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Feasibility study on the use of MIL-53(Al) as a support for iron catalysts in the CO hydrogenation reaction

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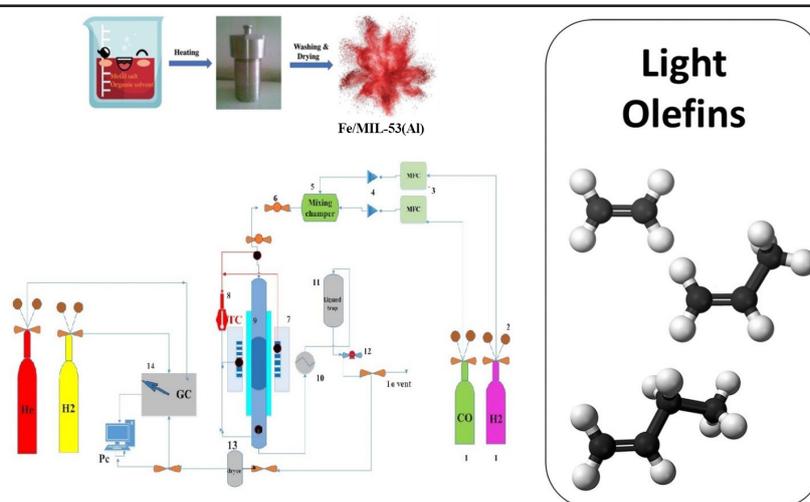
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HIGHLIGHTS

- A novel Fe/MIL-53(Al) Fischer-Tropsch catalyst was synthesized using a solvothermal method.
- MIL-53(Al) was found to be a proper metal-organic framework for use as a support of iron catalysts in high temperature and low pressure.
- Light olefins (up to 24%) were obtained during the Fischer-Tropsch synthesis over 0.5 g of Fe/MIL-53(Al) in a fixed-bed reactor.

GRAPHICAL ABSTRACT



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ABSTRACT

The study examined the potential use of MIL-53(Al), a metal-organic compound created through solvothermal synthesis, as a support for iron catalysts in Fischer-Tropsch Synthesis (FTS). Fischer-Tropsch synthesis is a crucial aspect of Gas-to-Liquid (GTL) technology used in the petrochemical industry to produce light olefins. The catalyst's activity was assessed under specific conditions, including a gas hourly space velocity (GHSV) of 2700 h⁻¹, a hydrogen to carbon monoxide (H₂/CO) feed ratio of 2:1, temperatures ranging from 310 to 330 °C, and pressures ranging from 5 to 9 bar. The feasibility study indicated that MIL-53(Al) has the potential to be a suitable support for iron catalysts in FTS, resulting in the production of light olefins (24%) at high temperatures and low pressure.



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1. Introduction

Porous materials, such as activated carbon, zeolite, porous silicon, metal oxides, and metal-organic compounds, have attracted significant attention due to their wide range of applications [1]. Metal-organic materials, in particular, are known for their crystalline structure, large specific surface area (greater than $10400 \text{ m}^2\cdot\text{g}^{-1}$), spacious pores (9 \AA), and low density ($0.13 \text{ g}\cdot\text{cm}^{-3}$) [2]. Metal-organic frameworks (MOFs) have garnered considerable scientific interest due to their diverse usage in gas absorption [3], purification and storage [4], drug release [5], sensors [6], and catalytic applications [7]. Currently, the exceptional structural stability and presence of regular pores with extensive contact areas make MOFs a popular choice in various research endeavors involving the development of nano-structured catalysts for significant processes [8], particularly the production of clean fuel and hydrocarbons [9] to address pertinent environmental concerns.

MIL-53(Al), which was first studied by Ferri *et al.* in 2002 [10] and later synthesized in 2003 [11], is a well-known MOF with several noteworthy properties. These include its micro-porosity structure, exceptional thermal stability [12], resistance to neutral and acidic organic solutions [13], unique aspiration effect, and adjustable pores [14]. These characteristics contribute to the improved selectivity and catalytic performance of MIL-53(Al). Additionally, MIL-53(Al) offers significant structural benefits and acts as catalyst support in the Fischer-Tropsch synthesis (FTS), leading to enhanced product quality and value [15].

