



# Production of Olefins by Dehydrogenation of Propane over Fe Modified ZSM-5

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**How to cite this paper:** Hadadi, S.M., Alshamrani, K.M., Bajunaid, A.S. and Alosaimi, A.M. (2024) Production of Olefins by Dehydrogenation of Propane over Fe Modified ZSM-5. *Open Access Library Journal*, 11: e12358.

<https://doi.org/10.4236/oalib.1112358>

**Received:** September 22, 2024

**Accepted:** October 26, 2024

**Published:** October 29, 2024

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## Abstract

In this report, a simple method was employed to synthesize Fe/ZSM-5 for propane (C<sub>3</sub>H<sub>8</sub>) dehydrogenation. Fe of varying loading 1.5 - 3 wt% was with ZSM-5. Catalyst physicochemical properties were analyzed using XRD and FTIR. The XRD and FTIR characterization showed that the modified catalysts retained their crystallinity after metal impregnation. The performance tests for propane dehydrogenation to produce olefins over synthesized catalysts were carried out in a fixed-bed reactor at 550°C and atmospheric pressure. The introduction of Fe into the ZSM-5 improved the ethylene (C<sub>2</sub>H<sub>4</sub>) selectivity.

## Subject Areas

Chemical Engineering & Technology

## Keywords

Dehydrogenation, Propane, ZSM-5, Fixed-Bed Reactor, Olefins

## 1. Introduction

It has become imperative to consider the conversion of abundant gas resources (light alkanes such as propane) to olefin compounds (methane, ethane, ethylene, and propylene), which are indispensable feedstocks to the chemical and petrochemical industries [1]-[6]. Large amounts of propane can be found in gas condensate, refinery gases, and natural gas reserves. Additional innovation in oil-to-olefins production technology is required [7] [8]. The need for olefins has gradually increased globally in recent years [9]. When compared to the conventional method of cracking and splitting petroleum products to produce olefins, the benefits of using low-carbon alkanes to make olefins include excellent yields, economical production costs, and resource efficiency [10]-[12]. The production of light

olefins like ethylene and propylene through the dehydrogenation of short-chain alkanes has been extensively documented in recent decades due to its great efficiency [13] [14]. As the two most important platform materials in the petrochemical sector, propylene and ethylene are produced globally each year in quantities of 160 and 110 megatons, respectively, and are extensively utilized in the synthesis of several chemicals [15]-[17]. Since shale gas is now a plentiful and easily available resource due to technological advancements in the field, direct dehydrogenation of short-chain alkanes like propane has gained popularity as a means of producing light olefins [18]-[20]. Pt- and Cr-based catalysts, which are costly or environmentally unfriendly, are the most regularly employed catalysts for the process. It also frequently requires greater temperatures, which raises the energy cost [21] [22]. Recently, direct dehydrogenation of alkanes has also been achieved with various environmentally benign and non-noble metal-based catalysts, such as Fe-containing zeolite, which has shown excellent performance in this process; however, these investigations are still in the laboratory research stage and are still a long way from being used in practical applications [23] [24]. Fe and ZSM-5-based catalysts have the benefit of being less expensive and having better reactivity than other catalysts [25] [26]. Overall, in the petrochemical sector, propane dehydrogenation is a critical step that produces vital raw materials for a variety of chemical products [27] [28]. The iron's catalytic activity in hydrogen transfer processes and the acidic characteristics of ZSM-5 for hydrocarbon activation and shape-selectivity may be advantageous to the combination. Such a combination catalyst's precise performance would be contingent upon variables like the ZSM-5 pore size, the iron loading, and the reaction circumstances. Enhancing these factors can aid in attaining elevated activity and selectivity [29]-[31]. Key challenges in employing mixed Fe and ZSM-5 catalysts include balancing the roles of both components and addressing problems such as catalyst deactivation and by-product production [32] [33]. To sum it all up, ZSM-5 zeolites and Fe-based catalysts both have benefits when it comes to propane dehydrogenation. Though careful optimization and management of catalyst stability are necessary to achieve optimal performance, combining these materials may be able to develop a catalyst system that benefits from the special qualities of both. In this work, olefins were prepared from the reaction of propane using catalysts prepared in a simple, green and safe way without using any solvents and the catalysts were characterized by XRD and IR device.

