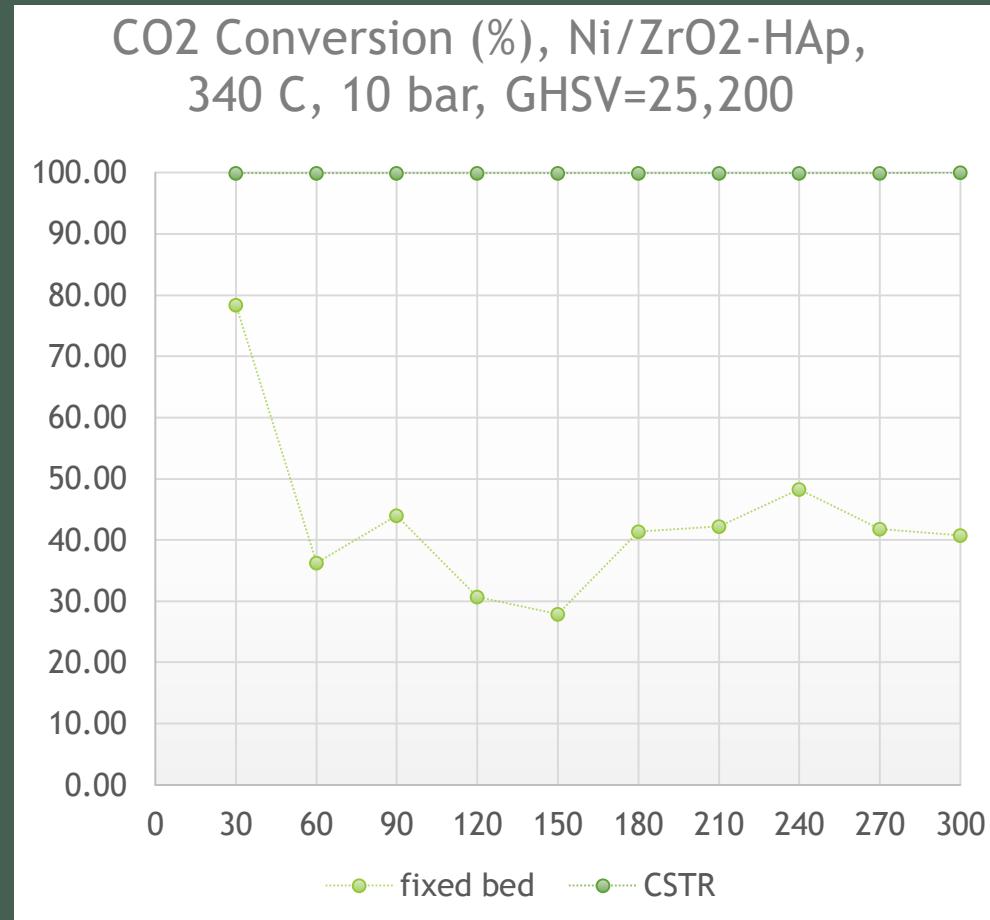
Operating Conditions

Pre-treatment conditions:

Item	Desc.	Value
1	Temp.	500 °C
2	Press.	3 bar
3	Flow composition	50 vol% H ₂ /N ₂
4	Flow rate	500 mL/min
5	TOS	2 h

