Bimetallic Fe-Cu catalysts for selective hydrogenation of CO_2 to olefin-rich C_2+ hydrocarbons

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ABSTRACT

Carbon dioxide is an important pollutant that has recently led to deterioration of the environment. In particular, CO_2 can be used not only as a greenhouse gas, but also as a source of carbon for the production of a number of high-quality products, such as light olefins. Lower olefins (C_2H_4 and c3h6) are the main petrochemical building blocks used to produce various chemicals.

Keywords: hydrogenation, olefins, diffraction, catalyst, nickel, catalytic, Composite, pH value of solution, conversion, selectivity.

The direct nutrition of light olefins from CO_2 hydrogenation has generated considerable interest in its important role in reducing CO_2 emissions. We study a significantly efficient, robust and multifunctional NiCu/CeO_2-YUKS catalyst here, with CO_2 selectivity up to 78.2% (C_2H_4 ¼ 22.7%, C_3H_6 ¼ 35.5% and c4h8 %) ¼ 18 .It is able to convert 4 directly into light olefins. Under Optimal reaction conditions, CO selectivity is below 65%. The physico-chemical description of the catalyst was carried out using the methods BET, NH3-TPD, h2-TPR, XRD, TEM and SEM. Compared to the X-ray diffraction patterns of YUKS and NiCu/CeO_2, all characteristic X-rays of both compositional samples showed diffraction peaks. In addition, our developed hybrid catalyst also has useful catalytic stability, it can work for 90 hours without loss of specific activity.

1. Introduction

Carbon dioxide is an important pollutant that has recently led to deterioration of the environment. In particular, CO_2 can be used as a carbon source to produce not only greenhouse gas, but also a number of high-quality products such as light olefins [1-7] lower olefins (C_2H_4 and C3H6) are the main petrochemical building blocks used to produce a variety of chemicals [8-13] however, carbon dioxide is certainly a stable molecule due to the double bonds between carbon and oxygen, making it chemically inert. Accordingly, CO_2 activation appears to be a significant success in the use of carbon dioxide. As for the hydrogenation mechanisms of CO_2 to light olefins, two reaction pathways are usually recognized: MTO (methanol-mediated reaction) and modified Fisher-Tropsch synthesis (CO-mediated reaction) in the modified Fisher-Tropsch synthesis method, CO_2 is first converted to carbon monoxide using reverse water gas shift (RWGS) and then hydrogenated to lower olefins using the modified Fisher-Tropsch synthesis method [14-19]. n the methanol process, CO_2 and H_2 are absorbed by a hybrid catalyst to synthesize methanol and then converted to light olefins [20-25]. The selectivity of olefins in modified FT catalysts is usually less than 60%, as the Anderson-Shults-fluorine distribution is limited [26]. We expect the methanol assistance process (MTO) to overcome this constraint using a two-function K+atalizer. At inzr-YUKS, olefin selectivity at 9 L g 1 h 1, 380 ° C and 20 bar in space, CO_2 conversion reached 74.5% with 26.2%. When using ZnZr - YUKS, the CO_2 conversion was 13% with 80% selectivity at a temperature of 380 °C and a pressure of 20 bar [27]. The introduction of metals such as nickel-bonded copper is another way to synthesize catalysts for the synthesis of CH_3OH from CO_2 [28,29]. Due to its unique properties, transition metals are often used in heterogeneous catalytic processes compared to pure transition metals [30]. Ni-Cu alloys are currently of great interest for several chemical processes such as $CH_4/C_2H_5OH/C_2H_6O$ vapor processing, CH_4 hydrogenation, and decay+ processes using carbon monoxide, carbon dioxide, and c4h6o4. +[29,31-34]. At the same time, the use of metal oxides (Al_2O_3, CeO_2, MnO_2 and La_2O_3) as a base has been documented to improve catalytic selectivity as well as activity [30,35-38]. For example, the CeO_2-based Ni catalyst showed high selectivity (99%) due to the electron properties of Ni modified with CeO_2 through strong interaction between metal and base. [36] it is known that C-C bonds, such as lower olefins, may occur in the cexite catalyst during methanol conversion [39-42]. Zeolites are already widely used as completely useful adsorbents, catalysts and ion exchangers in various fields [43-51]. Among these zeolites, YUKS showed perfect catalytic indicators due to its special CHA cage frame (9.4 Å) [52], as well as its moderate acidity [43.4-6.53], which allows it to exhibit perfect catalytic indicators [39] , 40.54]. In this work, we first developed a highly efficient hybrid catalyst of NiCu / CeO_2 and YUKS zeolite that converts CO_2 directly into lower olefins. In this BiFunction catalyst, methanol produced on the surface of NiCu/CeO_2 can be converted to in situ downstream olefins in a porous cargo zeolite. The effect of working conditions consisting of gas space velocity (SV), reaction temperature, and H_2 / CO_2 ratio on the conversion of CO_2 to lower olefins has been extensively studied. In addition, the stability of hybrid catalysts was measured in the best cases.