The Fischer-Tropsch synthesis offers a promising approach to producing fuels that are both ultra-clean and environmentally friendly [16]. One crucial aspect of this process is the catalyst, which plays a vital role in enhancing reaction rates and selectivity towards desired products, such as clean fuels and lightweight olefins [15]. The choice of catalyst support is also of great importance, as it directly impacts the selectivity and activity of the catalyst [17,18]. Various options are available for support materials, including refractory oxides like alumina, silicon carbide, zeolite, silica, and metal-organic frameworks (MOFs) [15,19].

In recent years, researchers have conducted numerous studies on the use of MOFs as supports for iron and cobalt catalysts in the Fischer-Tropsch synthesis. Considering the importance of catalyst support in the Fischer-Tropsch synthesis, An *et al.* observed that the interaction between metal and MOF leads to the formation of a $\text{Fe}_3\text{O}_4@x\text{-Fe}_5\text{C}_2$ shell structure, which contributes to the high activity of the FTS [20]. Wezendonk *et al.* utilized various MOFs to synthesize an iron catalyst, varying in porosity, surface area, and thermal stability. The results showed that the iron loading

(36–46 wt%) and nanoparticle size (3.6 and 6.8 nm) of the catalyst were influenced by the porosity of the initial MOFs, resulting in high stability, activity, and reduced CH_4 content [21]. Isaeva *et al.* synthesized Co/MIL-53(Al) using both microwave and solvothermal methods, with the microwave method showing higher activity and selectivity [19]. Comparing the synthesis of Co/MIL-53(Al) catalyst with Co/ $\gamma\text{-Al}_2\text{O}_3$ catalyst, Sun *et al.* observed that Co/MIL-53(Al) exhibited higher activity and selectivity in the production of gasoline and diesel [22]. Isaeva *et al.* reported the production of Co@MIL-53(Al) catalyst, which demonstrated excellent catalytic effects for the production of C_{5+} hydrocarbons in the Fischer-Tropsch synthesis [15].

The unique combination of high specific surface area, tunable pore size, thermal stability [23], enhanced mass transfer, catalyst immobilization capabilities, and versatility [24] make MIL-53(Al) an advantageous support for catalysts in various processes. The potential applications of MIL-53(Al) in FTS for light olefin production are promising. The tunable porosity, thermal stability, and resistance to acidic and moisture-rich environments make MIL-53(Al) an attractive candidate for commercial-scale processes. However, further research and development, along with economic evaluations, are necessary to assess and optimize the commercial viability of this approach. Still, studies on the application of MIL-53(Al) in Fischer-Tropsch synthesis have so far been limited to cobalt catalysts. This study aims to investigate the synthesis of MIL-53(Al) as a support for an iron catalyst under low-pressure conditions, with a focus on its activity in producing valuable lightweight olefins.

2. Experimental

Chemicals obtained from Merck and Aldrich companies were utilized as received without any additional purification steps.

2.1. MIL-53(Al) support synthesis

The solvothermal method is a technique used for the synthesis of materials such as MIL-53(Al). In this method, a reaction mixture containing the desired precursors, solvent, and sometimes a template is sealed in a high-pressure autoclave and heated at an elevated temperature for a certain period of time. The solvent plays a crucial role in the synthesis process as it provides the necessary conditions for the reaction to occur.

During the solvothermal reaction, the precursors dissolve in the solvent, and under the high temperature and pressure conditions, they undergo chemical reactions and form the desired product. In the case of MIL-53(Al), the precursors

typically include aluminum salts and organic linkers. To synthesize MIL-53(Al), a solution containing 2.246 g of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in 10 ml of methanol was prepared. In a separate solution, 0.895 g of terephthalic acid was dissolved in 20 ml of dimethyl formamide (DMF). The two solutions were mixed and subjected to ultrasonication at 40 °C for 15 min to ensure a uniform mixture. The resulting blend was then transferred to an autoclave and heated in an oven at 150 °C for 5 h. Afterward, centrifugation was carried out at 5000 rpm for 30 min to obtain a white gel, which was subsequently washed with DMF, water, and acetone in that order. The obtained product was then dehydrated at 150 °C for 5 h. To activate the MIL-53(Al) metal-organic compound, it was heat-treated at 330 °C for 72 h [25]. Finally, 0.6 g of MIL-53(Al) was obtained.

2.2. Preparing the catalyst

To prepare the catalyst with 10 wt% iron, we used 0.5 g of the support (MIL-53(Al)) and 0.4 g of iron nitrate. The process involved dissolving 0.2 g of iron nitrate in 1.5 ml of ethanol and subsequently adding it to the support using the dry impregnation method. The mixture was then dried at 80 °C for 2 h. The resulting solid was subjected to calcination in an oven at 300 °C for 4 h, followed by a second impregnation stage using a similar procedure.

