

Catalytic Technologies for CO<sub>2</sub> ConversionHydrogenation of CO<sub>2</sub> to Fuels and Chemicals

**KEYWORDS:** Carbon dioxide / Carbon Materials / Catalyst / Composites / Hydrocarbons / Fuels / Environment

Functionalized activated carbon (AC) and carbon nanotubes (CNTs) demonstrated to be high-performing supports for Ni-based CO<sub>2</sub> methanation catalysts, while the formation of a composite of AC and CeO<sub>2</sub> showed excellent performance in this application. Property-performance relationships and reaction mechanisms were established by *ex situ* and *in situ* characterization to understand the importance of carbon materials in the performance of the catalysts. For CO<sub>2</sub> hydrogenation to methanol and the reverse water-gas shift reaction, the best performing catalysts were 15%Cu/CNT-O-R-ZnO (90:10) and Cu/CNT-N-ZnO (90:10), respectively, notably both supported by composites. In the CO<sub>2</sub> hydrogenation to hydrocarbons through Fisher-Tropsch reactions, CNT and Al<sub>2</sub>O<sub>3</sub> are suitable catalytic supports for the reaction under study, but AC is not. The most promising metals impregnated for C<sub>2</sub> - C<sub>4</sub> selectivity were Fe, Co and Cu, whereas CO<sub>2</sub> conversions are higher over catalysts containing Na.

## Introduction

Since the Industrial Revolution, energy has played a fundamental role in social and economic development. The use of energy sources has grown exponentially, which increases the amounts of fuel used and, consequently, the release of carbon dioxide (CO<sub>2</sub>) into the atmosphere. In line with the 13th goal for sustainable development "Climate Action", to limit warming to 1.5 °C above pre-industrial levels, global greenhouse gas emissions need to reach zero by 2050; therefore, the use of fossil fuels must be eliminated.

CO<sub>2</sub> methanation reaction has appeared as an interesting option since it is the most thermodynamically favourable reaction of CO<sub>2</sub> hydrogenation and is a promising way to convert anthropogenic CO<sub>2</sub> back to CH<sub>4</sub> fuel. The hydrogenation of CO<sub>2</sub> to methanol (MeOH) is an interesting research field since methanol is a liquid that is capable of becoming an alternative to fossil fuels. MeOH is also easier to transport because it is liquid at room temperature, and when used as fuel, it does not release harmful gases. The reverse water-gas shift (RWGS) reaction has also drawn interest from researchers. This reaction enables the conversion of CO<sub>2</sub> to CO, an intermediary in CO<sub>2</sub> hydrogenation reactions. Since CO is more reactive than CO<sub>2</sub>, processes such as methanol synthesis, Fischer-Tropsch synthesis (FTS) or Cativa acetic acid synthesis still use syngas as feedstock.

The hydrogenation of CO<sub>2</sub> to C<sub>2+</sub> products is more challenging due to the extreme inertness of CO<sub>2</sub>, the high C - C coupling barrier, and the occurrence of competing reactions that lead to the formation of C<sub>1</sub> products. However, the study of new catalytic processes to produce C<sub>2+</sub> hydrocarbons has become more attractive since these compounds have higher economic values than C<sub>1</sub> compounds. The direct FTS reaction has been shown to be an effective route for this type of reaction due to its ease of operation and the obtaining of promising amounts of C<sub>2+</sub> products. This process integrates the reduction of CO<sub>2</sub> to CO through the RWGS reaction, followed by the hydrogenation of CO to hydrocarbons via FTS. In this synthesis, it is necessary to develop catalysts with compatible bifunctional active sites for the 2 reaction steps under the same operating conditions.

## Current Development

CO<sub>2</sub> Methanation

Different carbon materials were prepared and their performance as CO<sub>2</sub> methanation catalyst supports were evaluated in different sets of experiments [1,2,3,4]. All of the performed works on this topic were developed in collaboration with the International Iberian Nanotechnology Laboratory (INL).

It has been demonstrated that different surface chemistries of activated carbon (AC) can have an influence on its performance as support for Ni-based CO<sub>2</sub> methanation catalysts. Reduced activated carbon (AC-R) with O-free Lewis basic sites showed the best performance with a CO<sub>2</sub> conversion of 76% and selectivity to methane of 97%, at 450 °C [1].