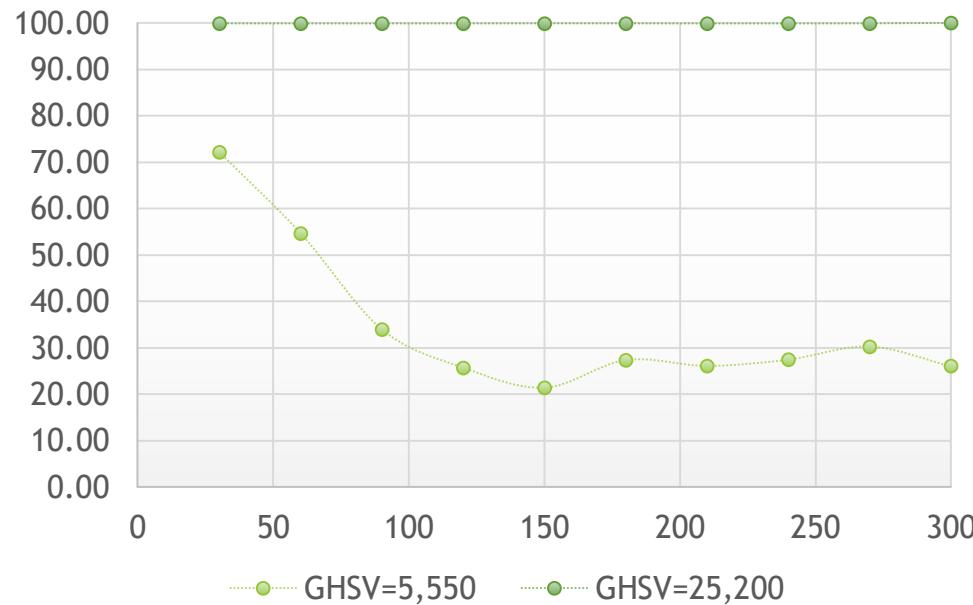


Effect of reactor configuration on CO₂ Conversion & product Selectivity

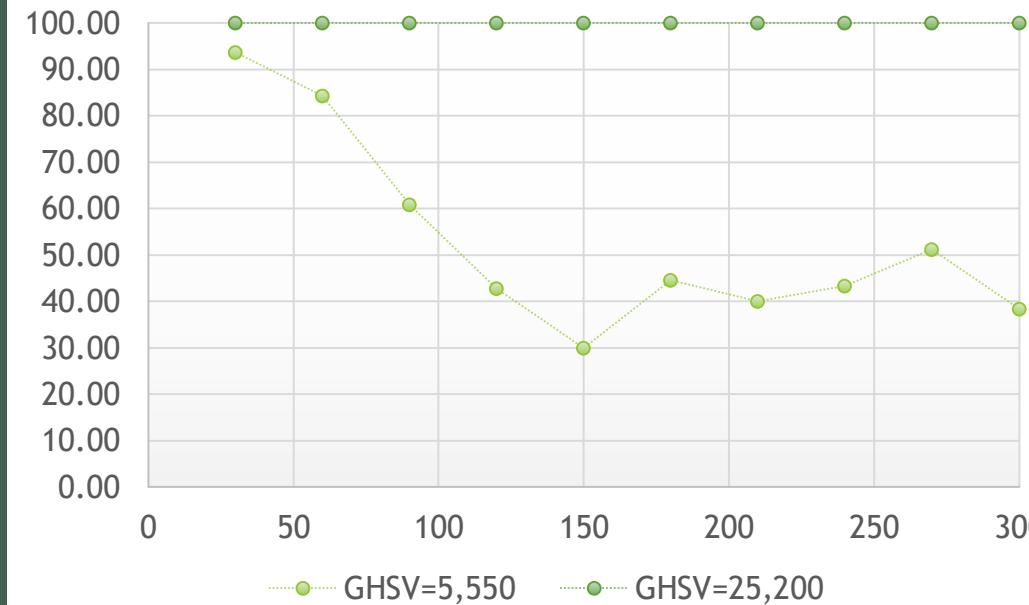


Effect of GHSV on CO₂ Conversion & product Selectivity

CO₂ Conversion (%), Ni/ZrO₂-HAp, 340 C, 10 bar



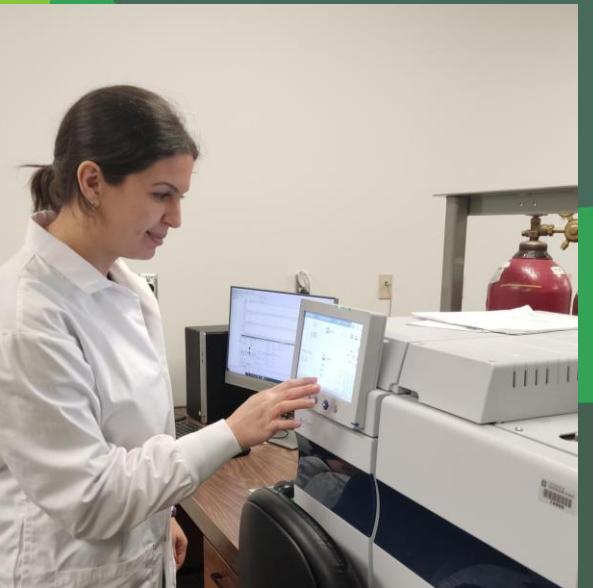
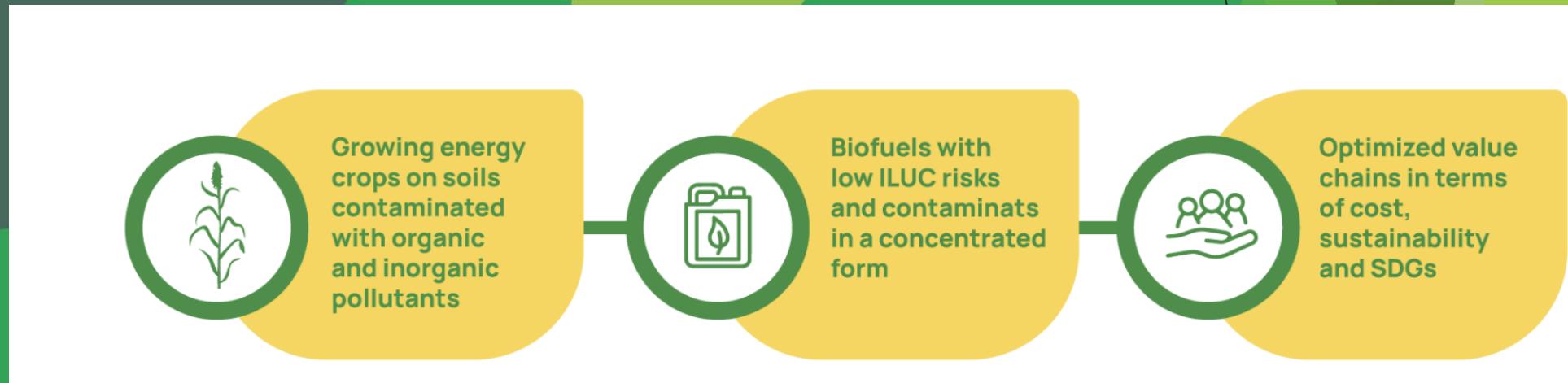
Selectivity (C5+), Ni/ZrO₂-HAp, 340 C, 10 bar



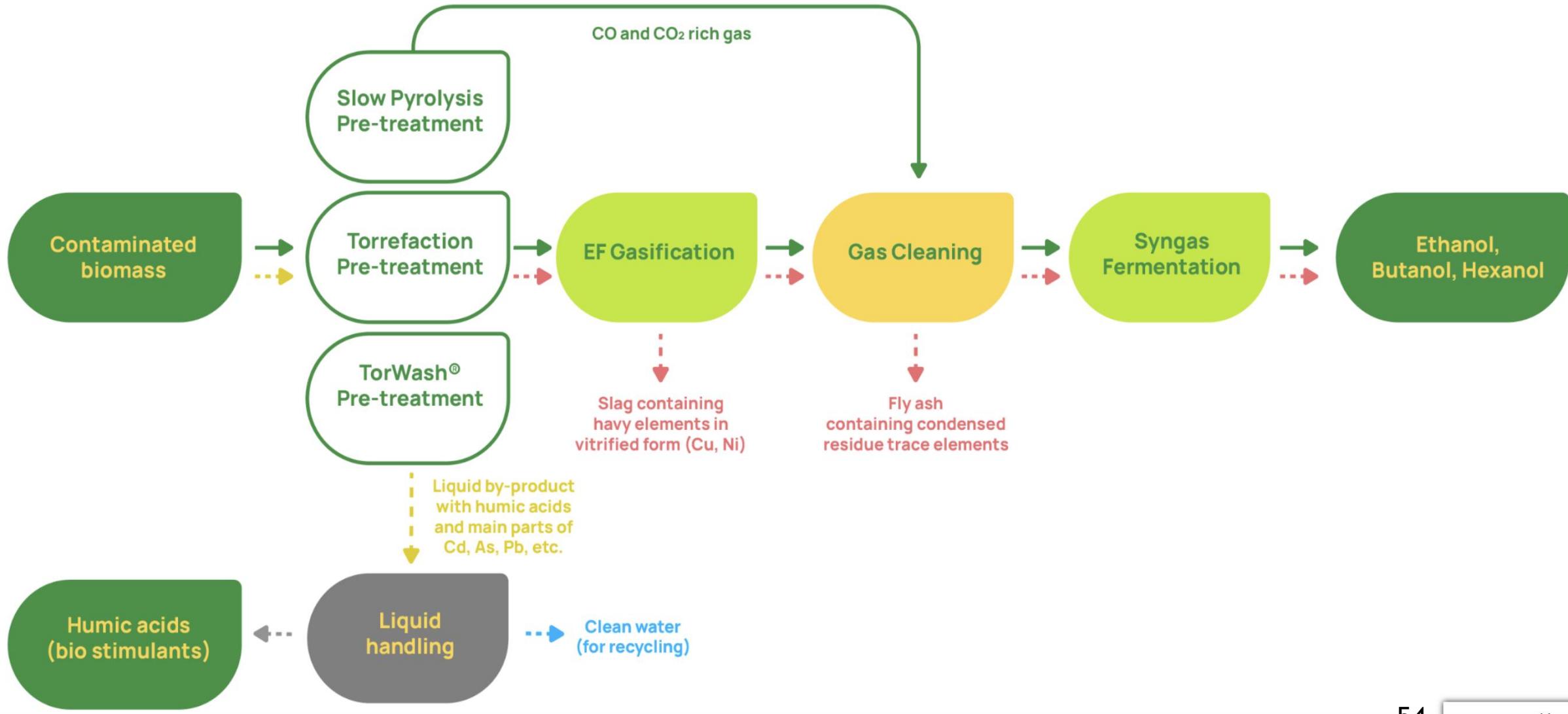
Project 3: Pyrolysis and FTS for clean low ILUC biofuels production

Horizon project: *Bridging the gap between phytoremediation solutions on growing energy crops on contaminated lands and clean biofuel production*