2. Experience

2.1. Katalizator synthesis

2.1.1. NiCu / CeO_2 synthesis

In a special preparation, the NICU / CeO_2 catalyst is synthesized by the co-deposition method. First, 12 mmol Ce(No. 3) was dissolved in 3.6 H_2O distilled water (DV). Then, 0.05 M solutions of 15% KOH (by mass) and Ni(No_3)2 and Cu(No_3)2 were applied simultaneously at a temperature of 85 ° C and in constant mixing. The pH of the solution is maintained at 11. The resulting sediment was digested for 36 hours with stirring at 80 ° C. To remove the resulting solid potassium impurities, it is thoroughly washed with DW several times, then air-dried at 110 ° C for 10 hours and then calcined at 600 ° C for 5 hours.

2.1.2 Cargo synthesis

Crystal YUKS -34 was prepared hydrothermically using morpholine and TEAOH as templates. Sources of Si, Al, and P were silicic acid (SiO_2, Merck), aluminium isopropoxide (AIP, Merck), and phosphoric acid (H_3PO_4, Merck weighing 85), respectively. The gel composition was 1 Al_2O_3: 1P_2O_5: 0.6 SiO_2: 0.5 TEAOH, 0.5 MOR, 60 H_2O (molar base). The full explanation for the preparation of YUKS is given in our previous work [39 41,55-59]. The synthesized product was then dried and calcined at 550 ° C for 6 hours.

2.1.3 Gibrid NiCu / CeO2-YuQS

Hybrid composite NiCu/CeO_2: YUKS ¼ was prepared by volumetric mixing with a mass ratio of 1:2, and designated as NiCu / CeO_2 - YUKS.

2.2 Catalysts characterization methods

The Bruker D8 Advance instrument recorded diffraction patterns of X-rays with CU Ka radiation (l ¼ 1.5406 a). The particle size of the catalysts was determined using a transmission electron microscope using JEOL, JEM- electron microscopy. Microscopic morphology of the sample was observed using SEM, AIS 2100

(Seron Technology, South Korea). The specific surface area was studied using the Brunauer-Emmett-Teller (BET) equation using the multi-point method (Quantachrome Chem BET-3000). Microscopic morphology of the sample was observed using SEM, AIS 2100 (Seron Technology, South Korea). H_2-TPR measurements were carried out on the Micromerics AutoChem II 2920 instrument in the H_2-Ar mixture. Before return, the catalyst was pre-treated in airflow at 400 C for 2 hours and then cooled to 50 C. The H_2-TPR experiment then began by increasing the temperature to 800 °C at 10 °c per minute. The surface acidity of the catalysts was determined using NH_3-TPD in the Micromerics 2000 adsorption equipment.

2.3 Catalytic efficiency

In form. Figure 1 shows a laboratory scale scheme [60].