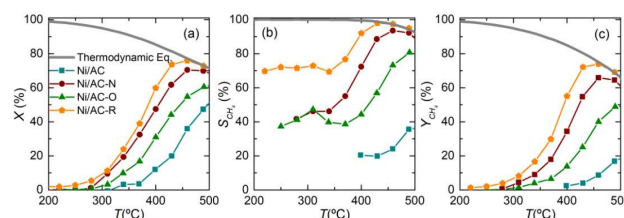
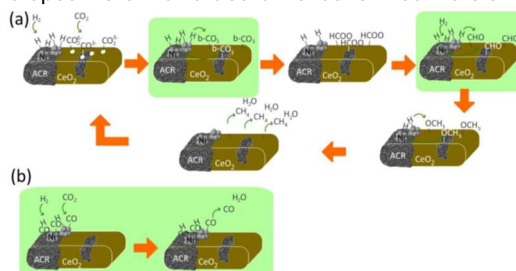


Fig 1. Comparison of the catalytic properties of the synthesized Ni catalysts supported on ACs with different surface functionalities: XCO<sub>2</sub> (a), SCH<sub>4</sub> (b) and YCH<sub>4</sub> (c) as a function of temperature, together with the respective thermodynamic equilibrium curves. Reaction conditions: P = 1 bar; WHSV = 60 000 mL g<sup>-1</sup> h<sup>-1</sup>; CO<sub>2</sub> : H<sub>2</sub> = 1 : 4 [1].

Moreover, it was demonstrated that Ni catalysts on N-doped CNT, could achieve an 81% CO<sub>2</sub> conversion with selectivity to methane of 99% at 400 °C, compared to Ni on non-doped CNT, exhibiting excellent stability over 48 h time-on-stream testing. The study demonstrated that the N-doped CNT support exhibited a better distribution of the Ni sites that were strongly interacting with the support, even with atomically dispersed Ni being observed [2]. This project was performed also in collaboration with the Ernst Ruska Centre for Microscopy and Spectroscopy with Electrons, in Jülich, Germany.

A collaborative work with INL, the Technical University of Denmark (DTU) and Laboratoire CRISMAT - CNRS-ENSICAEN in France led to a detailed study of the mechanism of the CO<sub>2</sub> methanation reaction in a CeO<sub>2</sub>-AC supported Ni catalyst, using the ME-PSD-DRIFTS technique [3]. The catalyst developed herein afforded an excellent conversion of around



87% with almost complete selectivity to methane and excellent stability Fig 2. Proposed mechanism for the CO<sub>2</sub> methanation over Ni/ARC-CeO<sub>2</sub> catalyst, displaying the formation/transformation of the reaction intermediates as elucidated by *in situ* ME-PSD-DRIFTS. (a) illustrates the mechanism of CH<sub>4</sub> formation while (b) depicts the mechanism of CO formation [3].

at a favourably low reaction temperature of 370 °C. The composite of carbon with CeO<sub>2</sub> in the supporting material allowed for an efficient reduction in the usage of the expensive rare-earth CeO<sub>2</sub> while affording not only a cost-effective but also a high-performing methanation catalyst. The mechanistic study determined the rate of formation/consumption of the various intermediates, demonstrating that the CO<sub>2</sub> methanation reaction follows both CO and formate pathways in Ni/CeO<sub>2</sub>, while in Ni/ACR–CeO<sub>2</sub>, it only follows the formate pathway (**Error! Reference source not found.**).

Importantly, it was found that a partial modification in the supporting material of the catalysts can lead to different mechanisms. A detailed ex situ TEM study demonstrated the modifications in the catalyst after reaction (Figure 3).

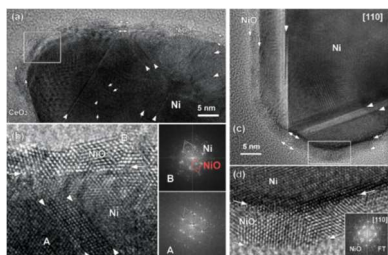


Figure 3. HRTEM imaging of the surface of a single Ni nanoparticle in the as-synthesized Ni/ACR–CeO<sub>2</sub> catalyst (a) and zoom-in (white rectangle) image (b) and in the same catalyst after its TOS testing for 90 h (c,d), revealing the core-shell appearance of the nanoparticles. The insets in (b,d) show the FT patterns from the corresponding regions. The white arrows mark the NiO shell and the white arrow heads depict the twinning within the Ni nanoparticles [3].