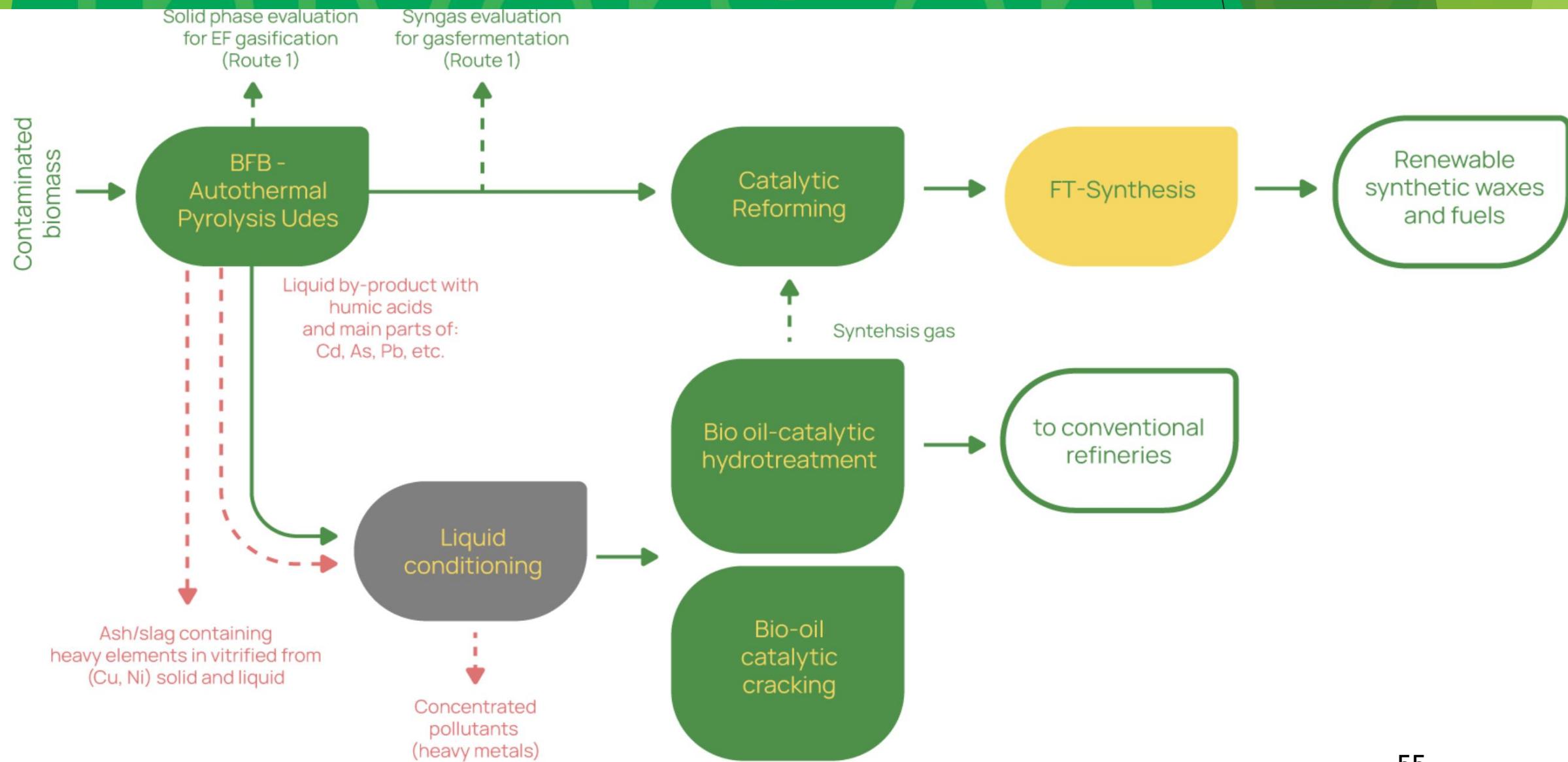
Collaborative project GRTP with 18 parteners around the world



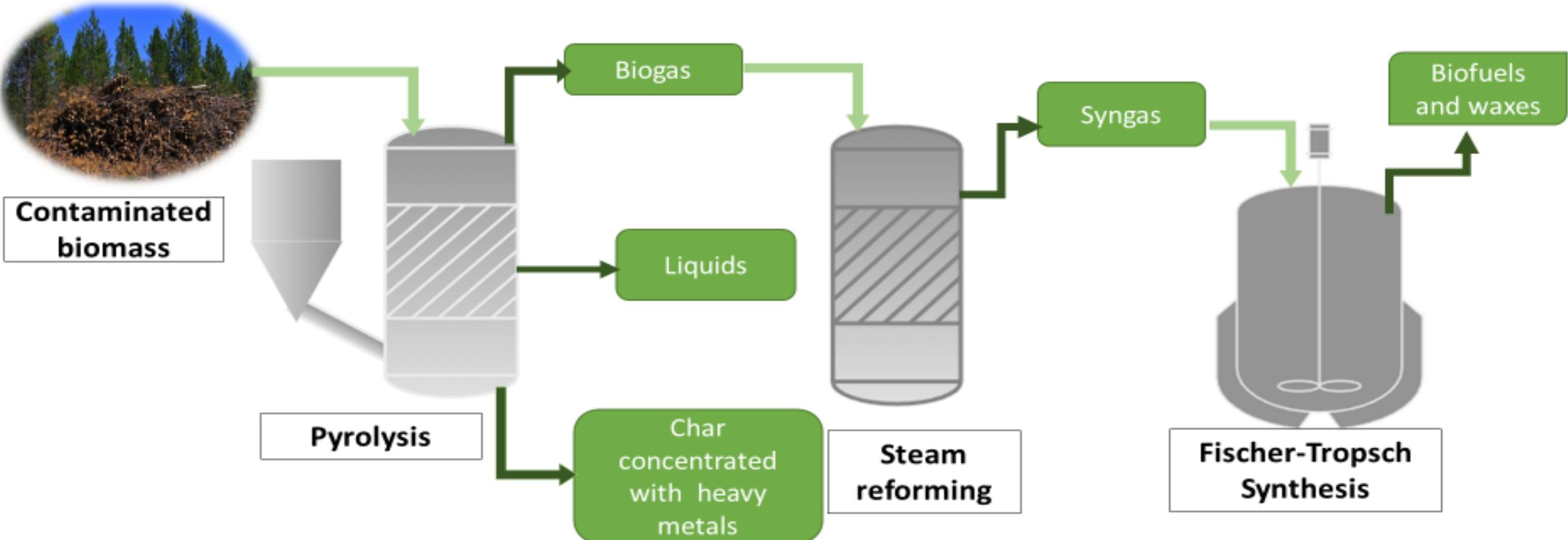
GOLD –route 1: Biofuels productions



GOLD -route 2 biofuels productions



GOLD Project at UDS



PhD 1 – Pyrolysis of contaminated biomass: Problematic

What is the behavior of heavy metals during the pyrolysis of naturally and artificially contaminated biomasses ?

What is the role of heavy metals during the pyrolysis of biomass

What are the optimal pyrolysis conditions to have an optimal yield of bio-oil with a minimum of heavy metal contamination?

Contamination sources

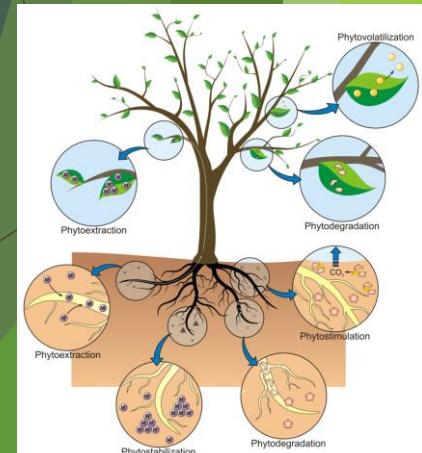


Fate of heavy metals

Thermochemical conversion



Phytoextraction



PhD 2 Contaminated lignocellulosic biomass to pyrolysis oil: Analytical and Upgrading studies



Heavy metal-Contaminated biomass

Problematic



- How can we fully identify and quantify the chemical composition of the pyrolysis liquid?
- How can we achieve contamination-free bio-oil production

Bio-oil production

*Clean biofuels
Low ILUC risk*



Pyrolysis

- *Thermochemical conversion*
- *System optimization*

Upgrading process

Problematic



- What is the appropriate route to upgrade the pyrolysis liquid into biofuels and added value chemicals ?

Phytoremediation to biofuel: Challenges and limitations



The key challenge in using biomass for bioenergy is the question of **pollution transfer** and **heavy metal content** of biomass.

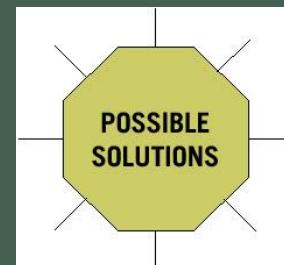


Emissions that may be generated in the use of contaminated plants for bioenergy.



