Hydrogenation of CO_2 to Methanol over Copper-Zinc Oxide-Based Catalyst

S. F. H. Tasfy, N. A. M. Zabidi, M. S. Shaharun

Abstract—Carbon dioxide is highly thermochemical stable molecules where it is very difficult to activate the molecule and achieve higher catalytic conversion into alcohols or other hydrocarbon compounds. In this paper, series of the bimetallic Cu/ZnO-based catalyst supported by SBA-15 were systematically prepared via impregnation technique with different Cu: Zn ratio for hydrogenation of CO_2 to methanol. The synthesized catalysts were characterized by transmission electron microscopy (TEM), temperature programmed desorption, reduction, oxidation and pulse chemisorption (TPDRO), and surface area determination was also performed. All catalysts were tested with respect to the hydrogenation of CO_2 to methanol in microactivity fixed-bed reactor at 250ºC, 2.25 MPa, and H_2/CO_2 ratio of 3. The results demonstrate that the catalytic structure, activity, and methanol selectivity was strongly affected by the ratio between Cu: Zn, Where higher catalytic activity of 14 % and methanol selectivity of 92 % was obtained over Cu/ZnO-SBA-15 catalyst with Cu:Zn ratio of 7:3 wt. %. Comparing with the single catalyst, the synergetic between Cu and Zn provides additional active sites to adsorb more H_2 and CO_2 and accelerate the CO_2 conversion, resulting in higher methanol production under mild reaction conditions.

Keywords—Hydrogenation of carbon dioxide, methanol synthesis, Cu/ZnO-based catalyst, mesoporous silica (SBA-15), and metal ratio.

I. INTRODUCTION

DUE to a serious threat that arises from the global warming, and climate change, alleviation of carbon dioxide, a major greenhouse gas, is a crucial issue [1]. The inert characteristic of CO_2, together with the lower reactivity in several reactions, intercepts the usage of CO_2 as a feedstock for many industrial processes. Therefore, converting a significant amount of CO_2 into valuable products such as methanol, which is considered as a substitute energy source, an intermediate for the storage and transportation of hydrogen as well as a starting material for the production of several chemicals, or other hydrocarbons through catalytic hydrogenation of CO_2 was known as promising CO_2 fixations process [2]. Production of methanol from alternative resources and its use as chemicals or fuel is an important accomplishment of the methanol economy where non-fossil fuel and inexpensive sources were used as feed in addition to mitigation of the greenhouse through recycling of CO_2 [3].

Methanol is currently produced from syngas using copper–zinc-based oxide catalysts at 5.0-10.0 MPa and 473-523 K.

$Cu/ZnO/Al_2O_3$ catalyst was commercially used in industrial methanol synthesis process due to its high catalytic activity, long life time, high poison durability and relatively low reaction temperature and pressure [4]. However, the low activity of industrial $Cu/ZnO/Al_2O_3$ catalysts especially at low reaction temperature initiates major barriers toward direct CO_2 hydrogenation. One of the most relevant requirements for solid catalysts to be particularly useful is an appropriate dispersion of the active species. Thus, a catalyst with good Cu and Zn dispersion is a key factor for high selectivity of methanol synthesis. Therefore, synthesis of a highly efficient catalyst is the key role for hydrogenation of low reactive CO_2 to methanol. Up to now, the exploitation of the catalyst is still not fully satisfactory for industrial application due to the lack of the necessary knowledge in catalyst design for the control of relevant catalytic properties and the mechanistic understanding of CO_2 hydrogenation reaction. The effect of reaction condition, type of oxide carriers, and usage of different active site on the performance of Cu-based catalyst for the hydrogenation of CO_2 have been investigated intensively in the past few decades [5]-[7].

Although, methanol synthesis is an old process that has been carried out over 90 years, debates still remain regarding the active sites. Therefore, in the present work, a series of bimetallic catalyst with different Cu: Zn ratio supported with mesoporous silica (SBA-15) with relatively high surface area was synthesized via impregnation technique and tested for methanol synthesis from CO_2 hydrogenation in microactivity fixed bed reactor under mild reaction conditions. The effect of the active sites ratio on the performance of the catalyst and methanol productivity was investigated and the relation between catalytic behavior and the physicochemical properties or catalytic performance was systematically addressed.