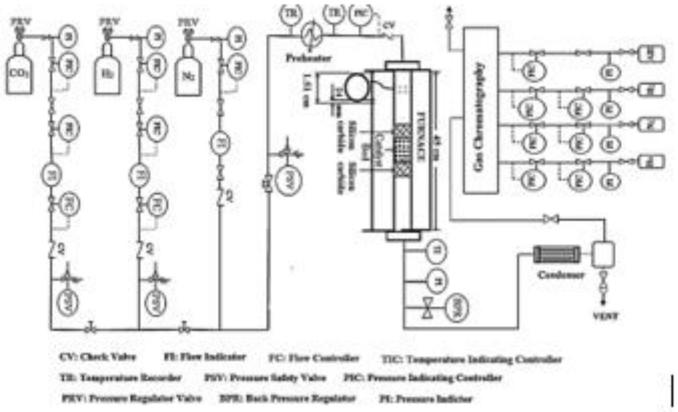


Figure 1. Schematic diagram of an experimental device for hydrogenating CO_2 to light olefins [60].

The efficiency of the catalyst was analyzed in a solid-bed reactor (D = 45 cm, ID = 1.51 CM). Typically, 1.0 g of the catalyst was placed in the reactor. The catalyst was pre-treated at 400 °C for 3 hours. Then the temperature was raised to the desired temperature to start the reaction. A gas mixture, including CO_2, H_2 and N_2, was sent to the reactor in a certain stoichiometric ratio. The products were analyzed using a Varian Chrompack CP 3800 gas chromatograph equipped with TCD and FID detectors. Activity tests were conducted at different temperatures ranging from 350 to 450 ° C, with gas field velocities ranging from 2 to 12 Lgcat 1 hour 1, H_2 / CO_2 ratio ranging from 1 to 5, pressure ranging from 20 bar and reaction time ranging from ¼ 10.

Conversion and selectivity are defined as follows:

$$CO_2 Conversion = \frac{CO_{2inlet} - CO_{2outlet}}{CO_{2inlet}} \times 100\% \qquad (1)$$

$$CO \;\; Selectivity = \frac{CO_{\,outlet}}{CO_{2inlet} - CO_{2outlet}} \times 100\% \qquad (2)$$

$$C_nH_m \; Selectivity = \frac{nC_nH_{moutlet}}{CO_{2inlet} - CO_{2outlet} - CO_{\,outlet}} \times 100\% \qquad (3)$$

3. Results and their discussion

3.1. Characteristic of catalysts

Figure 2 shows X-ray diffraction of various samples after calcination at 550 °C and after measurement at room temperature.

