



Potassium-Promoted Iron Carbide Interfaces for Direct CO₂ Conversion to Light Olefins

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ABSTRACT

Direct CO₂ hydrogenation to light olefins provides a promising route for producing carbon-based chemicals from non-fossil carbon sources. In this work, potassium-promoted Fe-based catalysts were prepared by co-precipitation and evaluated for CO₂ hydrogenation under Fischer–Tropsch conditions. XRD, Mössbauer spectroscopy, CO₂-TPD, H₂-TPR, and in situ XPS were used to identify the evolution of iron carbide phases and surface basicity. The catalyst with 1.5 wt% K showed a CO₂ conversion of 34.8%, C₂–C₄ olefin selectivity of 42.6%, and an olefin/paraffin ratio of 5.3 at 320 °C and 2.5 MPa. Potassium addition increased medium-strength CO₂ adsorption sites by 68.2% and promoted the formation of χ -Fe₅C₂, which was identified as the main active phase for chain growth. Excessive potassium loading suppressed hydrogen activation and reduced hydrocarbon productivity.

1. Introduction

Light olefins, particularly ethylene, propylene, and butenes, are essential building blocks for the production of polymers, synthetic rubbers, solvents, and numerous value-added chemicals. Industrial light olefins are currently produced mainly through steam cracking of hydrocarbons and fluid catalytic cracking of petroleum fractions, processes that rely heavily on fossil carbon and require substantial energy input. Direct hydrogenation of CO₂ to light olefins provides an alternative route for carbon utilization when low-carbon H₂ is available. Fe-based catalysts are particularly attractive for this process because they can integrate the reverse water–gas shift (RWGS) reaction and Fischer–Tropsch synthesis (FTS) within a single catalytic system. CO₂ is initially converted to CO through RWGS, after which adsorbed CO-derived species undergo hydrogenation, C–C coupling, and chain propagation to form hydrocarbons (Ait El Fakir et al., 2025; You, 2026). The catalytic surface involved in this sequence should not be regarded as a single static structure. Recent atomistic studies of CO₂ hydrogenation have demonstrated that catalytic activity may arise from an ensemble of structurally distinct configurations with substantially different reactivities, and that redistribution among these configurations under reaction conditions can strongly influence both catalytic rates and deactivation (Yang et al., 2026a). This concept is particularly relevant to Fe-based CO₂ hydrogenation because the Fe phase undergoes extensive structural and chemical reconstruction during activation and reaction. Achieving high light-olefin selectivity is consequently challenging, as CO, CH₄, paraffins, light olefins, and C₅⁺ hydrocarbons can be formed simultaneously, while primary olefins can undergo further hydrogenation or secondary chain-

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growth reactions (Guérette et al., 2025; Jiao et al., 2026a). The catalytic functions of Fe-based materials are closely associated with the dynamic transformation among iron oxide, metallic Fe, and iron carbide phases. Fe_2O_3 precursors are generally reduced through Fe_3O_4 -containing intermediates, and further reduction and carburization generate metallic or carbide-rich domains depending on the reaction environment. Fe_3O_4 is commonly associated with RWGS activity and the formation of CO, whereas iron carbides provide sites for CO dissociation, carbon insertion, and C–C bond formation (Wu et al., 2026; Yeom et al., 2026). Among the reported carbide structures, $\chi\text{-Fe}_5\text{C}_2$ has frequently been correlated with hydrocarbon chain propagation and Fischer–Tropsch activity (Bao et al., 2026a; Schmidt et al., 2025). The coexistence of oxide and carbide phases is therefore important for direct CO_2 conversion because the two reaction functions must operate at compatible rates. Insufficient RWGS activity limits the supply of CO-derived intermediates, whereas excessive CO formation without efficient carburization and chain propagation results in high CO selectivity. Conversely, highly hydrogenating surfaces can favor CH_4 and paraffins instead of olefins. The catalytic performance is therefore determined not simply by the total Fe content or the presence of a particular crystalline phase, but by the relative abundance, accessibility, and spatial interaction of oxide and carbide domains under working conditions.