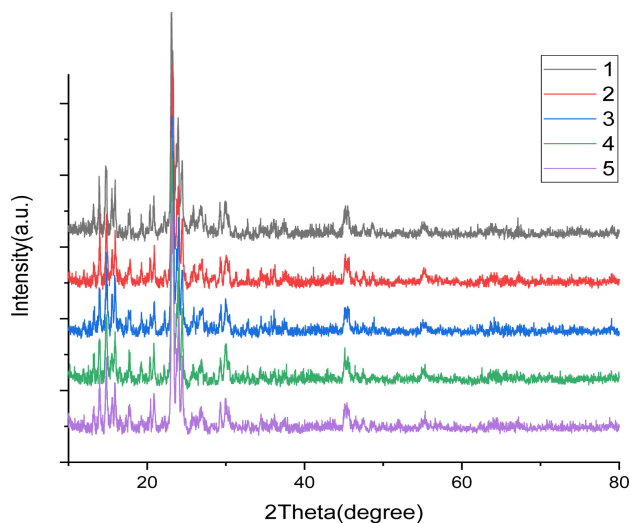
## 2. Results and Discussion

### 2.1. Catalyst Characterizations

#### 2.1.1. XRD Analysis

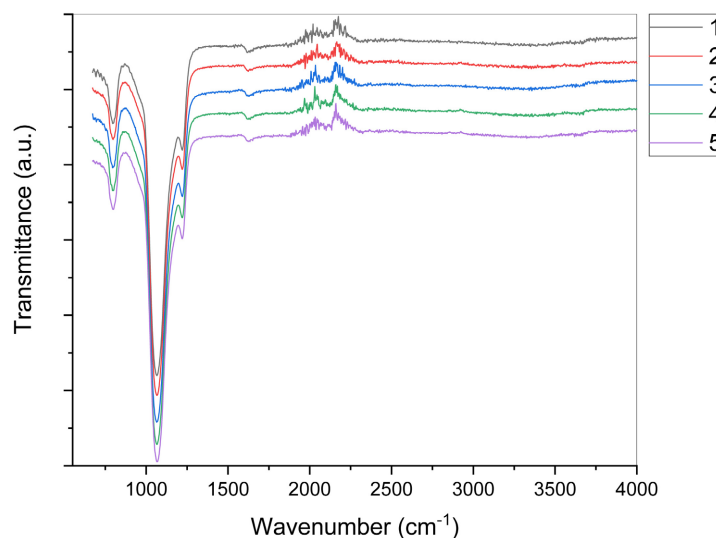
The X-ray diffractogram (XRD) of the parent HZSM-5 with the Fe-modified catalysts was presented in **Figure 1**. The loading of Fe had no significant effect on the ZSM-5 structure as loading increased. This indicates that ZSM-5 crystallinity was maintained and structure unaffected by metals. The characteristic peaks of ZSM-5

were found though suppressed at  $2\theta = 7.89^\circ$ ,  $8.73^\circ$ ,  $14.82^\circ$ ,  $23.04^\circ$ ,  $23.86^\circ$  and  $24.26^\circ$  at increased Fe loading [34] [35].