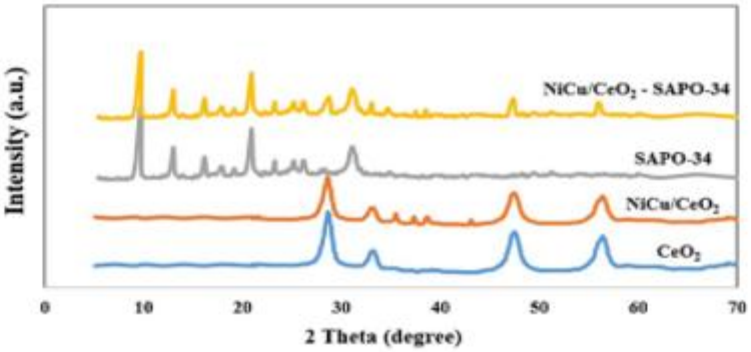


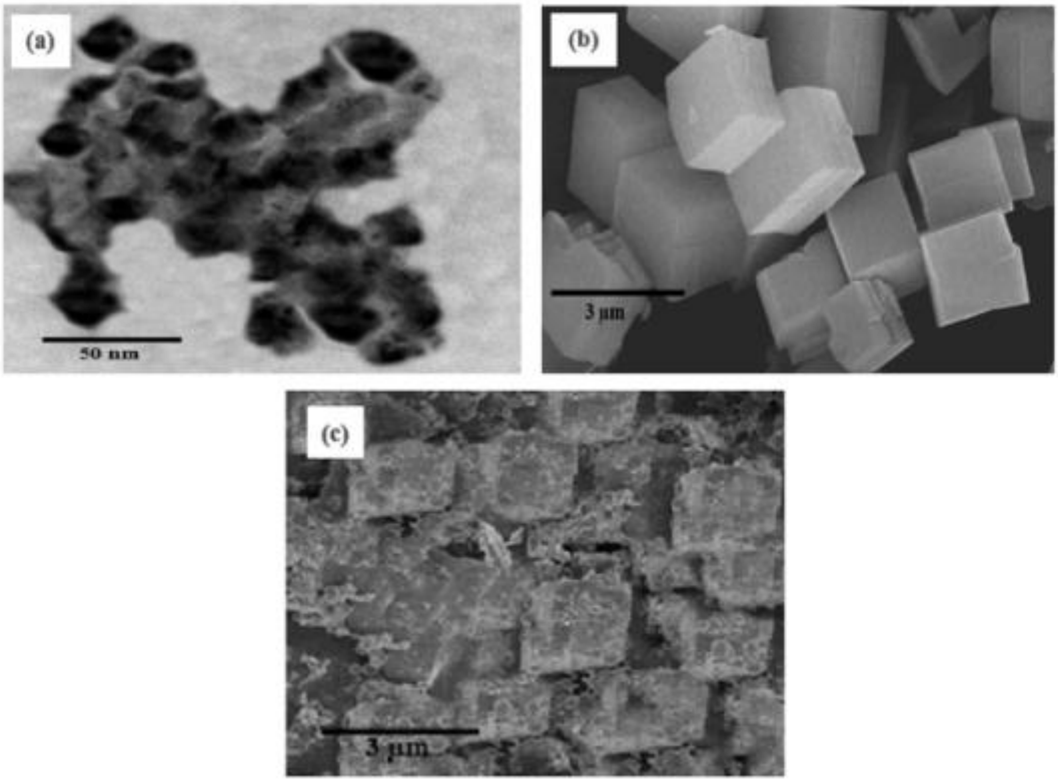
Figure 2. CeO$_2$, NiCu/CeO$_2$, YUKS, and NiCu/CeO$_2$ are diffraction lines of X - ray radiation from YUKS samples.

Diffraction peaks at 28,5, 33,2, 46,8 and 56,4 indicate the presence of a cerium fluorite-type structure in all samples [61-63]. In addition, phase characteristics of CuO (2 Theta = 35.3 and 39.1) and NiO (2 Theta = 37.2 and 43.1) have also been identified in a NiCu/CeO$_2$ oxide sample. This suggests that the sample synthesized by the co-deposition method contains cast oxides of both CuO and NIO. X-ray diffraction lines of synthesized cargo confirmed the structure of chabazite. It can be seen that the structure has 2 Theta = 9,6, 13,1, 16,1, 20,5, 24,9, 25,9, 30,7, it also has its highest positions at 31.2. Compared to Xrd samples of YUKS and NiCu/CeO$_2$, the composition showed all the characteristic XRD patterns of both samples. This suggests that the binary structure of the NiCu-CeO$_2$ / YUKS composition can be synthesized to physically mix synthetic M. Table 1 shows the crystallinity of all catalysts. The relative crystallinity of the samples was measured by the sum of the intensities of the most important peaks based on reference intensities. The crystallinity (87%) of the altered YUKS is lower than that of the synthesized YUKS (91%), which may be due to surface smoothness and reduced surface area (Table 1).

Table 1 physicochemical properties of all samples.

Sample	S_{BET} (m2 g^{-1})	V_{pora} (cm3 g^{-1})	Mean hole diameter (nm	Kristallini
CeO$_2$	57	0.14	9.45	94
NiCu/CeO$_2$	38	0.09	6.73	89
YUKS	427	0.36	0.47	91
NiCu/CeO$_2$ – YUKS	308	0.25	4.92	84

The specific surface area and pore size of CeO$_2$ and NiCu/CeO$_2$ are 57 (m^2g 1), 0.14 (sm^3g 1) and 38 (m^2g 1), 0.09 (sm^3g 1), respectively. The area of the BET YUKS surface (427 M2G-1) was reduced by mixing NiCu / CeO$_2$, which may be due to clogging of some channels and unmodified holes. The figure shows the distribution and distribution of 3A NiCu / CeO$_2$ powder. TEM tomonidan taxmin qilingan o'rtacha NiCu/CeO$_2$ zarracha hajmi tor taqsimot bilan taxminan 20 nm edi. Shaklda. 3b-rasm shuni ko'rsatadiki, YUKS namunasi taxminan bir xil o'lchamdagi xarakterli kubik kristallarga ega.