The distribution of iron carbide phases is also sensitive to carbon chemical potential, H_2 availability, water concentration, temperature, and promoter environment. $\chi\text{-Fe}_5\text{C}_2$ is often considered an important hydrocarbon-forming phase, but Fe_7C_3 and other carbide structures may become significant depending on synthesis and reaction conditions. Recent studies have shown that controlled carbide structures and alloy–carbide environments can alter chain-growth probability and hydrocarbon selectivity (Chen et al., 2025; Madadian et al., 2025). This complexity is important because carbide phases identified after reaction do not necessarily represent the structures responsible for steady-state catalysis. Water produced during RWGS and hydrocarbon formation can partially oxidize carbide surfaces, while carburization can restore carbide domains when the local CO or carbon activity increases. The working catalyst may therefore contain continuously interconverting oxide–carbide regions rather than a fixed bulk phase. Such dynamic behavior makes it necessary to connect phase composition measured under or near reaction conditions with catalytic rates and product distributions instead of assigning activity solely from post-reaction diffraction patterns. Alkali-metal promotion is one of the most effective approaches for shifting Fe-based CO_2 hydrogenation toward olefin production. Potassium is widely used because it modifies both the adsorption properties and the electronic environment of Fe surfaces. K increases surface basicity and generally strengthens the interaction of the catalyst with acidic CO_2 molecules. It can also alter Fe reducibility, facilitate carburization, change the adsorption strength of CO-derived species, decrease effective surface hydrogen coverage, and modify the desorption behavior of hydrocarbon products. At suitable loadings, these effects can suppress excessive methanation and secondary hydrogenation of primary olefins, thereby increasing the olefin-to-paraffin ratio. Studies of Fe–Co, Fe–Zn, and related promoted Fe systems have further shown that intimate contact among oxide, metal, and carbide domains can influence CO formation, carbide development, and the release of olefin products (Ali et al., 2026; Liang et al., 2025). K promotion has also been reported to accelerate the development of Fe_5C_2 and to improve the resistance of carbide phases to oxidation by reaction-generated water (Suppiah et al., n.d.). These effects provide a mechanistic basis for the frequently observed enhancement of olefin selectivity after alkali addition. The promotional effect of K, however, is not monotonic with loading. Increasing K content can enhance CO_2 adsorption and decrease the surface hydrogen activity that drives olefin hydrogenation, but excessive K can cover accessible Fe sites, inhibit H_2 dissociation, and reduce the rate of hydrogen supply to carbon-containing intermediates. The same promoter can therefore enhance olefin selectivity while

decreasing total hydrocarbon productivity when its concentration exceeds an optimum range. Computational studies on K-modified χ -Fe₅C₂ support this balance by indicating that K can alter CO₂ activation and C–C coupling while simultaneously decreasing the effective activity of surface hydrogen species (Du, 2025; Liang et al., 2026). The resulting catalytic behavior depends on a competition among CO₂ activation, CO production, carbide formation, chain growth, hydrogenation, and hydrocarbon desorption. A catalyst with very strong CO₂ adsorption is not necessarily the most active because strongly retained carbonate-like species may occupy surface sites, while insufficient hydrogen activation can restrict the conversion of adsorbed carbon species. Medium-strength CO₂ adsorption may therefore be more relevant to sustained turnover than total CO₂ uptake alone. This balance also provides a useful explanation for changes in the olefin-to-paraffin ratio. Primary α -olefins generated during chain termination can either desorb from the catalyst or readsorb and undergo hydrogenation, isomerization, or additional chain growth. Increasing surface hydrogen availability generally raises the probability of secondary hydrogenation and paraffin formation. Moderate K promotion can lower the effective hydrogen coverage and weaken these secondary pathways, allowing a larger fraction of newly formed olefins to leave the surface before further conversion. Excessive suppression of hydrogen activation, however, can restrict the hydrogenation steps required for productive Fischer–Tropsch turnover itself. Olefin selectivity and hydrocarbon productivity must therefore be evaluated together. An increase in olefin fraction accompanied by a severe decrease in total hydrocarbon formation may reflect suppression of hydrogenation activity rather than a genuine enhancement of the intrinsic C₂–C₄ olefin-forming pathway. Operando and in situ characterization has increasingly shown that Fe oxide, carbide, promoter, and support structures continue to evolve under CO₂ hydrogenation conditions (Bao & Wang, 2026b; Mishra & Lin, 2026a). Nevertheless, several important uncertainties remain. Iron carbide phases are still frequently assigned from catalysts characterized only after cooling and exposure to an inert atmosphere or air, even though surface oxidation and structural transformation can occur during these procedures. Conventional XRD is useful for identifying sufficiently crystalline bulk phases but may not reliably distinguish small, poorly ordered, structurally similar, or coexisting iron carbides. Mössbauer spectroscopy is particularly valuable for resolving Fe-containing phases with different local environments, yet it is still not routinely combined with surface-sensitive characterization and kinetic measurements across systematic promoter series. Surface information is equally important because bulk carbide fraction does not directly determine the number or electronic state of catalytically accessible Fe sites. In situ XPS can complement bulk phase analysis by monitoring changes in surface Fe species and the chemical environment of K, allowing promoter-induced surface reconstruction to be related more directly to catalytic performance. A further limitation concerns how K promotion is interpreted quantitatively. Many studies correlate promoter loading directly with CO₂ conversion or product selectivity without establishing how K simultaneously changes CO₂ adsorption strength, Fe reduction and carburization, surface hydrogen activation, and the relative abundance of oxide and carbide phases. Studies based on only one or two promoted samples cannot readily determine whether the observed improvement originates from increasing the density of beneficial K-modified sites or simply from moving toward an optimum balance between CO₂ activation and hydrogen availability. Short reaction tests introduce additional uncertainty because the Fe phase may still be undergoing carburization or promoter redistribution when product selectivity is measured. As a result, correlations among K loading, χ -Fe₅C₂ formation, adsorption strength, hydrogen activation, and olefin production remain insufficiently resolved.