**Figure 1.** XRD patterns of ZSM-5 (1), 1.5% Fe-ZSM-5 (2), 2% Fe-ZSM-5 (3), 2.5% Fe-ZSM-5 (4) and 3% Fe-ZSM-5 (5).

### 2.1.2. FTIR Analysis



**Figure 2.** FTIR spectra of ZSM-5 (1), 1.5% Fe-ZSM-5 (2), 2% Fe-ZSM-5 (3), 2.5% Fe-ZSM-5 (4) and 3% Fe-ZSM-5 (5).

The FTIR spectra of the 1.5% Fe-ZSM-5, 2% Fe-ZSM-5, 2.5% Fe-ZSM-5, and 3% Fe-ZSM-5 adsorbents displayed characteristic bands associated with the ZSM-5 structure in **Figure 2**. Specifically, the broad bands at approximately  $3422$  and  $1630$   $\text{cm}^{-1}$  corresponded to the stretching and deformation vibrations of the O-H bonds, respectively. The absorption bands at  $1230$   $\text{cm}^{-1}$  to  $1090$   $\text{cm}^{-1}$ ,  $800$   $\text{cm}^{-1}$ ,  $530$   $\text{cm}^{-1}$ , and  $440$   $\text{cm}^{-1}$  were attributed to the asymmetric-stretching of Si-O bonds, which indicate the zeolites formed by one or four chains of five-membered

rings. Additionally, the symmetrical-stretching of Si-O-Si bonds, the stretching of Si-O-T bonds (representing double five-membered rings in ZSM-5 zeolites), and the bending vibration of (tetrahedrons) bonds in the internal tetrahedral of  $\text{AlO}_4$  and  $\text{SiO}_4$  were observed [36] [37].

### 3. Materials and Methods

#### 3.1. Materials

All the reagents used in this work are of analytical grade and were used as received without further purification. The ZSM-5 zeolite (CBV 8014) powder in ammonium form was acquired from Zeolyst, USA, and Iron (III) nitrate nonahydrate  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (Alfa Aesar, 98%).

#### 3.2. Preparation of Fe/ZSM-5 Catalysts

The Fe/ZSM-5 was prepared by mixing  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and HZSM-5 in weight proportions (1.5, 2, 2.5, and 3 wt%) in 50 mL of distilled water with stirring for effective mixing at room temperature for half an hour. The catalysts were filtered and washed with distilled water; then, it was dried at 80 °C for 24 h and calcined at 500 °C (5 °C per minute ramping) for 5 h in air.

#### 3.3. Characterization of the Catalysts

Powder X-ray diffraction (XRD) patterns were carried out using a D2 phaser (Bruker, Germany) X-ray diffractometer, the wavelength of X-ray (1.540 Å). Fourier transform infrared (FTIR) spectra were recorded in a Perkin-Elmer spectrum BX (Perkin Elmer, USA) using the KBr disk method in the range of 400 to 4000  $\text{cm}^{-1}$ .

#### 3.4. Catalytic Tests

To assess the monolithic catalysts' performances, the reactor was equipped with a furnace to provide homogenous heating, and a K-type thermocouple was employed in the middle of the reactor to measure and monitor the temperature of the bed. The dehydrogenation propane (DHP) reaction over all the monolithic catalysts was a (0.5 g) monolithic catalyst with (0.5 g) of SiC packed into the reactor. All the connecting lines for the feed and product streams were equipped with heating tapes to prevent any condensation. Prior to the DHP reaction, the catalysts were activated with  $\text{N}_2$  at a flow rate of 50 mL/min and 550 °C for 20 min. After 20 min, the feed gas (propane) was flown into the reactor at a flow rate of 50 mL/min and a space velocity of 6000  $\text{mL gcat}^{-1} \cdot \text{h}^{-1}$ . The outlet stream of the reactor was fed to the VARIAN CP-3800 gas chromatograph (GC), which was equipped with a thermal conductivity detector (TCD) and flame ionization detector (FID). The stream was analyzed every 45 minutes to find out the composition of the product stream. DHP over the catalysts was performed at 550 °C for 3 h to assess and screen the reaction. The propane conversion and the methane, ethane, ethylene, and

propylene selectivities were calculated.