The synthesized YUKS exhibit regular cubic rhombedral morphology, almost identical to that of natural gabacite. SEM images of NiCu/CeO2 - YUKS (Figure 3 C) show a different morphology than YUKS, in which irregular crystals are observed on the surface. This suggests that the addition of NiCu / CeO2 significantly changes the normal morphology of YUKS, but the modified YUKS structure is almost identical to the YUKS structure. The two broad H2-TPR peaks, implemented at 388 and 758 ° C for Figure 4A, CeO_2, may be related to reducing surface coat oxygen, as well as mass oxygen, respectively [64,65]. In addition, a TPR profile of the NiCu / CeO_2 sample synthesized by the co-deposition method is provided.

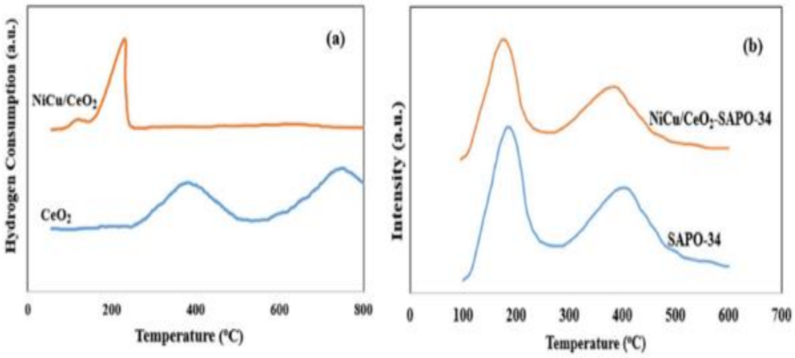


Figure 4. (a) H2-tpr profile and (B) NH3-TPD curve

Amorphous CuO, weakly interacts with CeO_2 support and reduces CuO, strongly interacts with the base. However, the shoulder peak above 200 °C was associated with a reduction in highly diffuse NiO [31,53,66-68]. image. 4B shows the acidity of the catalyst using NH3-TPD. Strong acid sites are usually directly related to olefin production [33-35]. The desorption curve shows two typical peaks corresponding to weak (120-260 °C) and strongly acidic areas (320-480 °c). The modified YUKS have a low acidity compared to the YUKS.

3.2. Catalytic activity

The concept of multifunctional catalysis was coined by Weiss [69]. Synthetic hybrid catalysts combining two separate types of active fields to convert carbon dioxide into light olefins in one step are usually prepared by a simple powder mixing method and are used for this reaction. NiCu / CeO_2 catalyst has been used to synthesize YUKS zeolite for methanol and MTO. Usually this process requires two independent reactors, where CH_3OH is achieved in the first reactor and light olefins can be produced from CH_3OH in the seolite in the second reactor. Alternatively, methanol can be synthesized in one reactor at a time and dehydrated to lower olefins [20,22,27,70-72]. This process is more thermodynamic and economical than the two-step process, as the thermodynamic limit for the methanol product is relaxed, which can change the equilibrium to a higher CO_2 conversion.

$$CO_2 + 3H_2 \rightarrow CH_3OH + H_2O \qquad (CO_2 \text{ to methanol}) \qquad (4)$$

$$nCH_3OH \rightarrow C_nH_{2n} + nH_2O \qquad (MTO) \qquad (5)$$

Here we will consider various factors such as reaction temperature, volumetric rate and H_2 / CO_2 ratio, which affect catalytic activity.