The present study addresses these issues using a systematically controlled series of K-promoted Fe catalysts prepared by co-precipitation and evaluated under identical CO₂ Fischer–Tropsch reaction

conditions. Rather than treating K content only as a compositional variable, the study examines how progressive K addition modifies the coupled functions required for CO₂ conversion to light olefins. XRD and Mössbauer spectroscopy are used together to determine the evolution and relative distribution of Fe oxide and carbide phases, with particular attention to χ -Fe₅C₂ formation. CO₂-TPD is employed to distinguish changes in adsorption capacity and adsorption strength, while H₂-TPR provides information on promoter-induced changes in Fe reducibility and hydrogen-related activation behavior. In situ XPS is further used to follow the surface chemical states of Fe and K and to determine whether changes observed in the bulk phase are accompanied by corresponding modifications of the catalytically accessible surface. These structural and chemical descriptors are correlated with CO₂ conversion, hydrocarbon productivity, light-olefin selectivity, and the olefin-to-paraffin ratio. The central objective is to establish how K loading controls the balance among medium-strength CO₂ adsorption, formation and stabilization of χ -Fe₅C₂, surface hydrogen availability, and secondary olefin hydrogenation. Particular attention is given to the transition from beneficial promotion to over-promotion, where additional K may continue to suppress paraffin formation but simultaneously reduce hydrocarbon productivity by restricting H₂ activation and accessible Fe sites. By resolving this activity–selectivity trade-off, the study aims to provide a mechanistic basis for optimizing alkali-promoted Fe catalysts and to clarify how promoter loading can be used to control the dynamic oxide–carbide surface required for efficient direct conversion of CO₂ into light olefins.

2. Materials and Methods

2.1 Catalyst Samples and Feed Gases

Five Fe-based catalysts were prepared with nominal K loadings of 0, 0.5, 1.0, 1.5, and 2.5 wt%. They were named FeK-0, FeK-0.5, FeK-1.0, FeK-1.5, and FeK-2.5, respectively. Iron nitrate nonahydrate was used as the Fe source, and potassium carbonate was used as the K source. The Fe precursor was dissolved in deionized water and precipitated with sodium carbonate at 70°C and pH 8.0. The suspension was aged for 3 h, filtered, and washed until the filtrate conductivity was below 50 $\mu\text{S cm}^{-1}$. The solid was dried at 110°C for 12 h and calcined in air at 400 °C for 4 h. Potassium was then added by wet impregnation. The samples were dried again and calcined at 350°C for 3 h. Before testing, the catalysts were sieved to 250–425 μm . CO₂, H₂, N₂, He, and Ar with purities above 99.999% were used in all experiments.