II. METHODOLOGY

A. Catalyst Synthesis

Due to the unique and exclusive characteristic of the ordered mesoporous Santa Barbara Amorphous (SBA-15) such as higher surface area, large pore volume, thicker wall, and hydrothermal stability. Synthesized SBA-15 has been used as a

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talented catalytic support for the hydrogenation of CO_2 to methanol.

In typical procedure, Cu/ZnO-SBA-15 (CZS) catalyst with different Cu:Zn ratio of 0.5, 1, and 2 were prepared using incipient wetness impregnation method with 15 wt. % of metal loading. Prior to metal loading, synthesis SBA-15 support was pre-treated at 773 K for 4 hours to remove impurities and to prepare the support surface to be active for adsorbing the precursor solution. Firstly, Desired amount of $Cu(NO_3)_2.3H_2O$ and $Zn(NO_3)_2.3H_2O$ was dissolved in deionized water to produce an aqueous solution (0.5 M) and then the solution was stirred for 1 h to form a homogenous mixture. Then certain amount of the prepared aqueous solution was added in drop wise manner into a beaker containing support under continuous stirring for 24 h. During the addition of the precursor solution, the pH of the mixture was kept at 7 using 25 M ammonia solution. Then, the slurry was filtered and washed with deionized water. The paste was air-dried at 120°C for 12 h followed by calcination in an Al_2O_3 boat at 350°C for 4 h.

In addition, monometallic catalyst of Cu and Zn supported with SBA-15 was synthesized under the similar condition to be use as benchmark.

B. Catalyst Characterization

The physicochemical properties such as BET surface area and morphology, of the obtained catalysts were observed using N_2-physical adsorption and Transmission Electron Microscopy (TEM), respectively. Furthermore, X-Ray Photoelectron Spectroscopy (XPS, Thermo-Fischer K-Alpha) were preformed to determine chemical state, surface composition, oxidation state, and the type of interaction between active sites and the support. Additionally, the catalytic reducibility and Cu surface area were determined using Thermo TPD/R/O 1100 equipped with a thermal conductivity detector and a mass spectrometer. N_2O-chemsorption was used to determine the Cu surface which was known to be the methanol synthesis facilitating agent.

C. Catalyst Screening

Methanol catalyst screening was performed in a micro-activity fixed bed reactor (Microactivity Reference PID-Eng). Prior to the measurement of the catalytic performance, 0.2 g fresh catalyst was reduced in 5 vol% of H_2 in Argon gas at 20 mL min^{-1} and 523 K for 4 hours. The reaction was performed at 2.25 MPa, 483 K, and the gas feed ratio of CO_2 to H_2 was 1:3 with total flow rate of 35 mL/min for 10 hours. The reactor effluents were analyzed by gas chromatograph (Agilent 6890) equipped with TCD detector for H_2 and CO_2 analysis, and FID detector for methanol and another hydrocarbons analysis. CO_2 conversion, methanol selectivity, turn over frequency (TOF), and space time yield (STY) were calculated using (1)-(4), respectively.

$$CO_2 \ conversion \ (\%) = \frac{Mole \ of \ CO_{2,in} - Mole \ of \ CO_{2,out}}{Mole \ of \ CO_{2,in}} \times 100 \quad (1)$$

$$CH_3OH \ selectivity \ (\%) = \frac{Mole \ of \ methanol \ produced}{Total \ mole \ of \ products} \times 100 \quad (2)$$

$$TOF_{MeOH} = = \frac{A.N_A}{3600. \ S_{Cu}. \ N_{Cu}} \quad (3)$$

$$STY = \frac{Quantity \ of \ desired \ product}{Weight \ of \ catalyst \times time} \quad (4)$$

where, A represents the reaction rate of methanol (mol h^{-1}g^{-1}), N_A refers to Avogadro's number (6.02×10^{23}), S_{Cu} designates Cu surface are in m^{2}g^{-1}, N_{Cu} denoted number of Cu atoms in a monolayer (1.49×10^{19} atoms m^{-2}).

