

An Introductory Study about CO₂ Hydrogenation into Hydrocarbons Using Iron Catalysts

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Abstract

CO₂ hydrogenation reaction was performed on precipitated iron catalysts which were promoted by Si, Zn, K and Cu. The optimum SiO₂ content in the catalysts is about 15 wt% relative to Fe₂O₃ mass. With reaction temperature raised, CO₂ conversion is increased continually, but CO and CH₄ selectivity only fluctuate in a narrow range which is beneficial to the synthesis of C₂+ hydrocarbons. Two kinds of catalyst filling constitution were experimentally compared in order to increase the yield of C₅+ hydrocarbons.

Keywords

CO₂ Hydrogenation, Hydrocarbon Synthesis, Iron Catalyst

1. Introduction

Greenhouse effect is recognized as one serious threat to the environment. It would increase the global average temperature and make the sea level raised [1]. As the results, the land supporting billions of people would be submerged. CO₂ is a main factor for the heightening greenhouse effect, because it contributed about 64% to the total radiative forcings from the long-lived and well mixed greenhouse gases (CO₂, CH₄, N₂O and halogenated compounds) in 2011 [2]. Over 32.1 Gt CO₂ was emitted from fossil fuel combustion in 2013 [3]. To lessen fossil fuel consumption is a direct and effective method to decrease CO₂ emission, however, it can only be realized when sustainable fuels are supplied to replace the fossil fuels. Methanol, dimethyl ether and hydrocarbons are the products of CO₂ hydrogenation [4]-[12] which are able to be used directly as fuels. In case H₂ is generated from renewable energy source such as biomass or solar energy [4] [5] [13], the process of CO₂ hydrogenation is CO₂-neutral besides to supply sustainable fuels. Such process will solve fuel lack of isolate islands by recovering CO₂ from seawater [14].

On iron catalyst, CO₂ can be hydrogenated into hydrocarbons by two steps as shown in the following Equation (1) and (2) [9] [10] [11]. CO₂ is converted into CO by reverse water-gas-shift (WGS) reaction, and then the produced CO is further hydrogenated to hydrocarbons by Fischer-Tropsch (FT) reaction.



WGS reaction is thermodynamically favored at low temperature [15]. Therefore, reverse WGS reaction is favored at high temperature, *i.e.* high temperature can promote CO₂ conversion to hydrocarbons via the series of Equations ((1) and (2)). In order to find iron catalyst more active for CO₂ hydrogenation, we researched the influences of Zn, K and Cu on precipitated iron catalysts at low temperature (230°C) [12] [16] [17]. The functions of Zn and K to activate CO₂ into CO were explained based on the experimental data from CO₂ temperature-programmed desorption [16]. Recently, the catalysts were evaluated at increased reaction temperature, and the catalyst filling constitutions in the reactor were compared in order to increase hydrocarbon yield as high as possible.

2. Experimental

2.1. Catalyst Preparation

All the used reagents are analytical grade (Sinopharm Chemical Reagent Co., Ltd.). Catalysts were prepared in two methods. Method I was to precipitate a mixed solution of Fe(NO₃)₃ and potassium silicate with (NH₄)₂CO₃ solution at 50°C and pH = 6.5 ± 0.5. Method II was to precipitate Fe(NO₃)₃ solution with the mixed solution of (NH₄)₂CO₃ and potassium silicate at 50°C and pH = 6.5 ± 0.5. Then, the precipitate was washed with distilled water and centrifuged at 4500 RPM for three times after 1 h ageing at room temperature. Promoters of Zn, K and Cu were impregnated onto the precipitate with Zn(NO₃)₂, KNO₃, and Cu(NO₃)₂ solution. After the catalyst precursor was spray-dried at 250°C and calcined at 360°C for 6 h, it was shaped into particle of 80 - 150 μm for activity test. The obtained catalysts are expressed as *ZK_mC_n/FS_r*-I or *ZK_mC_n/FS_r*-II according to the method to introduce SiO₂. In the above abbreviation of catalysts, Z, K, C, F, and S represent Zn, K, Cu, Fe, and SiO₂, respectively. *L*, *m*, *n*, and *r* are the nominal mass percent of corresponding materials relative to Fe₂O₃.