#### CO<sub>2</sub> Hydrogenation to Methanol

Preliminary catalytic experiments were conducted for this reaction with Cu-based catalysts supported on pristine AC and CNT. This was followed by an assessment of the addition of ZnO to the catalytic system using carbon materials (CM):ZnO composites as supporting materials, and by an evaluation of the functionalization of carbon material's surface [\*].

The catalytic experiments were conducted at 300 °C and 25 bar. The catalysts supported on composites recorded an increase in CO<sub>2</sub> conversion and MeOH formation, confirming the benefit of adding ZnO to the catalyst structure. For the catalysts with functionalized CM, CNT-O-R resulted in a higher CO<sub>2</sub> conversion, while CNT-O-600 provided a higher MeOH formation. Both supports present a small amount of O-groups, which may have led to a better CO<sub>2</sub> adsorption and active phase dispersion. The best performing catalyst was 15%Cu/CNT-O-R-ZnO (90:10), with a maximum CO<sub>2</sub> conversion of 18.2% and MeOH space time yield of 1.75×10<sup>-1</sup> g<sub>CH<sub>3</sub>OH</sub> g<sub>cat</sub><sup>-1</sup> min<sup>-1</sup> [\*].

#### Reverse Water-Gas Shift Reaction

The addition of ZnO to a Cu-based catalyst supported on pristine CNT was assessed, via preparation of new supporting materials based on CNT:ZnO composites. The impact of functionalizing the carbon nanotube's surface on the catalysts' performance was also assessed. The catalytic performance was improved by the usage of a N-doped CNT:ZnO composite as supporting material, Cu/CNT-N-ZnO (90:10), with a CO<sub>2</sub> conversion of 54.8%, and CO selectivity of 100%, at 600 °C and 1 bar. CO<sub>2</sub>'s adsorption and catalytic performance were likely improved by this treatment. It was concluded that the catalytic performance of the RWGS reaction may benefit from combining the properties of CNT materials and metal oxides on composites, making this approach an interesting toolbox for developing highly efficient catalysts for this reaction [5].

#### CO<sub>2</sub> Hydrogenation to Hydrocarbons

The performance of carbon materials aluminium oxides as catalytic supports for the CO<sub>2</sub> hydrogenation reaction was assessed at 350 °C, 20 bar, GHSV = 3500 h<sup>-1</sup> and CO<sub>2</sub> : H<sub>2</sub> (V : V)

= 1 : 3. The CO<sub>2</sub> conversion for catalysts supported on CNT and Al<sub>2</sub>O<sub>3</sub> and impregnated with Fe varied between 33 and 47%, and between 85 and 98% materials with Na. On the other hand, selectivity to hydrocarbons ranged between 4.5 and 20% and between 0.98 and 1.7% in Fe and Na-based catalysts, respectively. Catalysts prepared with Fe show much lower conversions than catalysts prepared with Na, but the same trend does not occur for selectivity to C<sub>2</sub>-C<sub>4</sub> products since these are practically null in Na-based catalysts.

In general, in catalysts prepared with the same combination of metals, it was found that catalysts supported on the material with the highest specific surface area, AC, demonstrated the lowest CO<sub>2</sub> conversion values and practically zero selectivity to hydrocarbons. On the other hand, the results obtained with catalysts supported on CNT and Al<sub>2</sub>O<sub>3</sub> demonstrated much higher CO<sub>2</sub> conversions and selectivity to hydrocarbons from C<sub>2</sub> to C<sub>4</sub>. Unlike AC, these materials exhibit the absence of micropores, being essentially mesoporous, which allows diffusion limitations to be lower and, consequently, better results in both conversion and selectivity to be obtained.

#### Future Work

The prospective tasks defined for the reaction of CO<sub>2</sub> hydrogenation to methanol consist of preparing bimetallic catalysts supported on composites of CM and metal oxides, such as Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, CeO<sub>2</sub> and In<sub>2</sub>O<sub>3</sub>, as these metal oxides have been reported in the scientific community as beneficial for CO<sub>2</sub> adsorption, and therefore, may improve the catalytic performance. Another objective consists in the optimization of the reaction conditions by conducting a parametric study. As the reaction mechanism is still not consensual, it will also be studied through in-situ characterization techniques.

For the FTS reaction, carbon materials will be modified through physical and chemical treatments and different metals, namely Ru, Pd and Pt, will be impregnated into the commercial and modified supports. The reaction conditions will be evaluated to find optimal operating conditions that maximize results. It is intended to define the reaction mechanism, develop advanced multifunctional catalysts, and evaluate the sustainability of the process under real conditions.