III. RESULTS AND DISCUSSION

A. Textural and Morphological Properties

Due to the controversy regarding the role of ZnO in the catalytic properties and performance on the methanol synthesis process, a series of SBA-15 supported bimetallic Cu/ZnO-catalyst with different Cu:Zn ratio was synthesized. Moreover, monometallic based catalyst of Cu/SBA-15 (CS) and Zn/SBA-15 (ZS) was prepared under the same condition to provide a proper comparison.

The textural properties such as the catalyst surface area (S_{BET}), pore volume (V_P) and pore diameter (D_{BJH}) of synthesized catalyst with different Cu:Zn ratio is displays at Table I. The results obtained from N_2 adsorption/desorption technique shows that increasing Zn content resulted in decrease in total BET surface area and pore volume meanwhile it results in increasing the pore diameter. This result is similar to the data reported by Fujitani et al. [8] where they concluded that addition of ZnO up to 40% resulted in significant increase of the catalyst BET surface area due to increasing the Cu surface area. Furthermore, variation of the metal particles size as the results of verifying the ratio between Cu and Zn was observed via the TEM analysis. Fig. 2 shows the morphological analysis of the supported catalysts with different Cu:Zn ratio. Increasing the amount of ZnO resulted in formation of larger particles with bimodal particle distribution (Fig. 1). The obtained results from the TEM analysis support the textural analysis where formation of the larger particles due to increasing of ZnO content could result in block some of the support pore and result in decreasing the total catalyst BET surface area and pore volume. SBA-15 supported catalyst with Cu:Zn ratio of 7:3 exhibit highly dispersed copper with largest BET surface area compared to the other catalyst with different Cu:Zn ratio. Since this characteristic pivotal an essential role in the performance of catalysts, the right ratio of Cu to Zn was suggested to promote the hydrogenation of CO_2 to methanol.

TABLE I
TEXTURAL PROPERTIES OF SBA-15 SUPPORTED CU/ZNO-BASED CATALYST WITH DIFFERENT CU:ZN RATIO

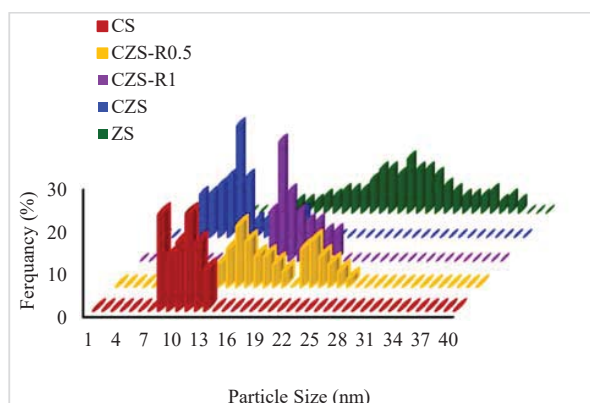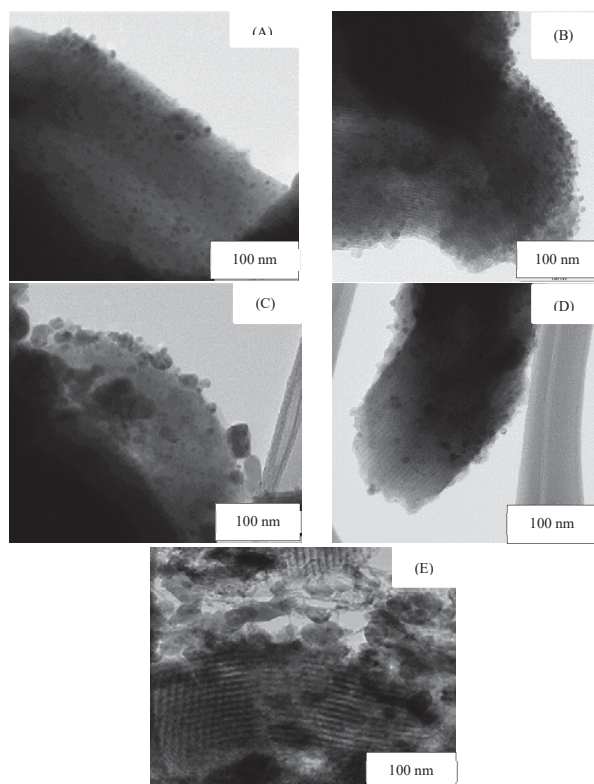| Catalyst	Cu:Zn ratio	S_{BET} (m^2/g)	V_P (cm^3/g)	D_{BJH} (nm)
SBA-15	-	510	0.87	6.8
CS	-	445	0.60	5.3
CZS	7:3	458	0.62	6.9
CZS-R1	1:1	387	0.59	7.1
CZS-R0.5	3:7	319	0.58	7.3
ZS	-	315	0.61	7.8