2.3. Characterization

In the conducted experiments, the synthesis gas was converted into gaseous products (CH_4 , C_2H_4 , C_2H_6 , C_3H_6 , C_3H_8 , C_4H_8 , and C_4H_{10}). This conversion process took place in the presence of a 0.5 g Fe/MIL-53(Al) catalyst. Gas chromatography (GC) was employed to quantify the gaseous products. The GC instrument was the Varian Model 3400 Series equipped with a packed column (Hayesep DB, Altech Company, USA). The column had dimensions of 1/800 OD, was 10 m long, and had a particle mesh size of 100/120. Fig. 1 depicts a schematic representation of the fixed bed microreactor used in this study. The microreactor was constructed using stainless steel with an internal diameter of 20 mm. It contained 0.5 g of catalyst, which was diluted with glass wool in a ratio of 1:4 (v/v).

3. Results and discussion

The experiments aimed to generate lightweight olefins by operating at temperatures ranging from 310 to 330 °C, with a GHSV (Gas Hourly Space Velocity) of 2700 h^{-1} , operating pressures between 5 and 9 bar, and a feed ratio of H_2 to CO of 2:1. A 0.5 g catalyst was used during the experiments. The results indicated that the Fe/MIL-53(Al) catalyst played a significant role in producing hydrocarbons, including

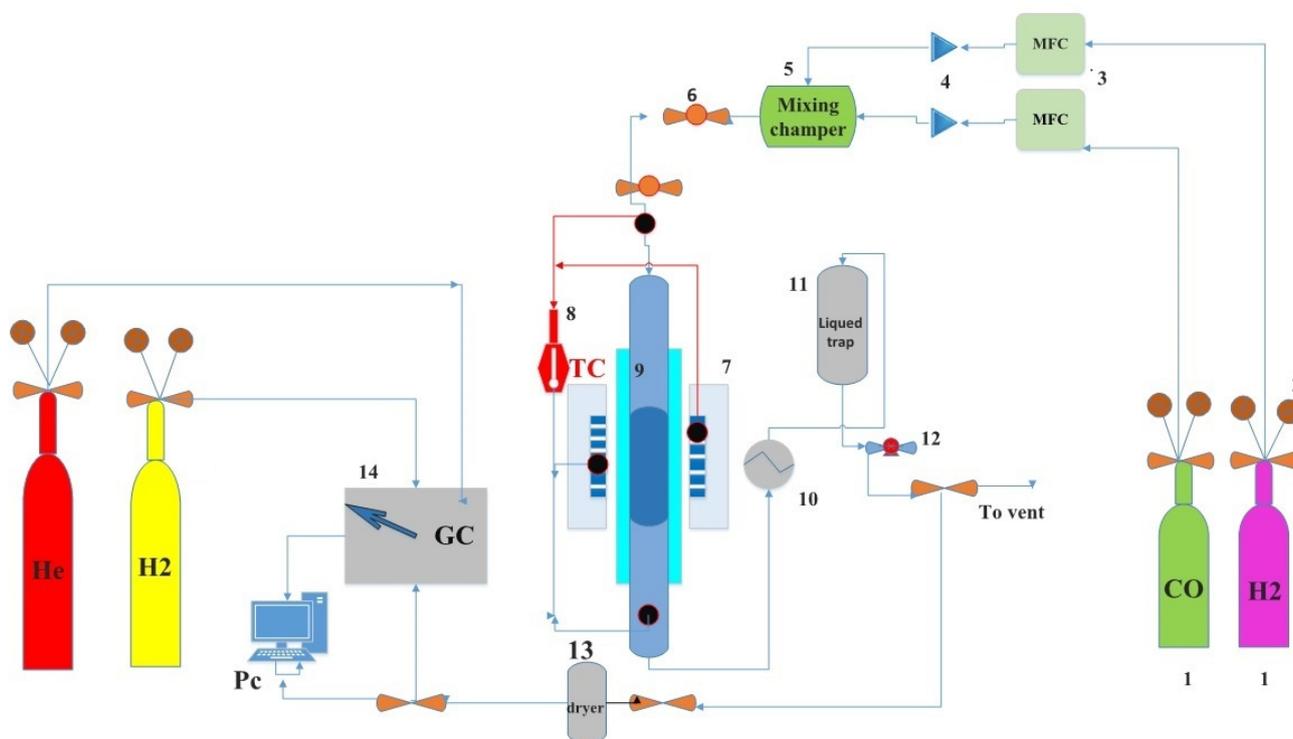


Fig. 1. Outline of Fixed-bed microreactor in the flow chart.