Bioenergy production with minimal environmental impacts

Management of by-products and residual products

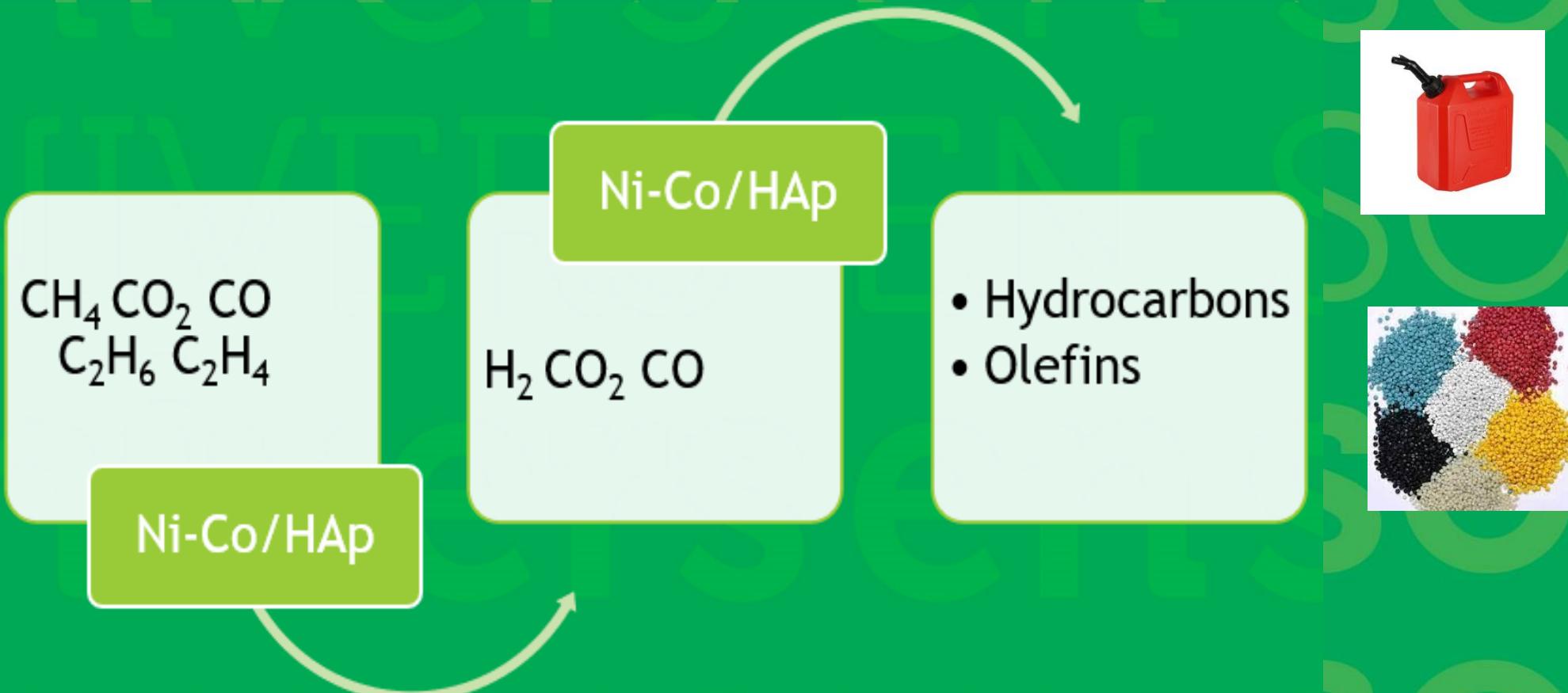


- Extraction of pollutants in concentrated form in the solid part (biochar).
- Valorization of biochar contaminated by heavy metals as a catalyst.