2.2. Characterizations

BET surface area, pore volume and average porediameter of the catalysts were measured using ASAP-2020 from Micromeritics at -196°C. The crystal structure of catalysts was analyzed by PNAlytical diffractometer (X'Pert Pro) with a Cu Kα radiation source (λ = 0.15406 nm).

2.3. Activity Test and Product Analysis

The activity of catalysts was tested in a stainless steel fixed bed reactor [12]. The catalyst diluted with four-fold quartz particle of equal size was filled into the reactor. After the catalysts were reduced in CO of 50 mL·min⁻¹ at 300°C for 6 h, it was cooled to room

temperature. Then, the feed gas was changed into reactants (64% H₂/32%CO₂/4%N₂) of 1.6 MPa. The catalyst was heated to reaction temperature in about 3 h and kept at it for 45 h. The detail parameters for activity test are given with the experimental results.

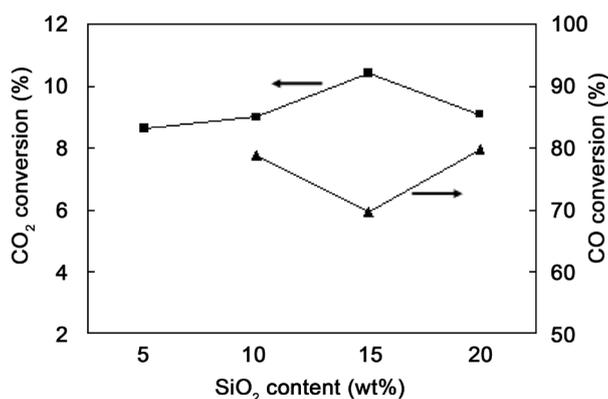
C₅+ hydrocarbons were collected in an ice trap of 0°C at system pressure. The distribution of C₅+ hydrocarbons was measured off line by GC-9860 (Qiyang Ltd.) with FID detector and OV-1701 capillary column. After the system pressure was released through a backpressure regulator, the exited gas was analyzed on line. The quantities of CO, CH₄, CO₂ and N₂ were supplied with TCD detector and TDX-01 column. N₂ was used as an internal standard for the quantitation with the GC/TCD. C1 - C5 hydrocarbons were analyzed with FID detector and Al₂O₃ capillary column [18].

3. Results and Discussion

3.1. Influence of SiO₂ Content in Catalyst

Different to the iron catalysts studied in our previous works [19] [20] [21], SiO₂ is used as structure promoter in this work. **Figure 1** contrasts the influence of SiO₂ content on the catalysts' activity for both of CO₂ hydrogenation (CO₂ conversion) and CO hydrogenation (CO conversion). These catalysts were prepared in Method I, and the content of Zn, K and Cu is 8 wt%, 5 wt% and 4 wt%, respectively. The catalyst with 15 wt% SiO₂ is the most active one for CO₂ hydrogenation, while this catalyst shows the lowest CO conversion in CO hydrogenation.

Table 1 gives the data of catalysts' texture. With raised SiO₂ content in the catalysts,



For CO₂ hydrogenation: H₂/CO₂ = 2, 230°C, 1.6 MPa, 6.0 L·h⁻¹·g·cat.⁻¹.

For CO hydrogenation: H₂/CO = 1.5, 235°C, 1.6 MPa, 3.0 L·h⁻¹·g·cat.⁻¹.

Figure 1. Influence of SiO₂ content on catalyst activity in CO₂ hydrogenation and CO hydrogenation.

Table 1. BET surface area and pore size.