1. Pure Gas cylinders, 2. Pressure regulators, 3. Mass Flow Controllers (Brooks MFC), 4. Valves, 5. Mixing chamber, 6. Tube Pressure gauges, 7. Tubular furnace, 8. Temperature indicators, 9. Tubular reactor and catalyst fixed bed, 10. Condenser, 11. Trap, 12. BPR: Back Pressure Regulator, 13. Silica gel column (dryer), and 14. Gas Chromatograph (GC).

lightweight olefins, during 72 h of reaction.

To analyze the outcomes, the following equations were introduced to evaluate the CO conversion, methane selectivity, and light olefinity.

$$\text{CO Conversion (\%)} = \frac{CO_{in} - CO_{out}}{CO_{in}} \times 100 \quad (1)$$

$$\text{Methane Selectivity (\%)} = \frac{\text{Moles of methane}}{\text{Moles of Hydrocarbons}(C1 - C4)} \times 100 \quad (2)$$

$$\text{Light olefinity (\%)} = \frac{\text{Moles of Olefins}}{\text{Moles of Hydrocarbons}(C1 - C4)} \times 100 \quad (3)$$

The findings obtained from the Fe/MIL-53(Al) catalyst demonstrated that the best reactor conditions for producing lightweight olefins resulted in a selectivity of 24%. These conditions included a temperature of 310 °C, GHSV of 2700 h⁻¹, a pressure of 5 bar, and an H₂/CO feed ratio of 2:1, as depicted in Fig. 2. Additionally, the highest conversion efficiency (41%) was achieved at a temperature of 330 °C, a pressure of 10 bar, an H₂/CO feed ratio of 2:1, and a GHSV of 2700 h⁻¹, as shown in Fig. 3.

Under these operating conditions and based on the experimental data, it was observed that lightweight olefins exhibited the highest selectivity but the lowest conversion efficiency at lower pressures and temperatures.

Fig. 4(a) illustrates the MIL-53(Al) XRD spectrum after heat-treatment activation at 330°C for 72 hours. According to reference [26], the crystalline structure of the studied MOF was confirmed. When the support's crystalline structure is activated through heat treatment, the guest molecules are displaced, causing a reduction in the intensity of the dominant peaks. Fig. 4(b) displays the XRD spectrum of the Fe/MIL-53(Al) catalyst after the reactor test, revealing that the support retained its structure in the reactor.

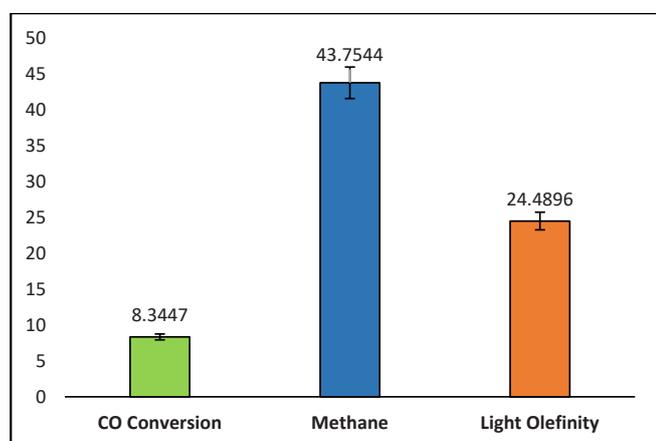


Fig. 2. CO conversion and light hydrocarbon products over the Fe/MIL-53(Al) catalyst (T = 310 °C, P = 5 bar, Feed ratio = 2, and GHSV = 2700 h⁻¹).