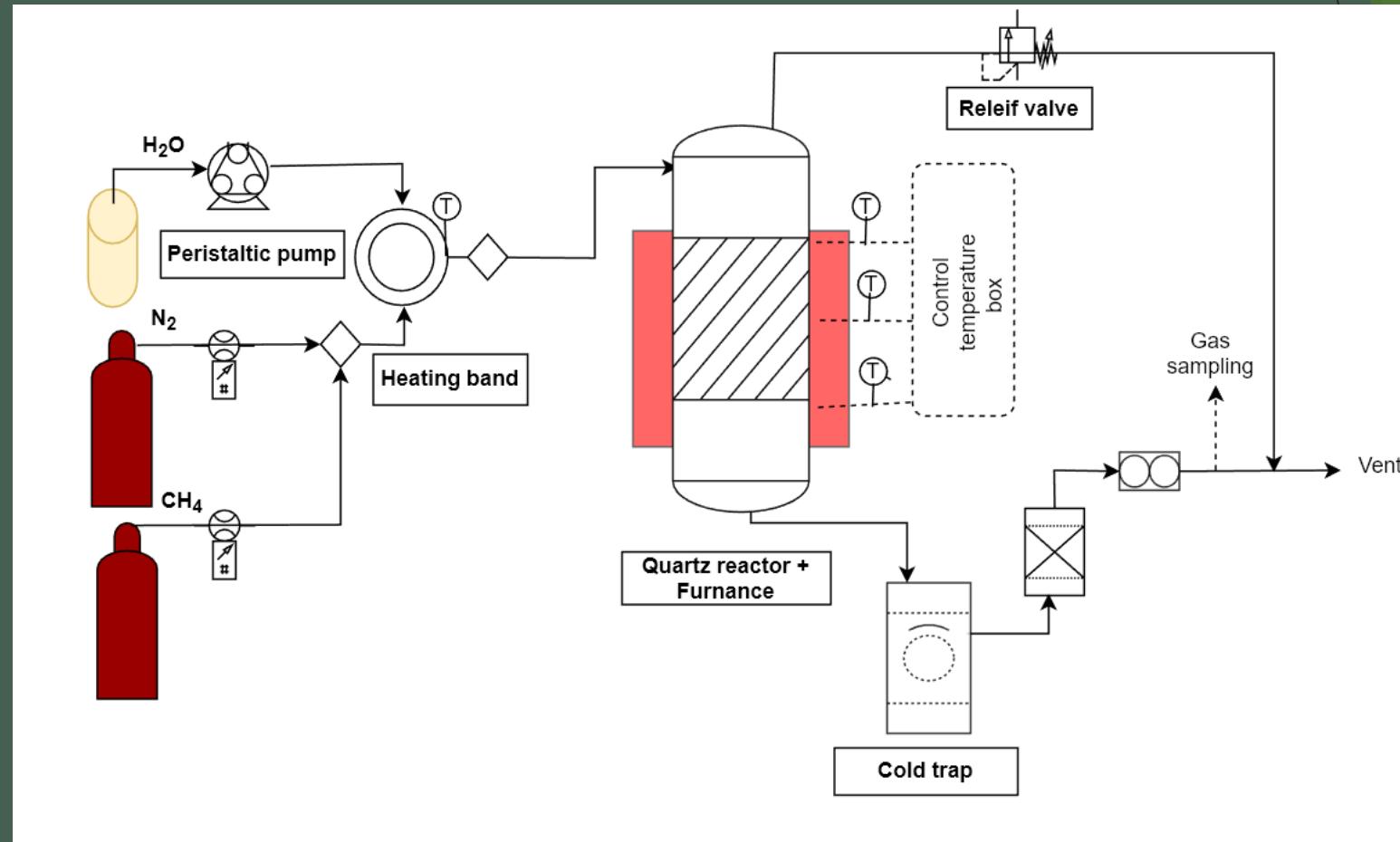


Feedstock for pyrolysis

PhD 3- Steam reforming and Fischer-Tropsch Synthesis



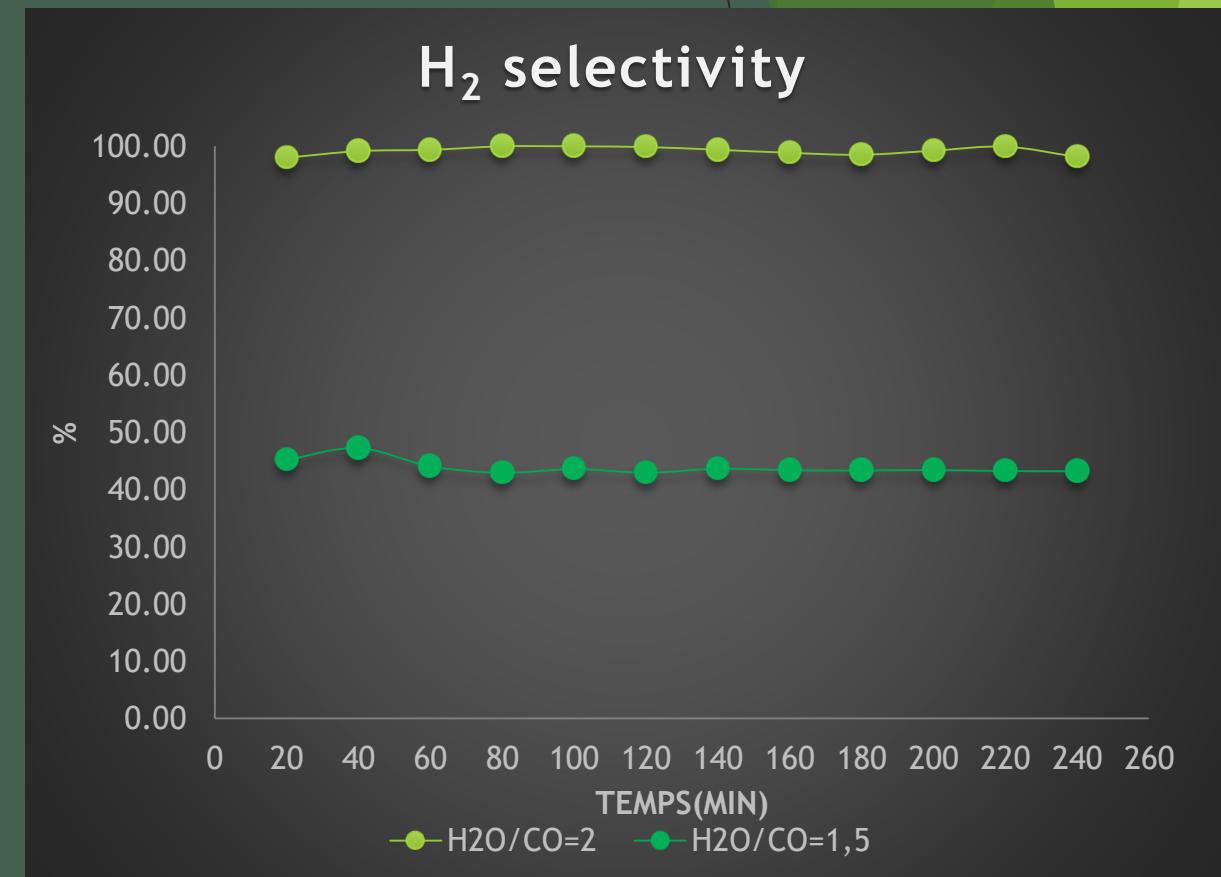
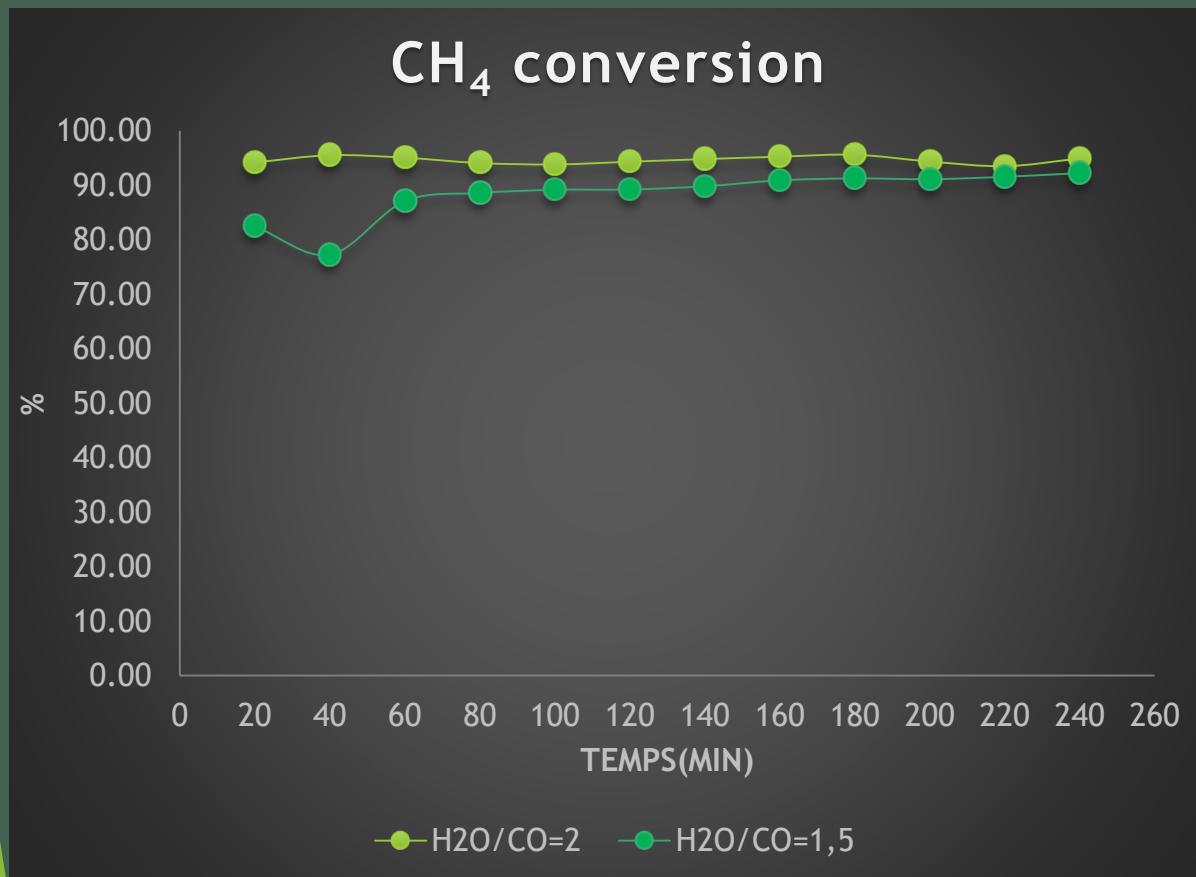
Reactor design for steam reforming