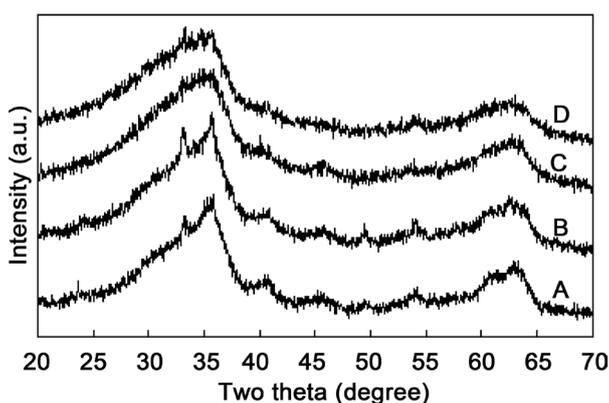
Catalyst	Surface area (m ² /g)	Pore volume ^a (mL/g)	Average pore diameter ^a (nm)
Z8K5C4/FS5-I	72.9	0.117	6.4
Z8K5C4/FS10-I	94.7	0.182	7.7
Z8K5C4/FS15-I	101.4	0.230	9.1
Z8K5C4/FS20-I	131.2	0.322	9.8

^aIt is calculated by BJH method from the desorption branch.

the specific surface area, pore volume and average pore diameter increase monotonically which do not match to either the volcano shape of CO₂ conversion or reverse volcano shape of CO conversion in **Figure 1**. The enlarged surface area usually reflects increased dispersive degree of every component in the catalysts.

Figure 2 is the XRD results of the four catalysts. There are peaks around 23.92°, 33.48°, 35.88°, 40.72°, 49.68°, 54.08°, 62.76° and 63.76° in Z8K5C4/FS5-I, Z8K5C4/FS10-I and Z8K5C4/FS20-I. They are assigned to Fe₂O₃ (JCPDS 89-0597). These peaks almost disappear in Z8K5C4/FS15-I. The disappearance of crystalline Fe₂O₃ indicates that iron is highly dispersed by the added SiO₂ in Z8K5C4/FS15-I. This dispersing effect of SiO₂ is happened to the promoters, too. It has been reported that the amount of effective potassium is decreased by the addition of SiO₂ into iron catalysts because of K-Si interaction [22] [23]. Therefore, the amount of effective potassium is probably the lowest in Z8K5C4/FS15-I. It results in effective Zn/K ratio higher than the nominal Zn/K ratio calculated by the added amounts of zinc and potassium. We have disclosed that the iron catalyst with higher Zn/K ratio is more active to hydrogenate CO₂ [12] [16].

Table 2 lists the ethene and propene ratio in the C₂ and C₃ hydrocarbons (sum of olefin and paraffin), respectively. For CO hydrogenation, both of the ethene and propene content are almost constant in spite of the SiO₂ content is different for the studied catalysts. It indicates that the ratio of adsorbed carbon atom (C_{ad}) to adsorbed hydrogen atom (H_{ad}) [24] is independent of the SiO₂ content in catalysts. Contrarily, for CO₂ hydrogenation, the ethene and propene content decrease with SiO₂ content raised in the studied catalysts. It reflects that the ratio of C_{ad} to H_{ad} is declined by the added SiO₂ for CO₂ hydrogenation. According to the two steps to hydrogenate CO₂ into hydrocarbons



A: Z8K5C4/FS5-I, B: Z8K5C4/FS10-I, C: Z8K5C4/FS15-I, D: Z8K5C4/FS20-I

Figure 2. XRD pattern of the catalysts with different SiO₂ content.

Table 2. Olefin ratio in C₂ and C₃ hydrocarbons.

Catalyst	CO + H ₂		CO ₂ + H ₂	
	C ₂ ⁼ /C ₂	C ₃ ⁼ /C ₃	C ₂ ⁼ /C ₂	C ₃ ⁼ /C ₃
Z8K5C4/FS10-I	0.78	0.82	0.47	0.62
Z8K5C4/FS15-I	0.79	0.81	0.25	0.50
Z8K5C4/FS20-I	0.78	0.81	0.22	0.48

The reaction conditions are as same as those shown in the caption of **Figure 1**.

[9] [10] [11], the C_{ad} is from the CO which was evolved from CO_2 , rather than directly from the adsorbed CO_2 . On the same iron catalyst, the H_{ad} quantity in CO_2 hydrogenation is probably as same as in CO hydrogenation. Therefore, it can be thought that the co-adsorption of CO_2 and CO on iron catalyst induces decreased C_{ad} . However, it is only an assumption at present. We are trying to measure the C_{ad} and H_{ad} quantity after CO and H_2 or CO_2 and H_2 are co-fed to the iron catalysts.