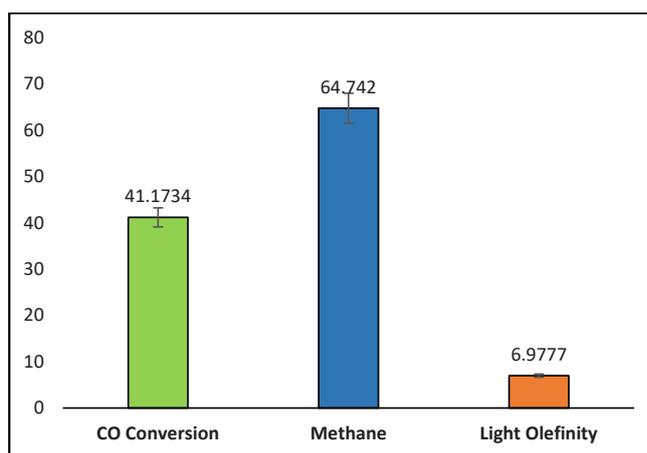


Fig. 3. CO conversion and light hydrocarbon products over the Fe/MIL-53(Al) catalyst (T = 330 °C, P = 10 bar, Feed ratio = 2, and GHSV = 2700 h⁻¹).

Table 1 presents the results of the BET experiments, where the specific surface area of the support (A₁) was measured at 1136.0702 m².g⁻¹, aligning with previous research [13]. However, the specific surface area of the catalyst decreased after impregnation. The average diameter of the pores fell within the range of mesoporous compounds (2 – 50 nm).

Considering the results from the XRD and BET analyses, it can be concluded that the guest molecules were expelled from the clusters, and the large surface area and pore sizes demonstrate the feasibility of using MIL-53(Al) metal-organic compounds as support for iron catalysts in the FTS process.

Table 2 provides an overview of previous research regarding the use of metal-organic frameworks as a support of catalysts in Fischer-Tropsch synthesis. The catalysts' specific surface areas range from 32 to 693 m².g⁻¹, with the catalyst employed in this study exhibiting the highest value. A comparison of the specific surface area of the utilized catalyst (286 m².g⁻¹) with previous works reveals that it surpasses certain values observed in fresh catalysts, such as 20% Co/MIL-53(Al). Furthermore, the catalyst

Table 1. BET analysis for the support and catalysts (before and after the test).

Sample	Specific surface area (m ² .g ⁻¹)	Pore volume (cm ³ .g ⁻¹)	Pore diameter (mm)
A ₁	1136.1	1.356	4.77
A ₂	693.3	0.337	1.94
A ₃	286.2	0.792	12.91