Operation conditions of the experiment

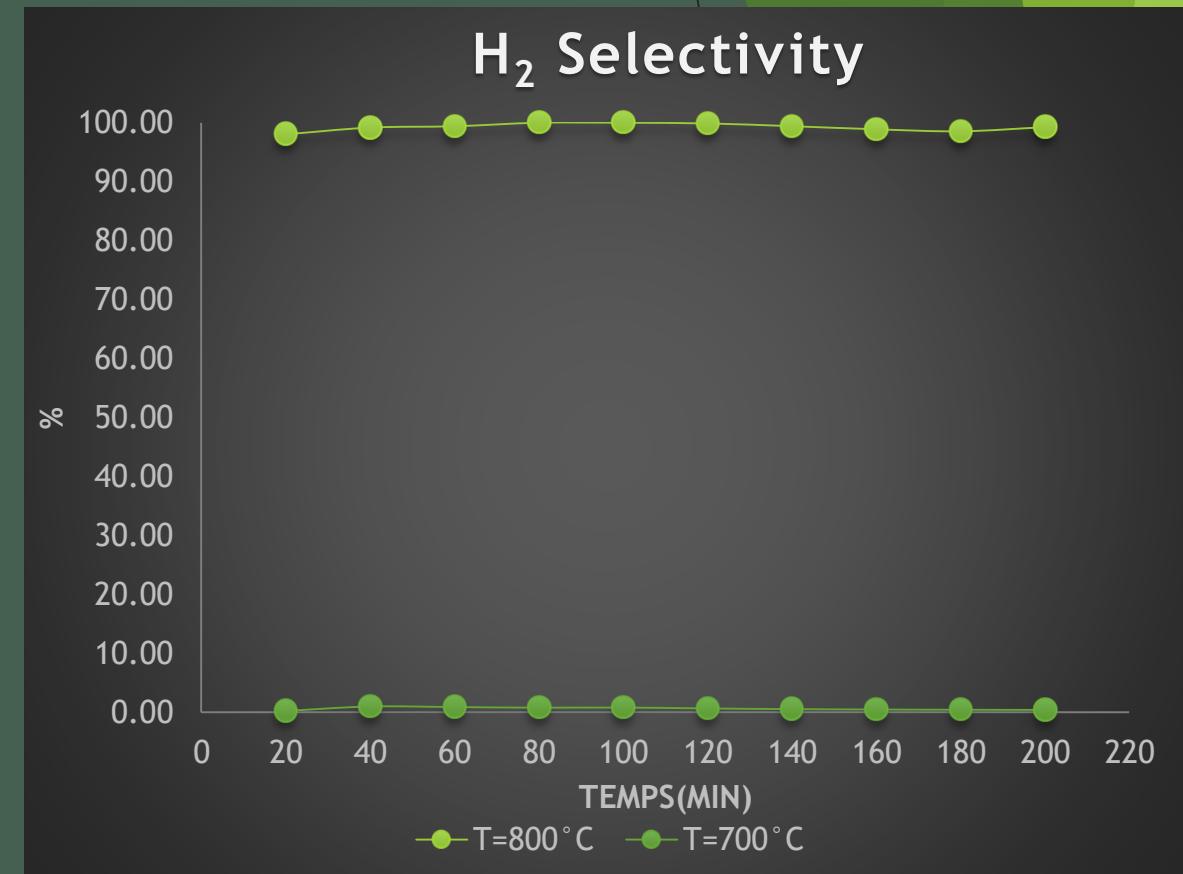
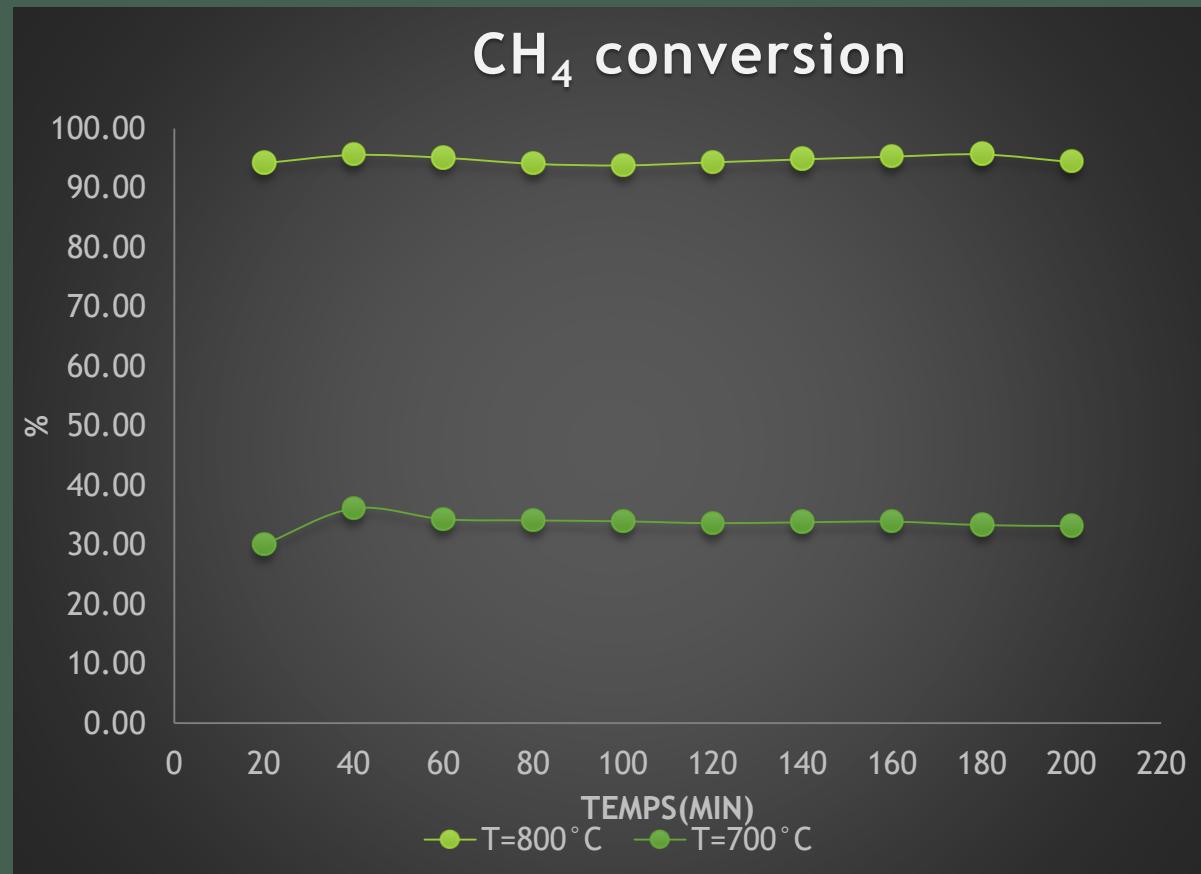
Run	P (atm)	T (°C)	H ₂ O/CH ₄	GHSV (ml.h ⁻¹ .g ⁻¹ _{cat})
1	1	800	2	1656
2	1	800	1,5	1656
3	1	700	2	1656
5	1	800	2	4140

Effect of $\text{H}_2\text{O}/\text{CH}_4$ ratio on catalyst activity



T=800 °C, P=1atm, GHSV=1656 ml.h⁻¹.g⁻¹_{cat}

Effect of temperature on catalyst activity



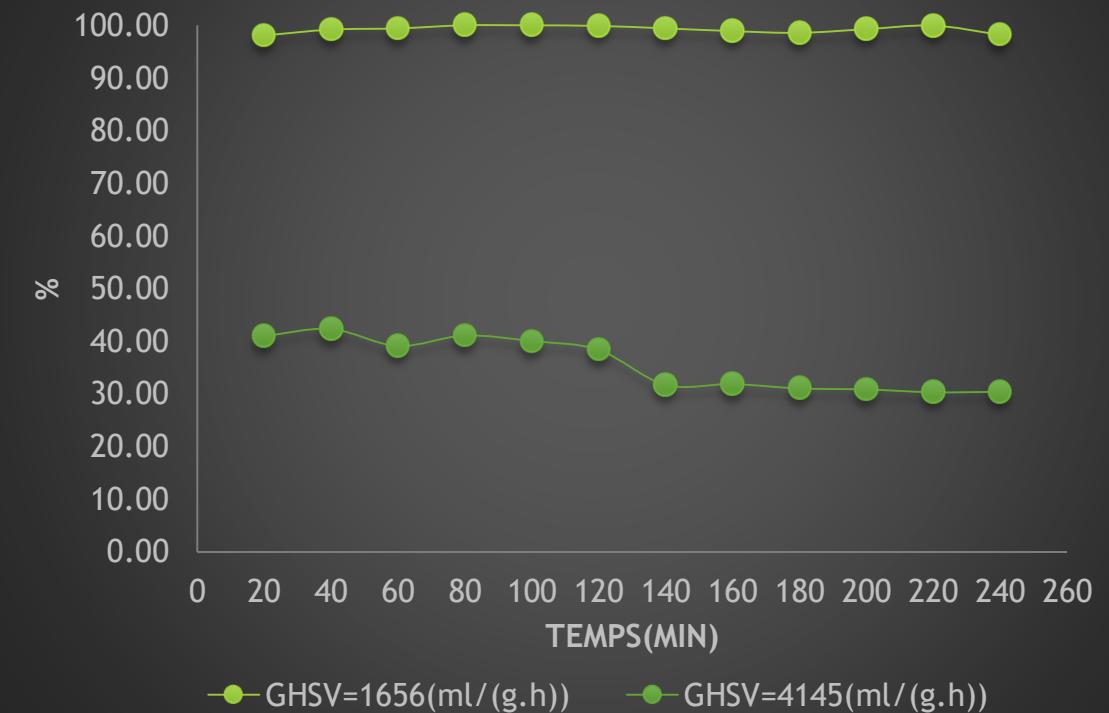
H₂O/CH₄=2, P=1atm, GHSV=1656 ml.h⁻¹.g⁻¹_{cat}