3.2. Influence of Reaction Temperature

Catalyst Z2K3C4/FS5-I was evaluated under different reaction temperature. The results are given in **Table 3**. CO_2 conversion is increased continually with raised reaction temperature. With reaction temperature increased, the equilibrium of Equation (1) shifts to the right which is beneficial to supply CO for FT reaction according to Equation (2). It had been found that the extent of CO converted to hydrocarbons increased with raised reaction temperature [22] [25]. Both of CO selectivity and CH_4 selectivity show an evident decrease from 230°C to 240°C, but there is no monodirectional change under higher temperature. The ethene content in C_2 hydrocarbons increases with raised reaction temperature. Under high temperature, the residence time of adsorbed ethene on the catalysts would be shortened due to the enhanced thermal motion. It leads to decreased probability for the adsorbed ethene to initiate carbon-chain growth or be hydrogenated into ethane.

The observed phenomenon about ethene selectivity in **Table 3** is different from the results found in CO hydrogenation, where the ethene selectivity was decreased with increased reaction temperature [25] [26]. Combining the above difference between CO_2 hydrogenation (**Table 3**) and CO hydrogenation [25] [26] with the disparity shown in **Table 2**, much research is needed to understand the reactive mechanism of CO_2 hydrogenation on iron catalyst.

3.3. Comparison of Catalyst Filling Constitution in the Reactor

For CO_2 hydrogenation in a fixed bed reactor, the H_2O and hydrocarbons produced in the upper part of catalyst bed can absorb on the downstream catalyst, which probably influence the reaction of remained CO_2 and H_2 on the downstream catalyst. The influences are examined using catalyst Z8K3C6/FS5-I and Z8K3C6/FS10-II. Two constitutions are adopted to fill the catalysts as described in **Figure 3**. Constitution I is to install

Table 3. Influence of reaction temperature on CO_2 hydrogenation.

Temperature (°C)	CO_2 conversion (%)	CO selectivity (%)	CH_4 selectivity (%)	$C_2^=/C_2$
230	10.7	42.8	22.0	0.57
240	15.4	35.1	17.4	0.61
250	16.6	34.2	18.3	0.66
260	18.3	32.5	17.0	0.70
270	20.3	35.7	19.8	0.72
280	22.5	35.2	15.6	0.73

$H_2/CO_2 = 2$, 1.6 MPa, 6.0 L·h⁻¹·g-cat.⁻¹

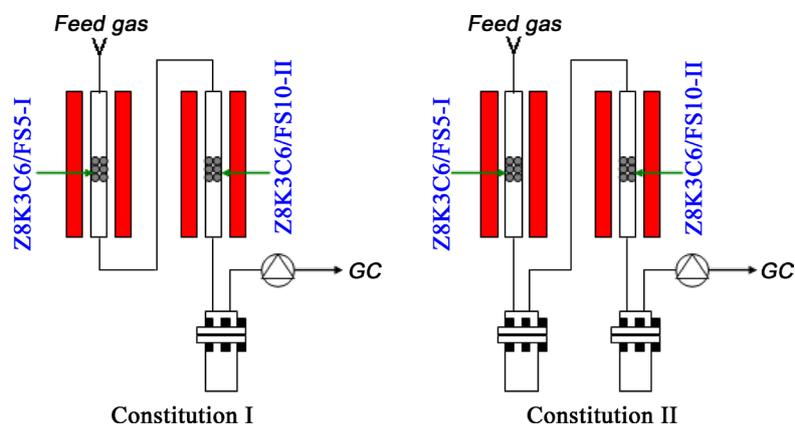


Figure 3. Principal scheme of catalyst filling constitutions.

Table 4. CO₂ hydrogenation with different catalyst filling constitution.