3.2.1 effect of reaction temperature

Temperature has been found to be a more important factor affecting a product with lower olefin selectivity. NiCu/CeO_2 - YUKS catalyst 350, 375, 400, 425 was also studied at several reaction temperatures of 450 °C at a pressure of 20 bar; volumetric velocity 12 Lgcat 1H 1; molar ratio H_2 / CO_2 3 and process time 10 hours. CO_2 conversion increases significantly with reaction temperature. The co selectivity, which depends on the reaction temperature, follows the same pattern for carbon dioxide conversion. When temperatures dropped from 450 to 375 ° C, CO selectivity decreased significantly from 85.87% to 64.27% (figure 5A), but C_2-C_4¼ selectivity increased from 60.14% (C_2H_4 = 18.6%, C_3H_6 = 26.4% and C_{48}) = 15.1% to 76.66% (C_2H_4 = 22.7%, C_3H_6 = 35.5% and C_4H_8 = 18.4%) (figure 5B) and selectivity in CH_4 decreased slightly from 5.2% to 2.1% (figure 5B).

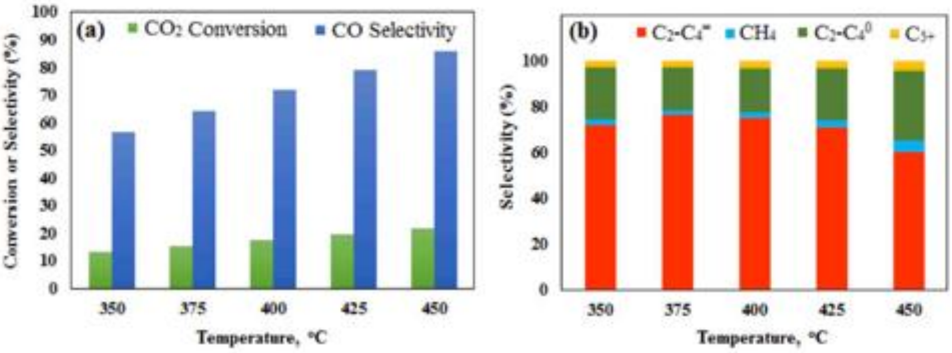


Figure 5. Effect of different temperatures on (a) CO_2 conversion and CO selectivity, (b) olefins (C_2–C_4¼), alkanes (C_2–C_{40}), CH_4 and C_5+ selectivity; P = 20 bar, SV = 12 Lgcat 1 h 1, H_2/CO_2 = 3:1 and reaction time ¼

The formation of light paraffins (C_2-C_{40}) and C_5+ fractions is also facilitated by an increase in temperature from 375 to 450 ° C. It is believed that the CO_2 conversion is a suitable temperature of 375 ° C for the synthesis of light olefins from carbon dioxide through NiCu / CeO_2 - YUKS catalyst, taking into account the selectivity of olefin and CH_4.

3.2.2. SV effect

The study of the volumetric rate of NiCu/CeO_2 - YUKS hybrid catalyst is shown in Figure 6. The temperature is 375 °C; the pressure is 20 bar; the molar ratio of H_2 / CO_2 is 3, while the reaction time is set to 10

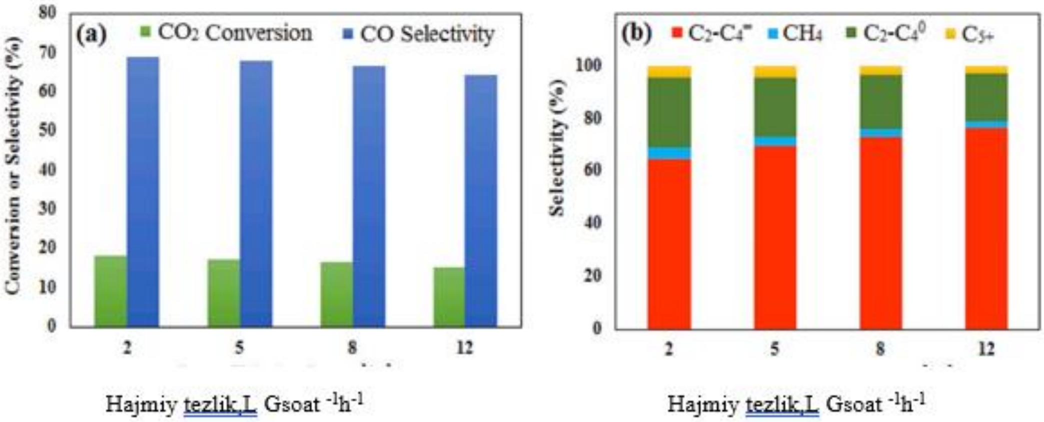


Figure 6. Effects of different cosmic velocity (A) on CO_2 conversion and CO selectivity, (b) olefins (C_2–C_4¼), alkanes (C_2–C_{40}), CH_4 and C_5+ selectivity; T = 375 °C, P = 20 bar, H_2/CO_2 = 3:1 and reaction time ¼