2.2 Experimental Design and Control Tests

CO₂ hydrogenation was carried out in a stainless-steel fixed-bed reactor with an inner diameter of 8 mm. Each run used 0.40 g of catalyst mixed with 0.80 g of quartz particles. The quartz improved heat transfer and reduced pressure drop. Before reaction, the catalyst was reduced in 20 vol% H₂/N₂ at 350°C for 4 h. The reactor was then cooled to 320°C and pressurized to 2.5 MPa. The H₂/CO₂ molar ratio was 3:1, and the gas hourly space velocity was 6000 mL g_{cat}⁻¹ h⁻¹. FeK-0 was used as the K-free control. The promoted samples were used to examine the effect of K loading. Blank tests were conducted with quartz and K₂CO₃ alone, and no hydrocarbons were detected. Further tests were performed at 280–340°C and 1.5–3.0 MPa. Each catalyst was tested for at least 24 h. The FeK-1.5 sample was tested for 100 h to evaluate long-term stability.

2.3 Measurement Methods and Quality Control

Crystal phases were identified by X-ray diffraction with Cu K α radiation over a 2 θ range of 10–80°. Mössbauer spectra were collected at room temperature before and after reaction. The spectra

were used to measure the relative amounts of Fe₂O₃, Fe₃O₄, Fe₃C, and χ -Fe₅C₂. CO₂ temperature-programmed desorption was measured after CO₂ adsorption at 50 °C. The samples were then heated to 600°C at 10°C min⁻¹ in He. H₂ temperature-programmed reduction was carried out from 50 to 700°C in 10 vol% H₂/Ar. In situ X-ray photoelectron spectroscopy was used after reduction and during CO₂/H₂ exposure. The measurements followed changes in Fe, K, O, and surface carbon species. Binding energies were corrected using the C 1s peak at 284.8 eV. Gas products were measured by online gas chromatography with thermal-conductivity and flame-ionization detectors. Hydrocarbons were grouped as CH₄, C₂–C₄ olefins, C₂–C₄ paraffins, and C₅⁺ products. Gas flow meters were calibrated before each test series. Carbon balances were kept between 95% and 105%. Each catalytic test was repeated three times, and tests with a relative error above 5% were repeated.

2.4 Data Processing and Model Equations

XRD patterns were corrected for background before phase analysis. Mössbauer spectra were fitted with doublet and sextet components. CO₂-TPD peaks were divided into weak, medium, and strong adsorption regions according to the desorption temperature. Peak areas were normalized by catalyst mass. CO₂ conversion was calculated as follows:

$$X_{\text{CO}_2} = \frac{F_{\text{CO}_2,\text{in}} - F_{\text{CO}_2,\text{out}}}{F_{\text{CO}_2,\text{in}}} \times 100\%$$

where $F_{\text{CO}_2,\text{in}}$ and $F_{\text{CO}_2,\text{out}}$ are the inlet and outlet molar flow rates of CO₂, respectively. The carbon-based selectivity to C₂–C₄ olefins was calculated as

$$S_{\text{C}_2\text{--C}_4 \text{ olefins}} = \frac{\sum n_i F_i}{\sum n_j F_j} \times 100\%$$

where F_i is the molar flow rate of each C₂–C₄ olefin, n_i is its carbon number, and $\sum n_j F_j$ is the total carbon flow of all hydrocarbon products. The olefin-to-paraffin ratio was calculated from the carbon-based flow rates of C₂–C₄ olefins and paraffins. The relation between catalyst properties and olefin selectivity was examined using the following regression model:

$$S_{\text{olefin}} = \beta_0 + \beta_1 A_{\text{CO}_2,\text{med}} + \beta_2 f_{\text{Fe}_5\text{C}_2} + \beta_3 U_{\text{H}_2} + \varepsilon$$

where $A_{\text{CO}_2,\text{med}}$ is the amount of medium-strength CO₂ adsorption sites, $f_{\text{Fe}_5\text{C}_2}$ is the χ -Fe₅C₂ fraction, U_{H_2} is the H₂ uptake, and ε is the error term.