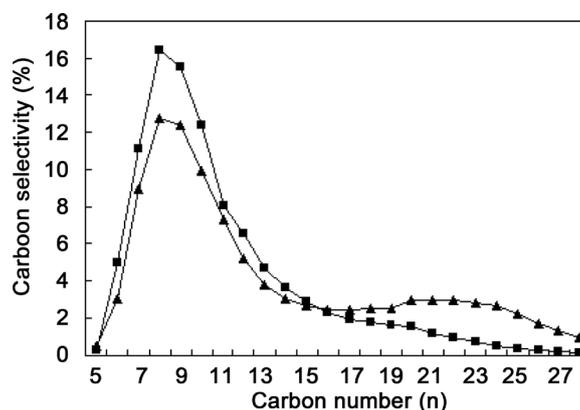
Constitution	I	II
CO ₂ conversion (%)	27.6	35.8
CO selectivity (%)	17.6	16.5
CH ₄ selectivity (%)	22.2	27.3
C ₂ - C ₄ selectivity (%)	27.5	31.7
C ₂ ⁼ /C ₂	0.30	0.17
C ₅ + yield (%)	9.03	8.77

H₂/CO₂ = 2, 280°C, 1.6 MPa, 3.0 L·h⁻¹·g·cat.⁻¹

Z8K3C6/FS10-II directly to the downstream of Z8K3C6/FS5-I, on the contrary, Constitution II is to insert one 0°C trap between Z8K3C6/FS5-I and Z8K3C6/FS10-II to collect H₂O and condensable hydrocarbons produced by the upper Z8K3C6/FS5-I.

Table 4 contrasts the reaction data from the two constitutions. Relative to the results of Constitution I, CO₂ conversion, CH₄ selectivity and C₂ - C₄ selectivity are increased in Constitution II. For Constitution I, the H₂O and long-chain hydrocarbons produced by catalyst Z8K3C6/FS5-I may adsorb on Z8K3C6/FS10-II. Their re-adsorptions on Z8K3C6/FS10-II occupy some sites which can activate CO₂. Such occupancy inhibits CO₂ activation and reaction on Z8K3C6/FS10-II. The occupancy also impairs H₂ adsorption on Z8K3C6/FS10-II, which is indicated by the lower CH₄ selectivity and higher ethene ratio in C₂ hydrocarbons for Constitution I than Constitution II. However, it is beneficial for carbon-chain growth to produce long chain hydrocarbons. As listed in **Table 4**, Constitution I has a higher C₅+ yield than Constitution II.

Figure 4 compares the distribution of liquid hydrocarbons from the two Constitutions. There is an increase from C₅ hydrocarbons to C₈ hydrocarbons. It does not mean that the selectivity of C₅, C₆ and C₇ hydrocarbons is less than C₈ hydrocarbons, because some molecule of C₅ - C₇ hydrocarbons cannot be completely condensed in the cold trap and are emitted with the exited gas. The C₅ - C₇ hydrocarbons had been found by the on line GC/FID designed to analyze the gaseous hydrocarbons. The hydrocarbon selectivity of FT reaction usually decreases with elongated carbon chain [27] [28]. However, the selectivity for the hydrocarbons in C₂₀ - C₂₄ is higher than that of C₁₅ - C₁₉



$H_2/CO_2 = 2$, $280^\circ C$, $1.6 MPa$, $3.0 L \cdot h^{-1} \cdot g \cdot cat^{-1}$

▲: Constitution I, ■: Constitution II.

Figure 4. Influence of catalyst filling constitution on liquid hydrocarbons distribution.

hydrocarbons for Constitution I. This phenomenon may result from the olefin re-adsorption [27] which enhances the hydrocarbon growth into long carbon chain.

Considering the simplicity of reactor structure, Constitution I is an optimum type to synthesize liquid hydrocarbons from CO_2 hydrogenation.

4. Conclusion

Iron catalysts were prepared by co-precipitation of Fe and Si. Zn, K and Cu were impregnated to FeSi precipitate as promoters. For CO_2 hydrogenation reaction, the catalyst with 15 wt% SiO_2 possesses the highest CO_2 conversion. The olefin content in C_2 and C_3 hydrocarbons is lower in CO_2 hydrogenation than in CO hydrogenation which may be influenced by the co-adsorption of CO_2 and CO on the catalyst. CO_2 conversion and ethene selectivity are increased with raised reaction temperature. For fixed bed reactor, the H_2O and hydrocarbons produced in the upper catalyst may re-adsorb on the downstream catalyst, and it is beneficial for carbon-chain growth by inhibiting H_2 adsorption.

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