A₁: Support (MIL-53(Al)), A₂: Fresh catalyst (Fe/MIL-53(Al)), A₃: Used catalyst

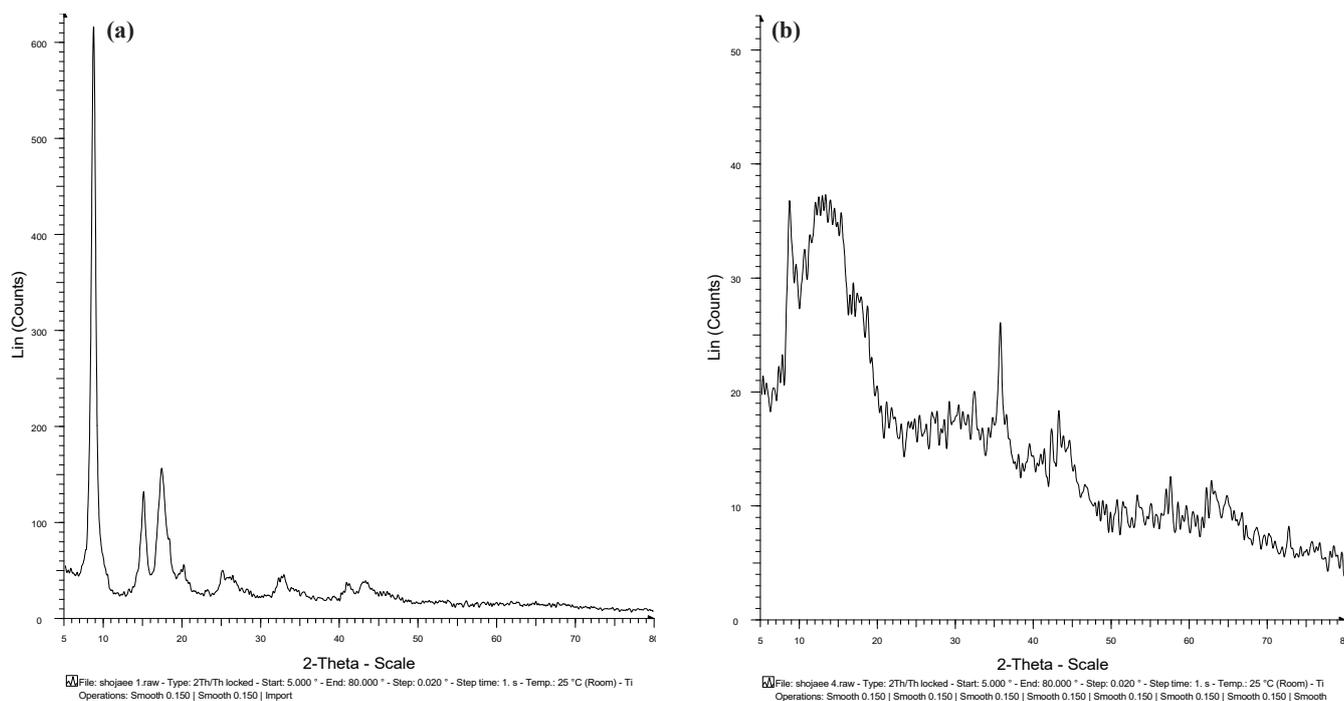


Fig. 4. XRD pattern of (a) activated MIL-53(Al) ($T = 330\text{ }^{\circ}\text{C}$ for 72 h) and (b) Fe/MIL-53(Al) catalyst after the reaction.

exhibited satisfactory structural stability during the FTS reaction, as evidenced by its stability time. Additionally, the Fe/MIL-53(Al) catalyst displayed a remarkable selectivity for producing lightweight olefins, particularly under lower pressures.

4. Conclusion

The research conducted in this study focused on examining the feasibility of using MIL-53(Al), a metal-organic compound, as a support for Fe/MIL-53(Al) catalyst

Table 2. Overview of some previous research on the use of MOFs as supports for iron and cobalt catalysts in the Fischer-Tropsch synthesis.

Catalyst	BET ($\text{m}^2\cdot\text{g}^{-1}$)	Reactor	Operating conditions	CO Conversion (%)	Stability time (h)	Light olefins (%)	Ref.
Co@MIL-53(Al)	400-600	Fixed bed	$T = 240\text{ }^{\circ}\text{C}$ $P = 20\text{ bar}$ $\text{H}_2:\text{CO} = 2:1$	60	20	Not reported	[19]
Fe-MIL-88B-NH ₂ /C	32.8	Fixed bed	$T = 300\text{ }^{\circ}\text{C}$ $P = 20\text{ bar}$ $\text{H}_2:\text{CO} = 1:1$ GHSV=18000 h ⁻¹	81.8	200	21.4	[20]
Fe-MIL-88B/C	117.8			33.8		18.3	
Fe@C-MIL-88	655	high-pressure reactor	$T = 340\text{ }^{\circ}\text{C}$ $P = 20\text{ bar}$	66	-	Not reported	[21]
Fe@C-MIL-101-NH ₂	635			65			
Fe@C-F300	570	assembly module-I	$\text{H}_2:\text{CO} = 1:1$	76			
20% Co/MIL-53(Al)	231.4	Fixed bed	$T = 220\text{ }^{\circ}\text{C}$ $P = 20\text{ bar}$ $\text{H}_2:\text{CO} = 2:1$	50.6	80	10	[22]
5% Co@MIL-53(Al)		Fixed bed	$T = 240\text{ }^{\circ}\text{C}$ $P = 20\text{ bar}$	23.8	20	13.5	[15]
10% Co@MIL-53(Al)				47.1		11.9	
15% Co@MIL-53(Al)	450		$\text{H}_2:\text{CO} = 2:1$	60.2		10.7	
Fe/MIL-53(Al)	693.3	Fixed bed	$T = 310\text{ }^{\circ}\text{C}$ $P = 5\text{ bar}$ $\text{H}_2:\text{CO} = 2:1$	41	72	24	This work

in the Fischer-Tropsch synthesis. The study emphasized the significant impact of the size and morphology of MIL-53(Al) on the catalytic performance of Fe/MIL-53(Al). The Fe/MIL-53(Al) catalyst was prepared using dry impregnation and evaluated in a fixed bed reactor under specific conditions, including an H₂/CO feed ratio of 2:1, temperatures ranging from 310 to 330 °C, pressures ranging from 5 to 9 bar, and a GHSV of 2700 h⁻¹. The results demonstrated that Fe/MIL-53(Al) can act as an active catalyst for producing light olefins through FTS, achieving a yield of 24% at high temperatures and low pressure.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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