Fig. 1 Particle size distribution

Fig. 2 TEM images for the SBA-15 supported Cu/ZnO catalyst with different Cu:Zn ratio (a) CS, (b) CZS-R0.5, (c) CZS-R1, (d) CZS, and (e) ZS

B. Chemical State

The chemical state of the synthesized SBA-15 supported catalyst with different Cu:Zn ratio was evaluated by XPS and the results were illustrated in Figs. 3 and 4. The Cu 2p XPS spectra in Fig. 3 shows that position of Cu 2p3/2 and Cu 2p1/2 peaks revealing that chemical state of the surface substance influenced by varying the ratio between the two active sites. All the calcined catalyst confirmed the presence of Cu^{2+} due to the existing of characteristic satellite peak at 938 eV. In spite of the presence of Cu^{2+} as characteristic peak in all the synthesized sample with different Cu:Zn ratio, the intensity of the peak

decrease with decreasing the amount of Cu in the sample which attribute to the existence of Cu^{1+} and Cu^{2+}. In addition, the intensity of the Cu 2p3/2 decrease for the catalyst with high Zn content which could be explained by coverage of Cu clusters by a thin Zn layer. The presence of Cu in two chemical state (Cu^{1+} and Cu^{2+}) in CZS catalyst which has the lowest amount of Zn confirmed by the disappearance of the second satellite peak around 947 eV.
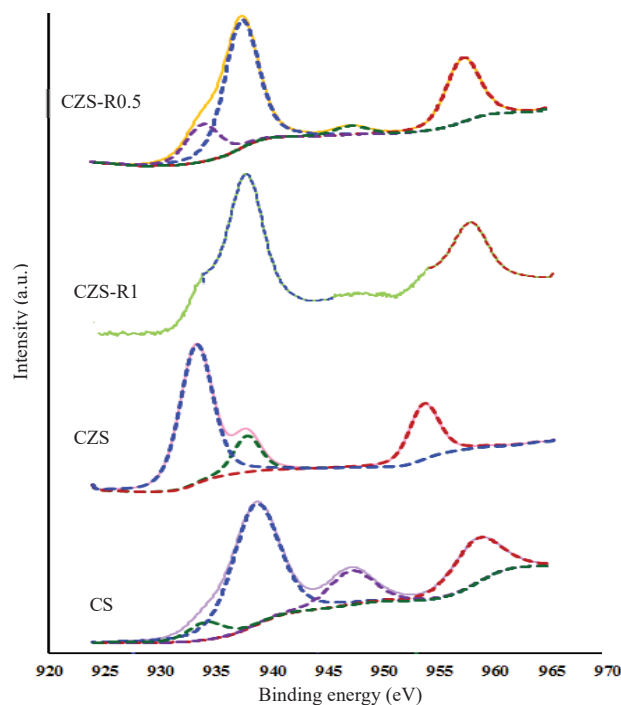

Fig. 3 XPS spectra of Cu 2p region for SBA-15 supported Cu/ZnO-based catalyst with different Cu:Zn ratio

Fig. 4 represent the XPS patterns of Zn 2p where two main peaks of Zn 2p3/2 and Zn 2p1/2 obtained at binding energy of 1022 and 1046 eV, respectively which were the characteristic peak of Zn^{2+}. In addition to the main Zn 2p peaks two shake-up satellite peaks was obtained for all the synthesized catalysts except CZS which exhibit the presence of only one satellite peak at around 1027.78 eV allowing the possibility for Zn to be existed in two different chemical states.