### 3.4.1. Catalytic Activity

The catalytic conversion of propane ( $C_3H_8$ ) to olefins such as methane, ethane, ethylene, and propylene at  $550^\circ C$  and 1 atm is an important reaction in the field of petrochemicals. ZSM-5 is a zeolite with a unique pore structure that facilitates the selective conversion of hydrocarbons. The acidic sites in ZSM-5 play a vital role in catalytic activity. The balance of Brønsted and Lewis acidity affects the reaction pathways. The acidity of ZSM-5 is crucial for the activation of propane, enabling its transformation into more reactive species. The acidity can influence the selectivity towards different products. Higher acidity may favor the formation of lighter olefins like ethylene and propylene, while lower acidity might lead to more cracking and the formation of lighter gases like methane and ethane. Propane can undergo dehydrogenation to produce propylene and hydrogen. Over-cracking can produce methane and ethane, while oligomerization and cyclization pathways can lead to the formation of ethylene and propylene. The decrease in propane conversion with iron loading suggests that the presence of iron may inhibit certain reaction pathways. However, the increased selectivity for ethylene indicates that while overall conversion is lower, the reactions that do occur are more favorable for forming ethylene. The low yield of ethane across all catalysts might suggest that the reaction conditions or catalyst properties favor cracking and dehydrogenation to produce olefins rather than saturating hydrocarbons. This behavior could be influenced by the specific properties of the zeolite, such as pore structure and acidity, as well as the nature of the iron species present. The observation you have made about selectivity trends with increasing iron loading in zeolite catalysts is quite insightful. Higher iron loading can enhance specific active sites that favor the conversion of substrates into desired products like ethylene and methane, while concurrently suppressing pathways leading to ethane and propylene. By adjusting iron loading, the catalyst can be optimized for selectivity, thus maximizing the yield of valuable products and minimizing undesired byproducts. This behavior is significant for industrial applications, where optimizing catalyst properties is crucial for achieving higher efficiency and cost-effectiveness in chemical processes.

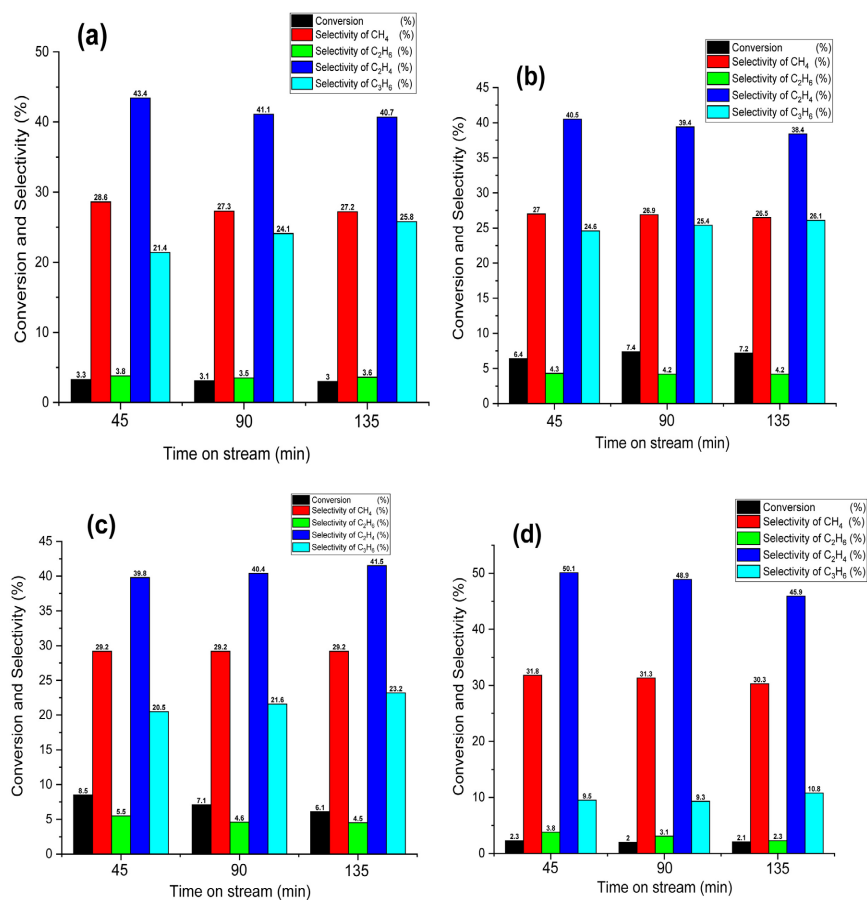
### 3.4.2. Catalytic Performance of ZSM-5 Catalyst

**Table 1.** Time on stream experiment of propane over (ZSM-5 (8014)); reaction conditions: catalyst loaded = 0.5 g,  $C_3H_8:N_2$  ratio of 1:1, and temperature =  $550^\circ C$ .