CO_2 conversion at SV 2 Lg-1 h 1 is 18.10% (Figure 6A) and olefin selectivity is 64.79% (C_2H_4 = 19.2%, C_3H_6 = 30% and C_4H_8 = 15.6%) (figure 6b). When the SV rises from 2 to 5 Lg-1 hrs 1, CO_2 conversion decreases to 17.33% and olefin selectivity improves to 69.36%. Subsequently, increasing SV from 8 to 12 Lg-1 h 1 would significantly reduce CO_2 conversion by 15.27%, and olefin selectivity (C_2–C_4¼) is slightly higher, at 76.66% (C_2H_4 ¼ 22.7%, C_3H_6 ¼ 35.5%, C_4H_8 ¼18, 4) %). As SV increases from 2 to 12 Lg-1 h 1, CO selectivity decreases slightly from 68.94% to 64.27% (Figure 6A). High SV leads to a reduction in the reaction time of CO_2 and H_2 molecules on the catalyst surface. Therefore, CO_2 conversion is low; however, light olefin production is slightly higher. This can be caused by the formation of CH_3OH, which reacts immediately with YUKS zeolite, resulting in more olefins. Thus, high olefin selectivity was achieved at high SV 12 Lg-1 h 1.

3.2.3 H_2 / CO_2 ratio effect

To study this effect, the temperature was 375 ° C; the reaction pressure was 20 bar; the flow rate was set at 12 l g 1 hour 1 and the reaction time at 10 hours. In the H_2 / CO_2 = 1 ratio, the CO_2 conversion is only 6.55% (Figure 7A), while the olefin selectivity is 79.85% (Figure 7B).

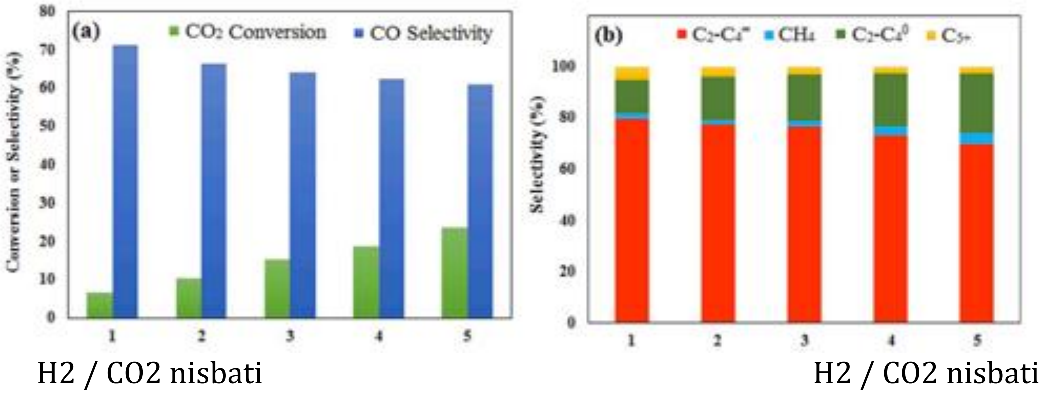


Figure 7 effect of different H_2/CO_2 ratios on (a) CO_2 conversion and CO selectivity, (b) olefins (C_2–C_4¼), alkanes (C_2–C_4 0), CH_4 and C_5+ selectivity; T = 375 °C, SV = 12 Lgcat 1 h 1, p = 20 bar and reaction time

