

## FROM LABORATORY TO SEMI-PILOT SCALE: FISCHER-TROPSCH SYNTHESIS OVER A Na-Fe<sub>3</sub>O<sub>4</sub> CATALYST FOR E-FUEL APPLICATIONS

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### Abstract

The net-zero commitments adopted by many countries require decisive steps to reduce reliance on fossil fuels. While electrification offers a pathway for several sectors, “hard-to-abate” areas such as aviation, international shipping, and heavy industry will continue to rely on liquid fuels, which must be produced from renewable or low-carbon sources. Among the emerging solutions, e-fuels produced from green hydrogen and captured carbon dioxide are considered a promising alternative.

At the Energy Innovation Test Station of the University of Szeged, renewable energy is used to generate hydrogen, which is subsequently converted into synthetic fuels via Fischer-Tropsch Synthesis (FTS). In this context, we scaled up a 1 wt% Na-Fe<sub>3</sub>O<sub>4</sub> catalyst, shown to operate well at the laboratory scale, and tested it under semi-pilot conditions more representative of industrial implementation. The catalyst showed high activity, selectivity toward C<sub>5+</sub> hydrocarbons, and stable performance across multiple regeneration cycles, confirming both its scalability and sustainability for future e-fuel production.

### Introduction

Global demand for aviation fuels continues to rise in line with air travel growth. According to the International Energy Outlook 2023, jet-fuel consumption is projected to increase significantly even as demand for gasoline declines [1]. At the same time, aviation sector accounted for approximately 2.5 % of global CO<sub>2</sub> emissions in 2023, highlighting its non-negligible contribution to climate change [2]. Today, most jet fuels are derived from crude oil, and the corresponding CO<sub>2</sub> emissions greatly contribute to the global carbon footprint. Given the urgency of limiting greenhouse gas emissions and the finite nature of fossil reserves, there is a growing imperative to explore sustainable, low-carbon alternatives. While road transportation can gradually shift toward electrification or biofuels, aviation faces stricter constraints: jet fuels must meet precise compositional and performance standards (e.g. Jet A-1), which limits the feasible substitution pathways.

FTS is a promising gas-to-liquid (GTL) technology for producing synthetic hydrocarbon fuels via syngas (CO + H<sub>2</sub>) conversion. When combined with a downstream reverse water gas shift (RWGS) reaction to generate CO, and supplied with green hydrogen, FTS can function as a carbon-recycling platform. In this configuration, synthetic fuel production is effectively decoupled from fossil feedstocks, offering a viable route to reduce the net carbon footprint of the transport and energy sectors.

Fe and Co catalysts are most commonly used in industrial FTS. Among Fe catalysts, Na-promoted Fe<sub>3</sub>O<sub>4</sub> (Na-Fe<sub>3</sub>O<sub>4</sub>) has been extensively studied: the alkali promotion enhances CO dissociation preserving the carbide phase which is active for both FTS and water-gas-shift (WGS) reactions, thus increasing activity and selectivity toward longer-chain hydrocarbons [3]. However, most performance studies of the Na-Fe<sub>3</sub>O<sub>4</sub> have been confined to lab scale; scaling such catalysts toward industrially relevant scales remains a critical challenge.

Within the framework of the RRF-2.3.1-21-2022-00009 project *National Laboratory for Renewable Energy*, supported by the EU Recovery and Resilience Facility, we successfully scaled up a 1 wt % Na-Fe<sub>3</sub>O<sub>4</sub> catalyst by a factor of 20 and tested it at semi-pilot plant level (TRL 4). The scaled-up experiments were conducted in a five-reactor test station capable of producing 1-2 kg of liquid hydrocarbons per day, using hydrogen supplied from solar-powered electrolysis.

## Experimental

### *Catalyst preparation*

Pure Fe<sub>3</sub>O<sub>4</sub> catalysts were synthesized by the coprecipitation method [4]. The iron salts were dissolved in dilute HCl, and precipitation was carried out by adding aqueous NH<sub>4</sub>OH under vigorous stirring until alkaline conditions were reached. The suspension was aged at elevated temperature, then the solid was separated, washed, and dried. Na-promoted catalysts (1 wt%) were obtained by the wet impregnation using an aqueous NaNO<sub>3</sub> solution, followed by sonication, solvent removal, drying, and calcination under inert atmosphere. The product was pressed, crushed, and sieved to the desired particle size for testing. For the larger-scale (90 g) batch, the same procedure was applied with adjusted reagent amounts, and the pellet size was increased accordingly for up-scale experiments.