Time on Stream (min)	Conversion (%)	Selectivity (%)			
		Methane	Ethane	Ethylene	Propylene
45	12.41	27.35	5.08	38.52	19.84
90	10.76	27.66	4.84	38.35	20.39
135	9.95	28.53	4.59	39.70	21.09

**Table 1** shows the conversion and selectivity values for the reaction of dehydrogenation of propane DHP at 550 °C and 1 atmosphere for the zeolite used after calcining at 500 °C and shows the formation of methane, ethane, ethylene and propylene. The catalytic conversion of propane to olefins is an important reaction in the field of petrochemicals. ZSM-5 is a zeolite with a unique pore structure that facilitates the selective conversion of hydrocarbons. The acidic sites in ZSM-5 play a vital role in catalytic activity. The balance of Brønsted and Lewis acidity affects the reaction pathways. The acidity of ZSM-5 is crucial for the activation of propane, enabling its transformation into more reactive species [38]-[40].

### 3.4.3. Catalytic Performance of Fe/ZSM-5 Catalysts



**Figure 3.** Time on stream experiment of propane over (a) 1.5% Fe/ZSM-5, (b) 2% Fe/ZSM-5 (c) 2.5% Fe/ZSM-5, and (d) 3% Fe/ZSM-5; reaction conditions: catalyst loaded = 0.5 g, C<sub>3</sub>H<sub>8</sub>:N<sub>2</sub> ratio of 1:1, and temperature = 550 °C.

The DHP catalytic activities of the prepared catalysts are shown in **Figure 3**. All catalysts demonstrate different trends. This can be seen from the catalytic evaluation results, the results from the catalytic stability experiments at 550 °C for 135 min are shown all the catalysts exhibited a stable performance during the prolonged DHP reaction. In agreement with catalytic tests, 3% Fe/ZSM-5 (**Figure 3(d)**) showed the highest methane and ethylene selectivity with the values of 31%

and 50%, respectively. The propane conversion is roughly in the same range from 12% to 2%, where the highest conversion was observed in ZSM-5. The decrease in propane conversion with iron loading suggests that the presence of iron may inhibit certain reaction pathways. However, the increased selectivity for ethylene indicates that while overall conversion is lower, the reactions that do occur are more favorable for forming ethylene. The low selectivity of ethane across all catalysts might suggest that the reaction conditions or catalyst properties favor cracking and dehydrogenation to produce olefins rather than saturating hydrocarbons. This behavior could be influenced by the specific properties of the zeolite, such as pore structure and acidity, as well as the nature of the iron species present. The observation you have made about selectivity trends with increasing iron loading in zeolite catalysts is quite insightful. Higher iron loading can enhance specific active sites that favor the conversion of substrates into desired products like ethylene and methane, while concurrently suppressing pathways leading to ethane and propylene. By adjusting iron loading, the catalyst can be optimized for selectivity, thus maximizing the yield of valuable products and minimizing undesired byproducts. This behavior is significant for industrial applications, where optimizing catalyst properties is crucial for achieving higher efficiency and cost-effectiveness in chemical processes.

#### 4. Conclusion

In summary, catalytic dehydrogenating of propane ( $C_3H_8$ ) was carried out over ZSM-5 and Fe/ZSM-5 catalysts. The characterizations were made by using XRD and IR, which revealed that the crystallinity and structure of the zeolite were preserved. It was observed that adding metal to zeolite caused a decrease in conversion and increased selectivity for olefin compounds such as ethylene ( $C_2H_4$ ) and methane ( $CH_4$ ). In addition, when using all the prepared catalysts, the dehydrogenation reaction path was not altered.

#### Acknowledgements

We thank King Abdulaziz City for Science and Technology (KACST) for funding this research project.

#### Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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