2.5 Kinetic and Statistical Analysis

Kinetic tests were started after the product distribution remained stable for at least 4 h. Apparent activation energies were calculated from hydrocarbon formation rates measured between 280 and 320°C. CO₂ conversion was kept below 15% by increasing the gas flow rate. This condition reduced heat- and mass-transfer effects. Reaction orders for CO₂ and H₂ were measured by changing one partial pressure while keeping the total pressure constant with N₂. Internal diffusion was checked with catalyst particles of 150–250, 250–425, and 425–600 μm . No clear rate difference was found between the two smaller particle ranges. Pearson correlation analysis was used to compare light-olefin selectivity with K loading, medium-strength CO₂ adsorption, χ -Fe₅C₂ fraction, and H₂ uptake. Differences among catalyst groups were tested by one-way analysis of variance followed by Tukey's test. A value of $p < 0.05$ was considered significant. Results are reported as the mean $\pm$ standard deviation from three independent runs.

3. Results and Discussion

3.1 Effect of Potassium Content on Catalytic Performance

Potassium loading strongly affected CO₂ conversion and hydrocarbon distribution. The K-free

FeK-0 catalyst gave a CO₂ conversion of 22.7%, a C₂–C₄ olefin selectivity of 23.8%, and an olefin/paraffin ratio of 1.7 at 320 °C and 2.5 MPa. Increasing the K content from 0.5 to 1.5 wt% improved CO₂ conversion and shifted the products from methane and paraffins toward light olefins. FeK-1.5 gave the highest CO₂ conversion of 34.8% and a C₂–C₄ olefin selectivity of 42.6%. Its olefin/paraffin ratio reached 5.3. Methane selectivity decreased from 21.4% for FeK-0 to 10.2% for FeK-1.5. This decrease suggests that moderate K loading limited excessive hydrogenation (Jiao et al., 2026b; Mishra & Mandal, 2026b). FeK-2.5 gave a slightly higher olefin/paraffin ratio, but its CO₂ conversion and total hydrocarbon rate were lower. Excess K likely covered part of the Fe surface and reduced the number of sites available for H₂ activation and chain growth. Thus, CO₂ conversion followed a volcano-shaped trend with K loading, while the olefin/paraffin ratio increased steadily. Ait El Fakir et al. (2025) also found that Fe phase composition and surface carbon content affected olefin formation over promoted Fe catalysts. Jiao et al. (2026) reported that promoter contents above the optimum level did not further improve CO₂ conversion. The effects of K loading and reaction temperature on product distribution are shown in Fig. 1.