### *Characterization*

XRD studies of the samples were performed on a Rigaku MiniFlex II instrument with a Ni-filtered CuK<sub>α</sub> source in the range of  $2\theta = 20\text{--}100^\circ$ .

The specific surface area (BET method) was determined by the BJH method using a Quantachrome NOVA 2200 gas sorption analyzer by N<sub>2</sub> gas adsorption/desorption at 77 K. Before the measurements, the samples were stored in vacuum (<0.1 mbar) at 473 K for 2 hours. To assess the Na content of the prepared catalysts, a Thermo Scientific Apreo 2 scanning electron microscope was used. Prior to the study, the samples were mounted on electrically conductive carbon tape, and then a thin layer of gold was evaporated onto their surface to prevent charge accumulation due to electron irradiation.

### *Catalytic activity tests*

The catalysts were tested at two different scales: laboratory scale (TRL1) and semi-pilot plant scale (TRL4). The Fischer-Tropsch reactions were performed in a steel, fixed-bed, continuous-flow reactor. Prior to each reaction, the catalysts were first heated to 400 °C under an inert atmosphere and subsequently pretreated in pure hydrogen for 16 h. After the pretreatment, the reactors were cooled to 300 °C, and the feed gas mixture consisting of CO and H<sub>2</sub> was introduced under the following common conditions: pressure 30 bar, temperature 300 °C, H<sub>2</sub>/CO ratio of 2, and a gas hourly space velocity (GHSV) of 2700 mL g<sup>-1</sup> h<sup>-1</sup>.

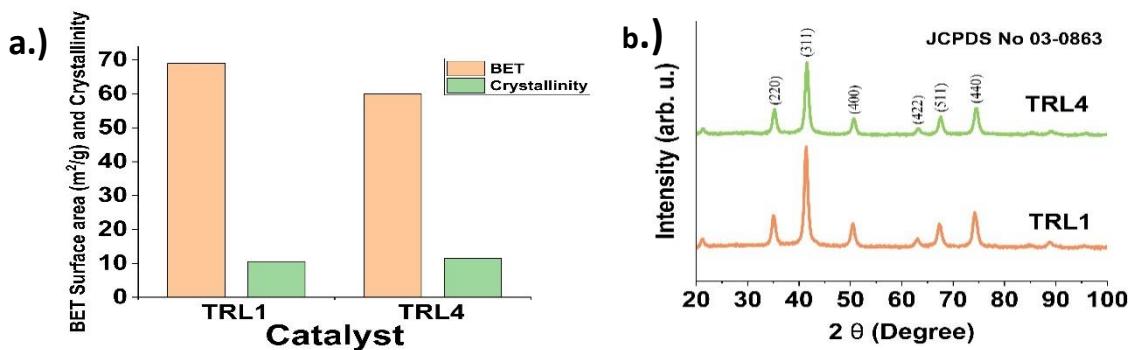
At the TRL1, the reactor parameters were as following: outer diameter was 2 cm and length was 36 cm. The temperature of the reactor was maintained by a digital temperature controller and an electric heating stove. A total of 2 g of catalyst was loaded into the reactor, positioned between quartz wool plugs, while the remaining void space was filled with quartz beads. The reaction time was 5 h.

At the TRL4, the reactor had a length of 115 cm and an outer diameter of 3.5 cm, equipped with five heating zones and monitored by six external and six internal thermocouples. A total of 70 g of catalyst mixed with SiC in different ratios was loaded into the reactor to account for the heat dissipation. The reaction was carried out for 76 h under otherwise identical conditions.

## Results and discussion

Both the small-scale (TRL1) and the up-scaled (TRL4) Na-Fe<sub>3</sub>O<sub>4</sub> catalyst batches were successfully synthesized and characterized by XRD, BET, and SEM-EDS measurements. The up-scaled sample preserved the structural and textural properties of the small batch, confirming the scalability of the synthesis method (**Fig. 1a**).

XRD patterns showed only reflections corresponding to Fe<sub>3</sub>O<sub>4</sub>, indicating phase purity and the absence of undesirable crystalline by-products (**Fig. 1b**). SEM-EDS mapping revealed a homogeneous distribution of Na across the catalyst surface, with a loading close to the nominal 1 wt% in both cases (*data not shown*).



**Figure 1.** Structural and textural properties of TRL1 and TRL4 batches of Na-Fe<sub>3</sub>O<sub>4</sub> catalyst: a) BET surface area and crystallinity (calculated from XRD); b) XRD patterns of freshly-prepared samples

The FTS catalytic activity of both batches was assessed under identical reaction conditions with 2 g of catalyst and 5 h reaction time at the lab-scale and 70 g of catalyst and 76 h reaction time at the up-scale.