#### Related Sustainable Development Goals



#### Outputs

##### PhD Theses

Liliana P. L. Gonçalves, CO<sub>2</sub> Methanation over Bimetallic Catalysts supported on Carbon Materials with tailored surface properties, PDEQB, FEUP, 2022

##### Master Dissertations

[\*] Ana Rita Querido, Catalysts for CO<sub>2</sub> Hydrogenation to Methanol, MEQ, FEUP, 2022

Inês Ferreira Quatorze, Metanação de CO<sub>2</sub> para ciclo neutro de carbono, MEA, FEUP, 2022

##### Selected Publications

- [1] L. P. L. Gonçalves et al., Catal.Sci.Technol., 10 (21), 7217 (2020).
- [2] L. P. L. Gonçalves et al., Carbon, 195, 35 (2022).
- [3] L. P. L. Gonçalves et al., Appl. Catal. B Environ., 312, 121376 (2022).
- [4] L. P. L. Gonçalves et al., ChemCatChem, 13 (22), 4770 (2021)
- [5] A. R. Querido et al., Catal.Sci.Technol., 13, 3606 (2023)

##### Team

Manuel F. R. Pereira, Full Professor; **Olívia S. G. P. Soares**, Auxiliar Researcher. Ana Rita Querido, Project Researcher, **Mariana B. S. Felgueiras**, PhD Student;; **Liliana P. L. Gonçalves**, former PhD Student;

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## Catalytic Technologies for CO<sub>2</sub> Conversion

### Multifunctional Catalysts for Synthetic Fuels Production

**KEYWORDS:** Fischer Tropsch / Carbon Materials / Cobalt Catalysts / Synthetic Fuels

**The ability to customize both the porous architecture and surface chemistry of carbon materials, not only during initial preparation but also through subsequent modifications, facilitates catalyst engineering for enhanced performance in Fischer–Tropsch synthesis. This enables heightened selectivity in the generation of targeted hydrocarbon groups, more specifically, the liquid range (C<sub>5-20</sub>).**

#### Introduction

Gas-to-Liquids (GtL) processes that convert natural gas or other gaseous hydrocarbons into longer-chain liquid hydrocarbons (e.g., jet fuel, gasoline or diesel) will be, in the near future, a decisive alternative to oil refining. These synthetic fuels can be processed into fuels of superior quality compared to those obtained by oil refining, leading to significant reductions in emissions of particles, NO<sub>x</sub>, SO<sub>x</sub>, and aromatics to levels lower than those found in oil-based fuels. These synthetic fuels are considered environmentally friendlier than fossil alternatives, and their production contributes to the carbon circular economy.

The Fischer-Tropsch (FT) reaction is at the core of the GtL process and is an alternative for transforming non-petroleum carbon resources into valuable chemicals, playing an essential role in producing sustainable and clean liquid fuels. However, the FT reaction still faces today's efficiency challenges, and the performance of FT is critical. It is a reaction that is generally performed in large-scale industrial GtL units using conventional fixed- or fluidized-bed reactors.

The primary motivation is to make an outstanding contribution to achieving more efficient GtL processes and developing new global technologies of national origin to promote the evolution of the future energy mix in a sustainable global environment. This study presents the exploration and advancement of an innovative catalytic solution (catalyst development) aimed at the continuous production of premium fuels. The objective is to surpass the efficiency of existing methods employed in conventional GtL plants. Significant innovation is anticipated through a creative design approach, enabling thorough examination and optimization of the impact of engineered multifunctional materials.

The selection of the support for the catalyst is critical as it helps to disperse the active metal phase, and metal-support interactions have a profound impact on the resulting performance of the catalyst. Through precise customization of the catalyst formulation, there is a resultant enhancement in activity, directly influencing operational parameters such as reduced reaction temperatures and pressures. This ultimately leads to energy savings, a significant cost factor in large-scale chemical processes. The most used supports in FT include metal oxides and carbon-based materials. Carbon-based materials possess the advantage of good thermal conductivity, high specific surface area, and high thermal and chemical stability under middle operation conditions. Furthermore, these materials allow for modification during synthesis or through subsequent treatments. The idea of chemically modifying the surface of these supports, creating multifunctional catalysts, attracts increasing attention because by changing the surface of the

support, we can influence other FT parameters, and with the proper catalyst formulation, it may be possible to selectively direct the reaction towards the desired products, liquid hydrocarbons (C<sub>5</sub>-C<sub>20</sub>), while reducing the number of reactants wasted, forming unwanted by-products.