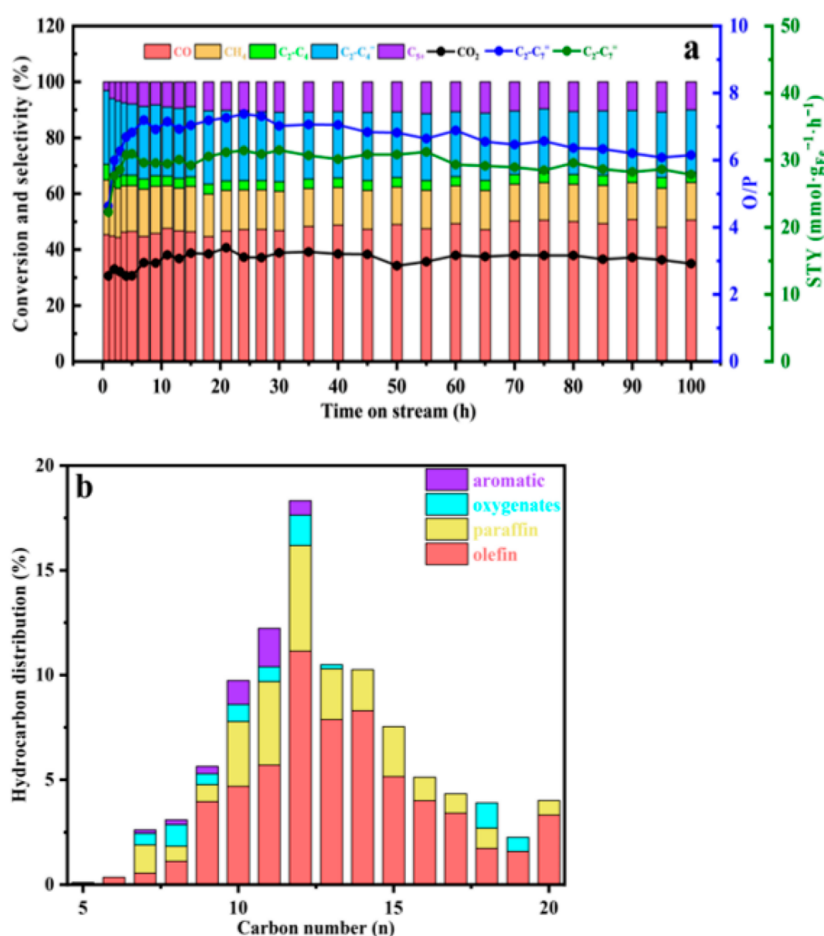


Fig. 1. Effect of K loading and reaction temperature on CO₂ conversion and hydrocarbon selectivity.

3.2 CO₂ Adsorption and Hydrogen Activation

The CO₂-TPD profiles contained weak, medium, and strong desorption regions. FeK-0 mainly showed weak adsorption sites below 180°C. K addition increased the medium-strength region between 180 and 380°C. FeK-1.5 contained 68.2% more medium-strength sites than FeK-0. These sites retained CO₂ at the reaction temperature and supplied carbon species for the reverse water–gas shift reaction. FeK-2.5 contained more strong adsorption sites above 380°C. However, part of

the adsorbed CO₂ may have remained as stable carbonate species. The H₂-TPR results showed that moderate K loading slightly promoted the first reduction step from Fe₂O₃ to Fe₃O₄. By contrast, the FeO-to-Fe reduction peak shifted from 438 °C for FeK-0 to 476 °C for FeK-2.5. The H₂ uptake of FeK-2.5 was 27.6% lower than that of FeK-1.5. Excess K therefore increased surface basicity but weakened H₂ activation. Moderate K loading provided enough CO₂ adsorption while keeping sufficient active hydrogen for hydrocarbon formation. At higher K contents, the lower hydrogen supply slowed the conversion of CO-derived species. Previous studies also found that promoter loading must balance CO₂ adsorption and surface hydrogen coverage. Too much promoter may raise olefin selectivity but lower total olefin yield because of reduced conversion (Karnaukhov et al., 2025; Yang, 2026b).

3.3 Evolution of Iron Oxide and Carbide Phases

The fresh catalysts mainly contained α -Fe₂O₃. After reduction, Fe₃O₄ and a small amount of metallic Fe were detected. The spent catalysts contained Fe₃O₄ and iron carbides. Their XRD peaks partly overlapped between 40° and 46°, which made accurate phase analysis difficult. Mössbauer spectroscopy gave a clearer phase distribution. The χ -Fe₅C₂ fraction increased from 18.6% for FeK-0 to 46.8% for FeK-1.5. At the same time, the Fe₃O₄ fraction decreased from 61.2% to 38.5%. FeK-2.5 contained 49.1% χ -Fe₅C₂, but its hydrocarbon rate was lower than that of FeK-1.5. A high carbide fraction alone was therefore not enough to maintain high activity. Open Fe sites and active hydrogen were also required. In situ XPS showed that the Fe 2p_{3/2} peak shifted slightly toward lower binding energy after K addition. This shift suggests electron transfer from K to surface Fe species. The carbide-related C 1s signal increased during the first 6 h of reaction and then remained stable. The phase changes are presented in Fig. 2. Jiao et al. (2026) reported that CO- and syngas-pretreated Fe catalysts formed more iron carbide than H₂-pretreated samples and gave higher olefin yields. Guérette et al. (2025) also found that Fe₃O₄ mainly supported CO formation, while Fe₅C₂ promoted hydrocarbon chain growth.