C. Catalytic Reducibility

In order to evaluate the reduction behavior of the catalysts, TPR measurements were carried out for the synthesized catalyst with different Cu:Zn ratio. TPR profile in Fig. 5 shows that all samples exhibit one peak for the hydrogen consumption which is attributed to one step reduction of $Cu^{2+} \rightarrow Cu^{0}$, while the position of this peak was strongly affected by the ratio of the active sites. In addition to the dominated reduction around 295ºC, CZS catalyst exhibit small reduction beak at higher temperature around 385ºC assigned to reduction of Cu^{1+} to Cu^{0}. Thus, the presences of the second reduction peak support the XPS results which indicate the presence of Cu in the CZS

sample at two chemical states. Monometallic Cu-based catalyst exhibits the reduction of Cu species at relatively low temperature of 198°C which is ascribed as the reduction of the well-dispersed copper species. These results indicate that addition of Zn alter the metal interaction and result in impeding the catalytic reducibility. Slight shift on the position of H_2 consumption peak to higher temperature was obtained due to incorporation of ZnO. This is in accordance with physical properties of small particle size and large amount of bulk Cu, strong bimetallic interaction and also metal support interaction as illustrated in Figs. 2 and 3.
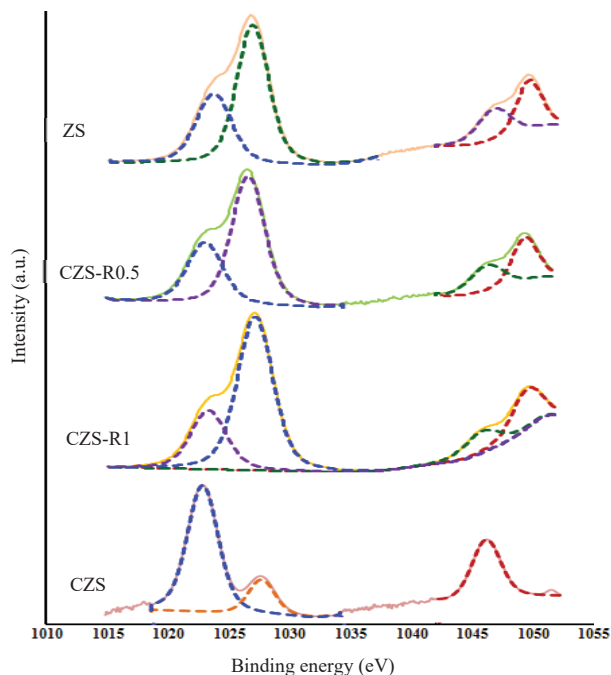
support the hypothesis of the promotional effect of Zn in synthesis of methanol catalyzed by Cu-based catalyst and the optimal Cu:Zn ratio is equivalent with the reported values [9], [10]. Different observation was presented by Tanaka et al. [11] based on their study about the effect of a novel binary Cu/ZnO catalyst in the presence of ethylene glycol as a solvent and their results show that higher methanol production was observed on 1:1 Cu/ZnO ratio and the atomic copper incorporated with oxide copper crystallites was assigned to be the main active site.