Furthermore, the use of different reaction media and reactor types can further influence the performance of the catalysts, opening possibilities for industrial and large-scale applications.

#### Current Development

This work has been carried out under a collaboration with Prof. Helder T. Gomes (IPB – Polytechnic Institute of Bragança, Portugal). Various multifunctional catalysts for use in the FT reaction were developed. The research was centred on the production and modification of the catalyst supports through diverse treatments. The materials exhibited characteristics that endowed them with the ability to function as catalysts, predominantly producing the liquid range of hydrocarbons with minimal generation of CO<sub>2</sub> and waxes. The focus of the study was on tailoring the catalyst supports to enhance their catalytic properties and optimize the desired product spectrum. The resulting catalysts demonstrated remarkable selectivity and stability over extended reaction periods, making them promising candidates for efficient and sustainable FT synthesis.

Various carbon materials with diverse treatments have been tested as supports for the catalyst with cobalt as the active metal (with/out the presence of rhenium as a promoter). The impregnation of the support with the cobalt was accomplished through incipient wetness impregnation (IWI). The produced catalysts were evaluated in FT reaction for synthetic fuel production, with a focus on the production of the liquid range of hydrocarbons, specifically Jet Fuel (C<sub>12-15</sub>), while minimizing the production of gases (C<sub>1-4</sub>) and waxes. Before being tested in the FT reaction, each catalyst was reduced to the appropriate temperature, as determined through characterization. The reactions were conducted using a fixed-bed reactor (Microactivity Effi, MAE, from PID

Table 1. Product selectivities for the produced catalysts

Catalyst	Selectivity (%)					
	CO <sub>2</sub>	CH <sub>4</sub>	C <sub>2</sub> -C <sub>4</sub>	C <sub>5</sub> -C <sub>11</sub>	C <sub>12</sub> -C <sub>20</sub>	C <sub>21+</sub>
Co/CNTF1	<1	9.5	2.3	42.0	43.2	2.4
Co/CNTF2	<1	11.2	2.4	40.2	42.7	3.0
Co/N-CNTF3	<1	8.0	2.1	37.3	46.6	5.6
Co/N-CNTF4	<1	9.3	2.5	41.4	41.5	4.7
Co/CNTFcalc350	<1	6.2	1.6	38.2	50.5	2.9
Co/CNTFcalc550	<1	5.9	2.1	44.2	45.5	1.6
Co/CNTFcalc750	<1	5.4	2.5	55.4	34.4	1.7
Co/CNTFcalc900	<1	1.7	1.1	36.6	55.3	4.8
Co/CNF	<1	3.5	1.6	40.3	48.5	5.8
Co/FLGF	<1	2.3	1.4	34.9	55.9	5.3

Eng&Tech) in collaboration with CoLAB Net4CO<sub>2</sub>. The reactor was fed with a synthesis gas composed of carbon monoxide, hydrogen and nitrogen with a Gas Hourly Space Velocity (GHSV) of 4 m<sup>3</sup>·kg<sub>cat</sub><sup>-1</sup>·h<sup>-1</sup> at 220 °C and 20 bar. The standard

reactions were conducted for a duration of 28 hours. The catalytic tests were carried out using the best operating conditions for fixed-bed reactors, which were identified after a detailed parametric study where the influence of pressure, temperature and GHSV was successfully evaluated. The gaseous products were examined using an online GC (YL6500, YL instrument CO. LTD), fitted with two columns and a TCD, using Helium as the carrier gas during the course of the reaction. The liquid products obtained were collected after the reaction had concluded and were analyzed using an offline GC equipped with a Supelcowax 10 capillary GC column with a flame ionization detector (FID) and using Helium as the carrier gas. The catalytic tests performed served as a basis for the optimization of the catalysts that were produced, as well as for the scale-up of the process using different reaction media and reactor configurations.