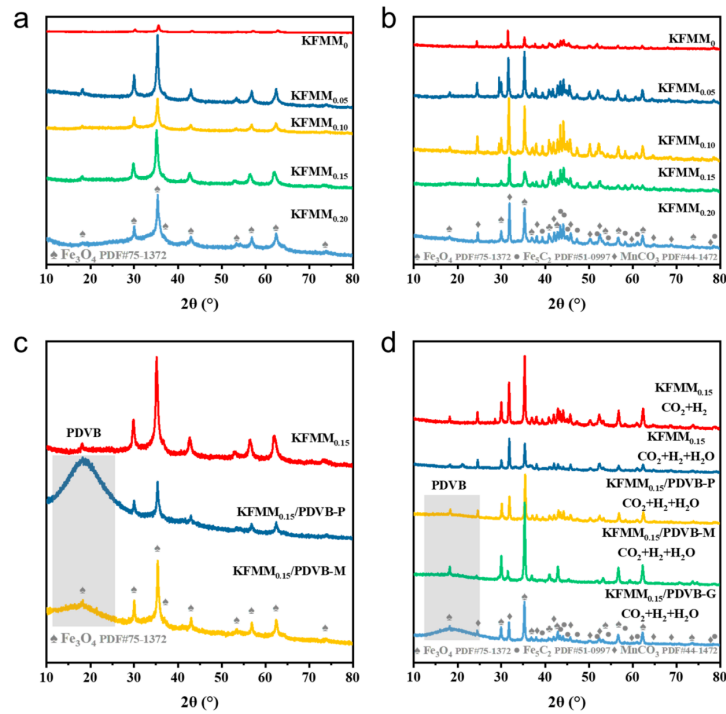


Fig. 2. Iron phases, CO₂ adsorption, and surface composition of the K-promoted Fe catalysts.

3.4 Relation between Catalyst Structure and Olefin Formation

C₂–C₄ olefin selectivity was compared with CO₂ adsorption, H₂ uptake, and iron phase composition. Olefin selectivity increased with the number of medium-strength CO₂ adsorption sites, with an R² value of 0.86. A stronger relation was found between olefin selectivity and the χ -Fe₅C₂ fraction, with R²=0.91. Total hydrocarbon productivity decreased at high K loading and increased with H₂ uptake. Multiple regression showed that the χ -Fe₅C₂ fraction and medium-strength CO₂ adsorption had positive effects on olefin selectivity. H₂ uptake promoted total hydrocarbon formation. However, excessive surface hydrogen lowered the olefin/paraffin ratio by promoting secondary hydrogenation. FeK-1.5 gave the best balance among these factors. Fe₃O₄ converted CO₂ to CO, while χ -Fe₅C₂ supported CO dissociation and C–C bond formation. Potassium increased surface basicity, promoted carbide formation, and reduced further hydrogenation of the formed olefins. Excess K blocked part of the Fe surface and limited H₂ activation. FeK-1.5 retained 93.6% of its initial hydrocarbon rate after 100 h. Mössbauer analysis of the spent catalyst showed only a small decrease in χ -Fe₅C₂ content. The results suggest that both the Fe₃O₄/ χ -Fe₅C₂ ratio and local K coverage must be controlled. Similar phase effects have been reported for other promoted Fe catalysts, although pretreatment conditions, feed composition, and water content can change the final oxide–carbide balance (Gu, 2026; Rolandi et al., 2025).

4. Conclusion

Potassium changed the phase structure and product distribution of Fe-based catalysts during CO₂ hydrogenation to light olefins. The catalyst with 1.5 wt% K gave the best performance, with a CO₂ conversion of 34.8%, a C₂–C₄ olefin selectivity of 42.6%, and an olefin/paraffin ratio of 5.3 at 320°C and 2.5 MPa. Potassium increased the number of medium-strength CO₂ adsorption sites by 68.2% and promoted the formation of χ -Fe₅C₂. This phase supported CO dissociation, C–C bond formation, and hydrocarbon chain growth. A moderate K content also reduced the further hydrogenation of olefins. However, excessive K covered part of the Fe surface, weakened H₂ activation, and lowered hydrocarbon production. This work links K loading with CO₂ adsorption, iron carbide formation, and light-olefin selectivity in the same catalyst series. The results may help improve Fe-based catalysts by balancing carbon supply and surface hydrogen. These catalysts may be useful for producing light olefins from captured CO₂ and low-carbon hydrogen. The present study was limited to laboratory-scale tests and a narrow range of reaction conditions. Longer stability tests, larger catalyst batches, and tests with recycled gas and water-containing feeds are still needed.

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