Effect of GHSV on catalyst activity

CH₄ conversion



H₂ SELECTIVITY



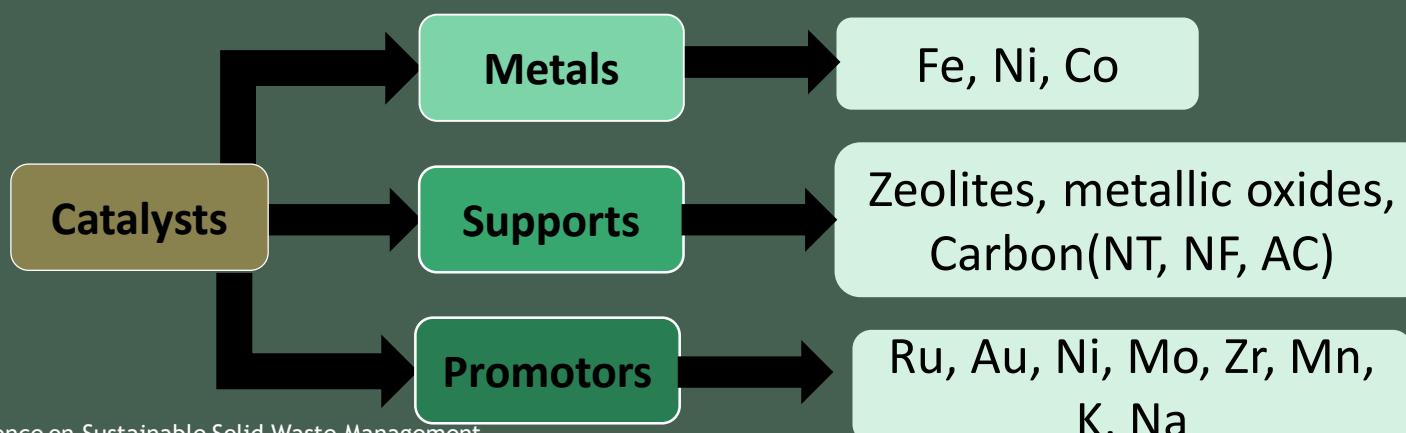
H₂O/CH₄=2, P=1atm, T=800 °C

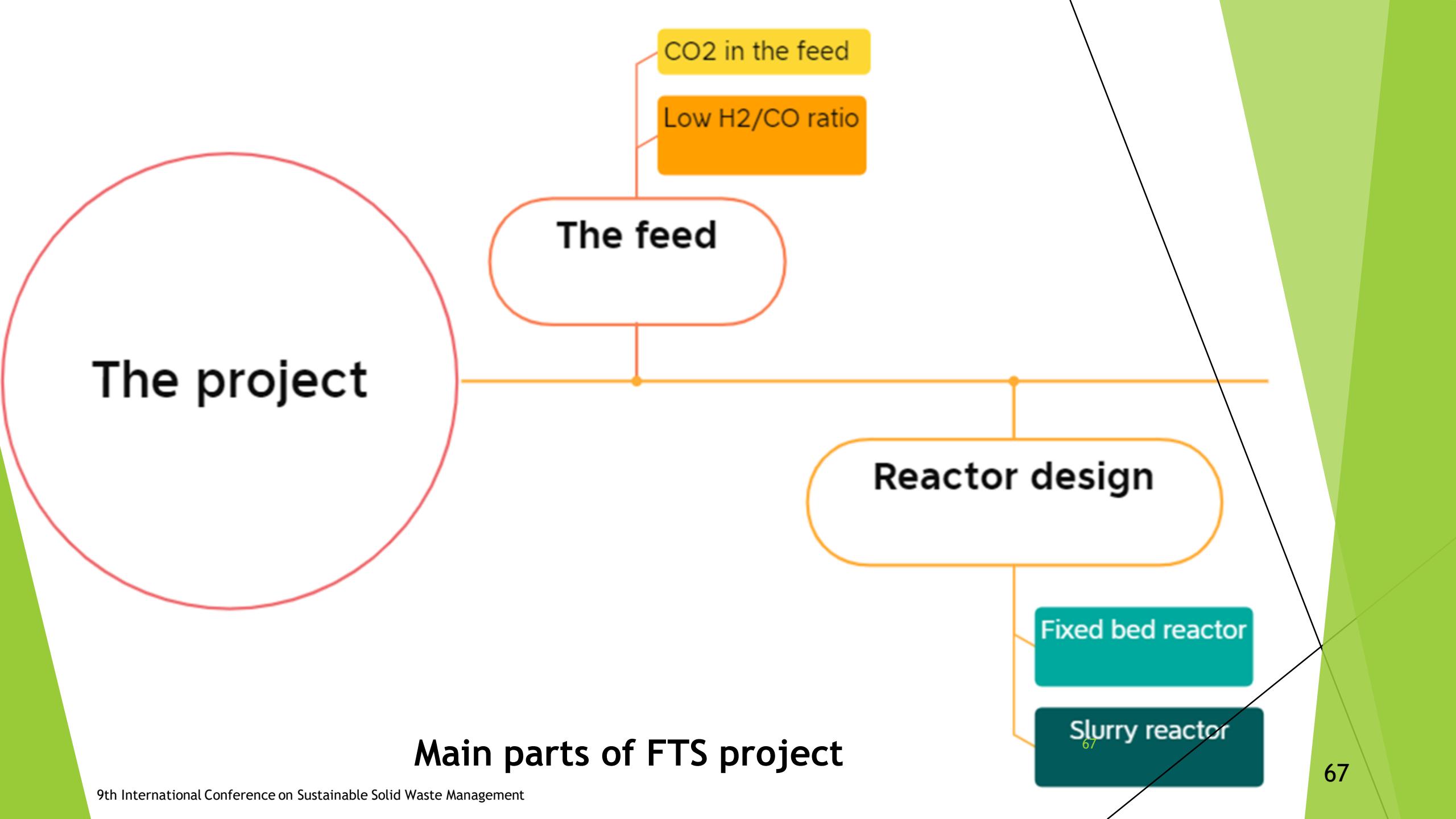
Fischer-Tropsch Synthesis

I. Fischer-Tropsch reactions and products

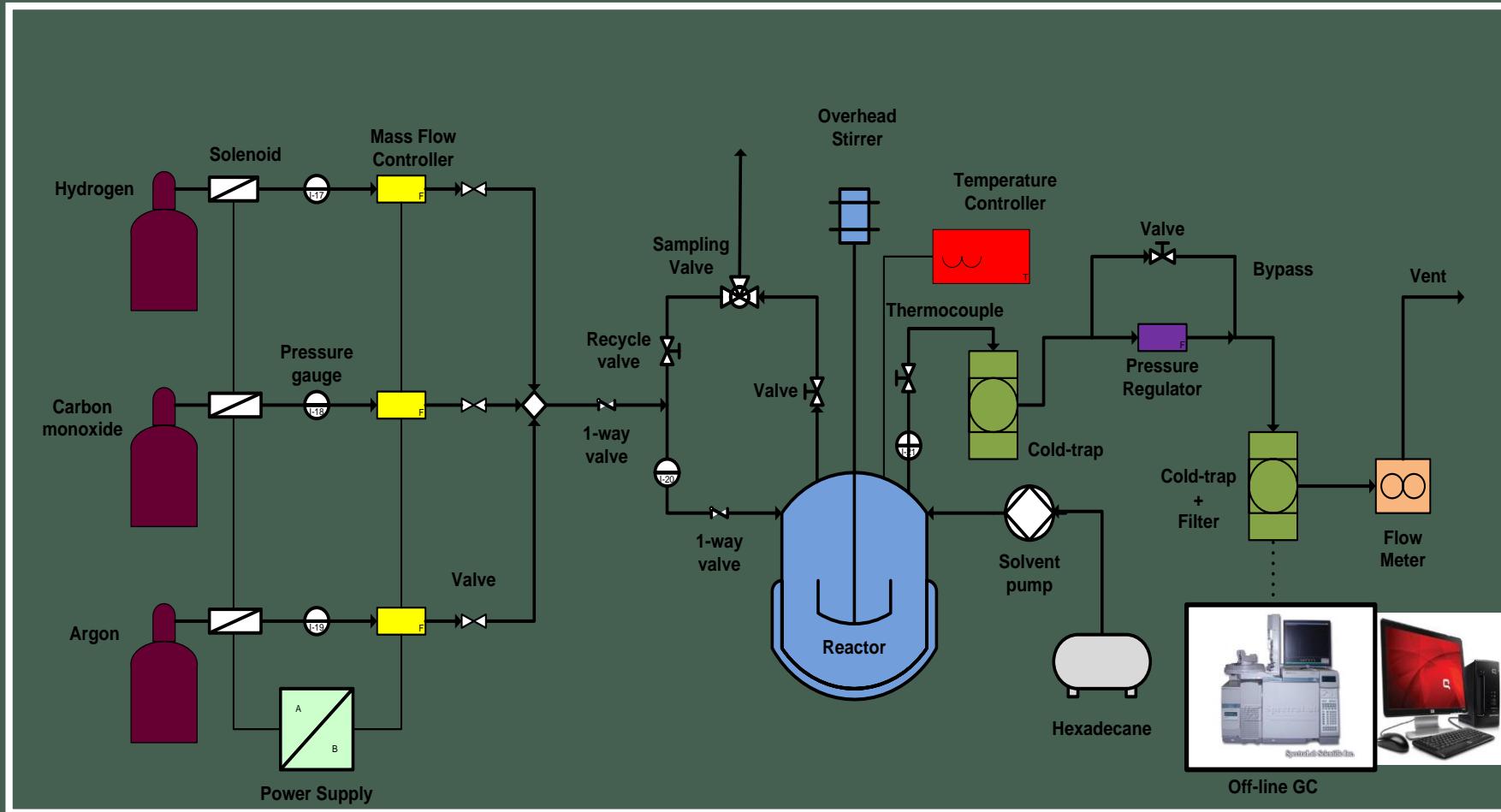
Main reactions	
1. Paraffins	$(2n+1)H_2 + nCO \rightarrow C_nH_{2n+2} + nH_2O$
2. Olefins	$2nH_2 + nCO \rightarrow C_nH_{2n} + nH_2O$
3. WGS	$CO + H_2O \rightleftharpoons CO_2 + H_2$