Efforts were also made to develop catalysts with dual functionalities with carbon materials. Various carbon materials were employed, including nanotubes (CNT), nanofibers (CNF), and graphene (FLG). CNF and FLG underwent only acid functionalization, whereas CNTs underwent chemical and/or thermal treatments. The functionalization (acidic and thermal) of the supports was employed to incorporate different groups onto the support surface, aiming to enhance performance. In this study, it was shown that the performance of carbon-supported cobalt catalysts for FT can be dictated by the modifications in the surface chemistry. The supports that underwent functionalization had outstanding performances compared to their pristine counterparts. All the produced carbon-supported catalysts showed excellent selectivity for the desired range.

The catalysts produced with the new supports proved to be efficient in producing the desired range ( $C_{5-20}$ ) while generating a reduced amount of gaseous hydrocarbons ( $C_{2-4}$ ) and waxes ( $C_{21+}$ ), as shown in Table 1. These results establish a potential future direction for the production of synthetic fuels.

In addition to the good selectivity exhibited by the catalysts, another observed characteristic in the produced catalysts is that they displayed excellent stability during long-duration reactions. To assess stability, the catalysts were subjected to a long-duration reaction lasting 100 hours in a fixed-bed reactor using the optimal conditions. They showed no variation in product distribution throughout the reaction. This stability in performance and selectivity is noteworthy for potential applications on an industrial scale.

Supports and catalysts produced were also characterized using various techniques such as  $NH_3$  temperature-programmed desorption, thermogravimetric analysis and differential scanning calorimetry, inductively coupled plasma optical emission spectroscopy,  $H_2$  temperature-programmed reduction, transmission electron microscopy (TEM) and scanning transmission electronic microscopy. Figure 1 shows the transmission electron microscopy (TEM) of two of the produced catalysts. This analysis was conducted to determine cobalt particle size distribution and the morphology of catalysts,

It can be seen from the images that the cobalt was successfully impregnated in the support, and most of the Co particles were confined inside the CNTs when these were functionalized with acid, with sizes ranging between 3-5 nm.

## Future Work

Further investigation is required to thoroughly examine the impacts of various modifications and catalyst designs, particularly concerning the application of modified catalysts in various reaction media, such as biphasic systems, as this

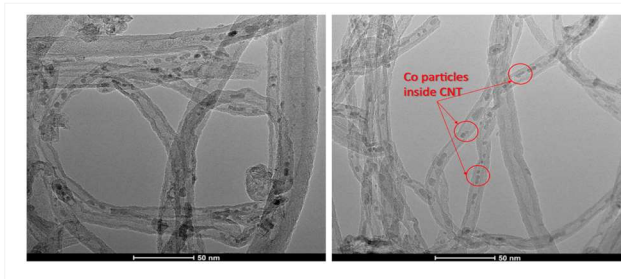


Fig 1. Transmission electron microscopy of the produced Co/CNTF catalyst.

A: CoRe/Purat; B: Co/CNTF1.

is also a parameter that can influence reaction outcomes and can further enhance selectivity for the desired range of hydrocarbons.

Once the best catalysts have been identified, the next step will be their application in a new type of reactor, the NetMIX technology. The FT reaction is severely constrained by the low interfacial areas achieved with currently used industry-standard reactors and the reduced heat/mass transfer coefficients, resulting in poor catalyst performance. The application of the NetMIX reactor technology initially developed at the University of Porto will help overcome these limitations. This technology is based on a unique micro/mesoreactor topology formed by a network of interconnected static mixing chambers through transport channels, enabling optimized and reproducible fluid mixing. The additional oscillatory solid dynamics of the reactive flows increase micromixing capacity, potentially leading to a continuous sweeping of the catalyst surface, minimizing the presence of stagnant zones and mitigating potential deactivation. Using this modular technology will result in net improvements directly impacting the operating conditions, such as lowering the temperature and pressure at which the FT reaction occurs. These results will affect the chain of value in fuel production, with significant savings towards consumer consumption at various levels while promoting decarbonization strategies. In addition, this technology offers ease of scale-up and opens doors to potential industrial applications.

## Related Sustainable Development Goals



## Outputs

### PhD Theses

Thais Sayuri Berberich, Multifunctional catalysts for improved Fischer-Tropsch synthesis using NetMIX-based microreactor technology towards the competitive production of synthetic fuels, PDEQB/PDEA, FEUP, ongoing

### Master Dissertations

David Faria Aguillar, Emerging catalytic systems for the Sustainable production of synthetic Fuels, MIEQ/MIEA, FEUP, 2023

### Team

Joaquim Faria, Professor/Group Leader/Principal Researcher; Thais Berberich, PhD Student; David Aguillar, Master Student; Helder

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