Fig. 4 XPS spectra of Zn 2p region for SBA-15 supported Cu/ZnO-based catalyst with different Cu:Zn ratio

D. Catalytic Performance

Hydrogenation of CO_2 to methanol over SBA-15 supported Cu/ZnO-based catalyst with different Cu:Zn ratio was evaluated on microactivity fixed bed reactor at 250°C, 2.25MPa, H_2/CO_2 ratio of 3. As shown in Fig. 6 the ratio between Cu and Zn significantly affect the catalytic activity and resulted in alteration of the CO_2 conversion. Increasing the Cu content within the catalyst structure enhance the CO_2 conversion until it reaches the maximum at 70% Cu while addition of Zn for more than 30% inhibit the catalytic activity. Similar trend regarding the effect of Cu and Zn content on the methanol formation was observed where the highest methanol selectivity of 92.1 was achieved over CZS catalyst with 7:3 Cu:Zn ratio (Fig. 7). Compered to Cu-free catalyst, Zn-free catalyst exhibits lower CO_2 conversion and methanol selectivity and higher methane production while bimetallic catalyst preformed extremely higher performance. Thus, it's clearly indicated that Zn plays important role in facilitating the catalytic activity as well as the methanol selectivity. This result
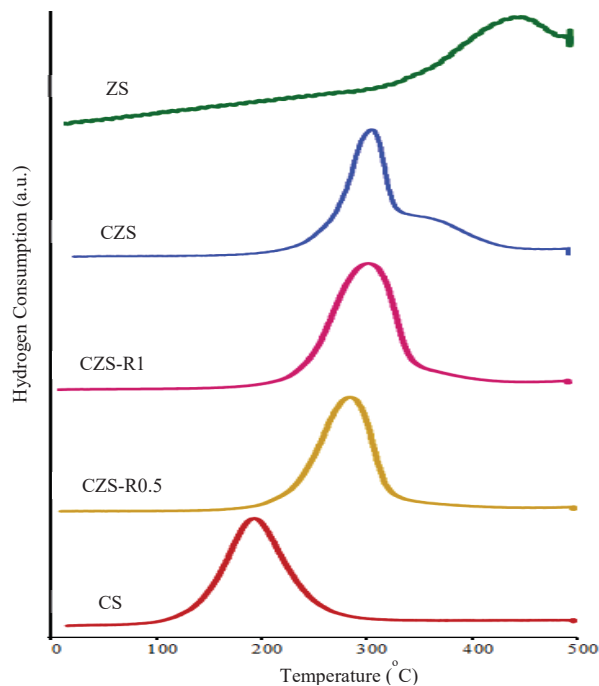

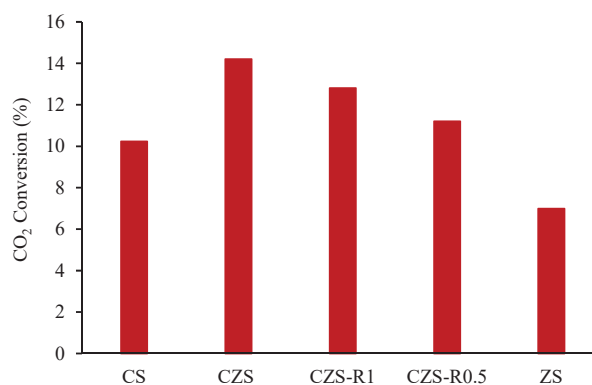Fig. 5 TPR profile for the SBA-15 supported Cu/ZnO-based catalyst with different Cu:Zn ratio

Fig. 6 Activity of catalyst with different Cu:Zn ratio, reaction conditions: 250°C, 2.25 MPa, 3 H_2/CO_2 ratio, and 0.2 g_{cat}

Table II shows the methanol productivity which is expressed by the space time yield as a function of the ratio between Cu and Zn. Furthermore, the relation between the Cu surface area which have been determined using the TPD-NO_2 chemisorption with methanol selectivity and CO_2 conversion over series of