Secondary reactions	
4. Alcohols	$2nH_2 + nCO \rightarrow C_nH_{2n+2}O + (n-1)H_2O$
5. Boudouard reaction	$2 CO \rightleftharpoons C + CO_2$

II. Catalyst formulation used for Fischer-Tropsch Synthesis





Reactor design for Fischer-Tropsch Synthesis



Process flow diagram of the 3-φ Mahoney-Robinson slurry reactor-system set-up



Basket of the reactor

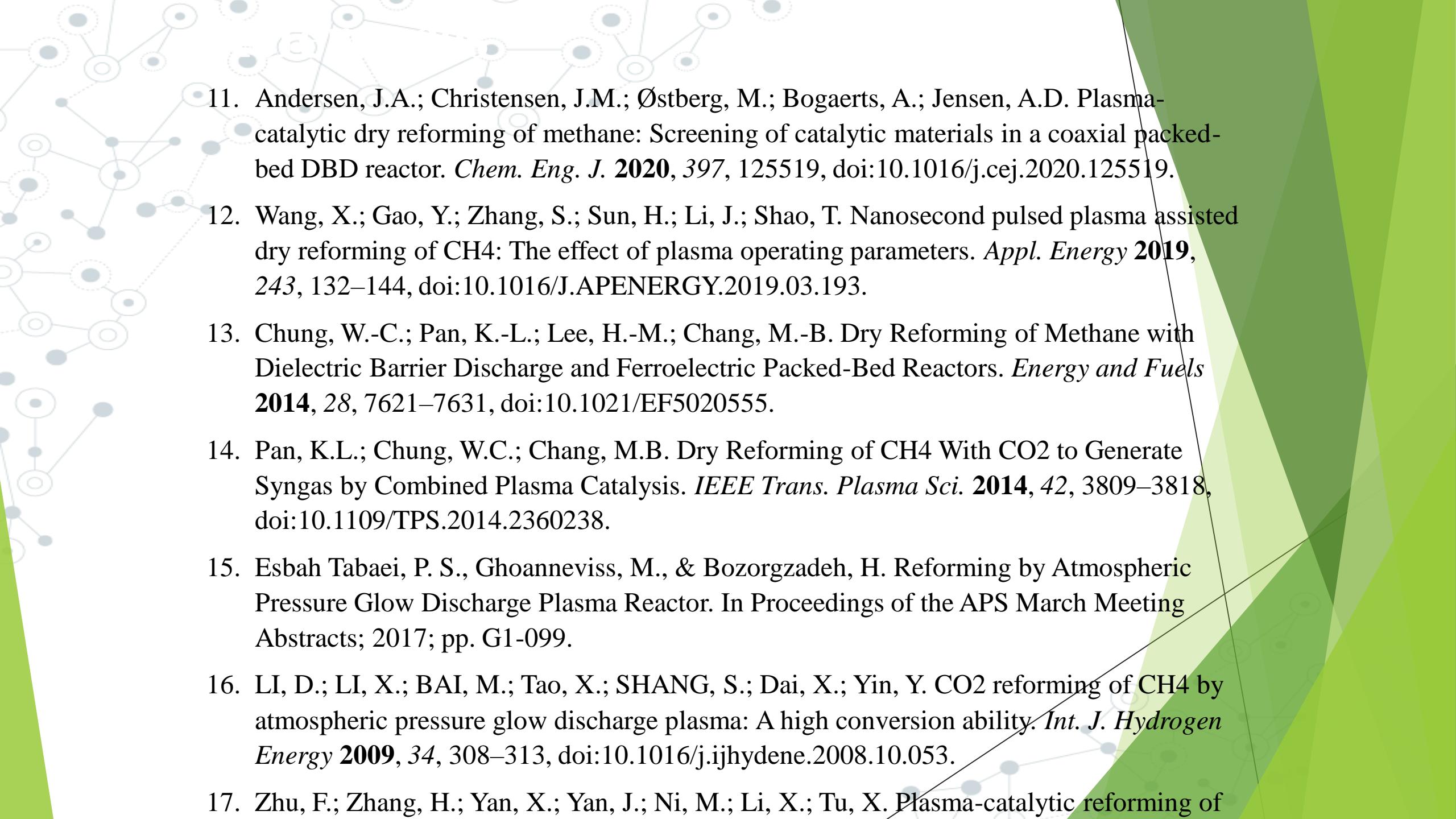


Thanks for listening!



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