In both cases the CO conversion was very high (> 90%) indicative of the catalyst efficiency. In the up-scaled system, the concentration of undesired gases (CO<sub>2</sub> and CH<sub>4</sub>) was slightly lower, which can be attributed to the stabilization of the active iron carbide phase during long-term operation and possibly more uniform Na promoter effect.

The collected in a cold trap liquid hydrocarbon product was analyzed offline with the GC-MS (**Fig. 2**). In contrast to the lab-scale, the TRL4 batch produced predominantly linear-chain hydrocarbons dominated by olefins. The longer reaction time and the larger catalyst bed most probably promote steady-state operation and favor chain-growth mechanisms over secondary transformations, which can account for the observed differences. Due to the presence of Na, the catalyst surface was rich in C\* species and poor in H\* species. The lower surface ratio of H\*/C\* is also favorable for the growing chain termination via  $\beta$ -hydride abstraction rather than hydrogenation, which explains high selectivity towards olefins. [5]

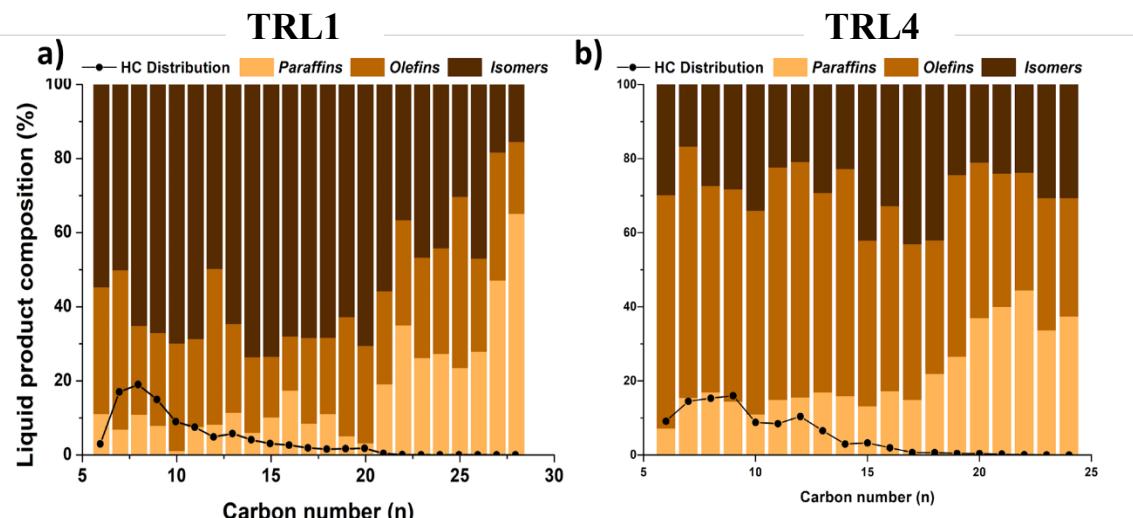


Figure 2. Hydrocarbon distribution for a) TRL1 batch; b) TRL4 batch

After verifying the proof of concept at TRL 4, the long-term stability and reusability of the  $\text{Na-Fe}_3\text{O}_4$  catalyst were also investigated. Following the initial run (76 h), the catalyst was regenerated by reactivation in  $\text{H}_2$  (16 h at 400 °C) and subjected to a second catalytic cycle (74 h). The procedure was repeated once more for a third run (68 h).

The space-time yield (STY) of collected liquid hydrocarbons, expressed as grams of product per gram of catalyst per hour, was determined after each cycle to directly assess and compare the catalyst performance. The calculated values were 148, 129, and 132  $\text{mg g}^{-1}_{\text{cat}} \text{h}^{-1}$  for runs I-III, respectively.

## Conclusion

Overall, the catalytic results demonstrate that up-scaling the  $\text{Na-Fe}_3\text{O}_4$  catalyst from TRL1 to TRL4 preserved both high activity and  $\text{C}_{5+}$  product selectivity. Moreover, the catalyst exhibited stable efficiency over three regeneration cycles, during which a total of ~1 L of liquid hydrocarbons was collected. These findings underline the scalability and sustainability of the system. Future work will focus on optimizing reaction parameters and catalyst loading, and on further scale-up toward production capacities of 5 L and 20 L.

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