catalysts with different Cu: Zn ratio is presented in Table II. Monometallic catalyst of ZS exhibit very low methanol productivity while Zn-free CS catalyst shows slight higher methanol productivity. The methanol productivity significantly increased with increasing the Cu content until it reaches the maximum of 51.4 and 39.3 g-MeOH/h.g_{cat} for 70% Cu in two-phase and three-phase system, respectively.
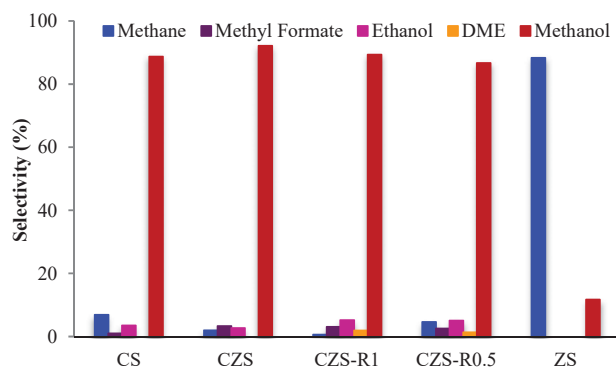

Fig. 7 Products selectivity of catalyst with different Cu:Zn ratio, reaction conditions: two-phase, 250°C, 2.25 MPa, 3 H_2/CO_2 ratio, and 0.2 g_{cat}.

TABLE II
PERFORMANCE OF SBA-15 SUPPORTED CU-ZNO-BASED CATALYST WITH DIFFERENT CU:ZN RATIO AT 250°C, 2.25 MPA, AND H_2/CO_2 RATIO OF 3

Catalyst	S_{Cu} (m²/g)	X_{CO2} (%)	S_{MeOH} (%)	STY (g/h.g_{cat}) CO	STY (g/h.g_{cat}) MeOH	TOF (s^{-1})
CS	2.6	10.2	88.7	0.96	17.72	2.39
CZS	4.2	14.2	92.1	0.48	51.37	4.29
CZS-1	3.7	12.8	89.3	1.64	20.57	1.95
CZS-0.5	3.4	11.2	86.6	1.85	30.63	3.16
ZS	-	6.9	11.7	2.68	2.3	-

Therefore, from the characterization results we can conclude that large BET and Cu surface area, well-dispersed active sites, and easily reduced Cu species occurred at total metal loading of 15 wt% of Cu+Zn over ordered mesoporous silica SBA-15 with Cu:Zn ratio of 70:30 which exhibits the highest catalytic activity as well as methanol selectivity. This could be clarified by synergetic effect between Cu and Zn which is responsible for H_2 spillover between both sites. In spite of ZnO being used for more than decades, the role of ZnO is still debatable where few research proved that with same amount of Cu active sites addition of ZnO exhibited higher activity compared with Cu-based catalyst [12], [13]. Therefore, it's proven that ZnO acts as a chemical and textural promoter in the Cu-based catalyst system.

Additionally, the relation between catalytic performance and Cu surface area was clearly preformed through this study and presented in Table II. Where, a linear relationship between Cu surface area and methanol production was observed where catalyst with larger Cu surface area reveal higher methanol yield as well as CO_2 conversion and TOF. Compared to TOF values in the available literature, TOF obtained from this work is higher than 2.2×10^{-2} s^{-1} [14], or in some cases it is relatively comparable. Linear correlation between Cu surface area and catalytic performance has been observed by Natesakhawat and his co-workers [14]. CO space time yield was calculated in order to determine the formation of the water-gas-shifting reaction. The results show that increasing of Zn content facilitate the production of CO as well as water content which is relevant to side reaction of WGS reaction. Thus, lower methanol formation was obtained via using catalyst with higher ZnO content.

IV. CONCLUSION

Specific activity of the supported catalyst with different ratios of active sites Cu and Zn were investigated for the hydrogenation of CO_2 to methanol at mild reaction conditions of 250°C, 2.25 MPa and H_2/CO_2 ratio of 3 in microactivity fixed bed reactor. The presented data shows that the activity, methanol selectivity, turn over frequency as well as the space time yield increased with increasing the Cu:Zn ratio until it reach the maximum values at Cu:Zn ratio of 2 while further addition of ZnO inhabit the catalytic performance. These could be attributed to the physicochemical properties of the synthesized catalyst where SBA-15 supported bimetallic catalyst with 7:3 % Cu:Zn ratio shows the largest BET and Cu surface area, smallest particle size with well dispersion of the active site over the support, and lower catalytic reducibility.

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