When the H_2 / CO_2 ratio increases from 2 to 3, CO_2 conversion increases to 15.27% and olefin selectivity decreases to 76.66% (C_2H_4 = 22.7%, C_3H_6 = 35.5% and C_4H_8 = 18.4%). With an increase in H_2 / CO_2 ratio from 3 to 5, CO_2 conversion increases to 23.53%, but olefin selectivity decreases to 69.87%. In addition, selectivity for methane and paraffin increases to 4.40% and 23.12%, respectively. According to equations (4) and (6), when the H_2 / CO_2 ratio decreases from 3 to 1, the reaction (6) proceeds further, increasing the selectivity of CO formation:

$$CO_2 + H_2 \rightarrow CO + H_2O$$

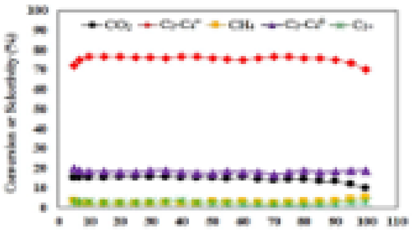
The selectivity of CH_4 also increases as the H_2 / CO_2 ratio increases from 3 to 5. When the resulting methanol reacts with an additional amount of H_2, it produces CH_4 and thus reduces the selectivity of olefins:

$$CH_3OH + H_2 \leftrightarrow CH_4 + H_2O$$

The above results showed that the H_2 / CO_2 ratio was 3. Thus, it can be said that the optimal reaction conditions are T = 375 c, SV = 12 L g 1 h 1, H_2 / CO_2 = 3 and P = 20 bar.

3.2.4 catalyst stability

Figure 8 shows the results of a long - term test of a NiCu/CeO_2-YUKS hybrid catalyst for 100 hours under optimal reaction conditions (T = 375 °c, P = 20 bar, H_2/CO_2 = 3, SV = 12 l G 1). h 1



Reaction time (h)

It is worth noting that the catalyst has good stability for 90 hours without a clear shutdown. The CO_2 conversion was maintained at a rate of about 15% for a 90-hour continuous reaction. In addition, the selectivity of methane and olefins remained stable at 2.6% and 76%, respectively. In addition, NiCu / CeO_2 - YUKS were found to be slowly switched off after 90 hours, for example carbon dioxide conversion decreased from 15% to 10%, olefin selectivity decreased from 76% to 70%. These data suggest that the catalysts developed are particularly stable and selective for the hydrogenation of CO_2 to downstream olefins.

4. Conclusion

To produce lower olefins directly from CO_2 hydrogenation, we synthesized a hybrid catalyst composed of NiCu / CeO_2 and YUKS zeolites to convert CO_2 directly into light olefins. Various factors affecting catalytic activity have been studied in detail, including gas space velocity (SV), reaction temperature, H_2 / CO_2 ratio, and stability test. Optimal reaction conditions were found to be T ¼ 375 ºC, P ¼ 20 bar, H_2/CO_2 ¼ 3, SV ¼ 12 L g 1 hour 1 at Optimal conditions C_2–C_4 ¼ olefins selectivity 76.6. Selectivity for% (C_2H_4 ¼ 22.7%, C_3H_6 ¼ 35.5% and c4h8 ¼ 18.4%) and kerosene (C_2–C_{40}) and methane was 18.3% and 2.1%, respectively, with carbon monoxide being less than 65% and carbon monoxide less than 65%. % CO_2. By increasing the reaction temperature, CO_2 conversion improves; however, mild olefin selectivity decreases. Increasing the gas field speed reduces CO_2 conversion and increases light olefin selectivity. When the H_2 / CO_2 ratio is increased from 1 to 3, the conversion of CO_2 increases by 15.27% and selectivity for olefins decreases to 76.66% (C_2H_4 ¼ 22.7%, C_3H_6 ¼ 35.5% and C_4H_8 ¼ 18.4%). As the H_2 / CO_2 ratio increases from 3 to 5, the conversion of CO_2 increases by 23.53%, but lower olefin selectivity decreases to 69.87%. In addition, the selectivity of methane and paraffin increases to 4.40% and 23.12%, respectively. In addition, our developed hybrid catalyst also provides useful catalytic stability, which can work continuously for 90 hours without loss of activity; a potential industrial catalyst for the use of CO_2 for light shows.

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