ETHANOL CO_2 REFORMING OVER Ce AND La PROMOTED Cu/Al_2O_3 CATALYSTS FOR SYNGAS PRODUCTION

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MASTER OF SCIENCE

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pembaharuan semula karbon dioksida etanol (ECR) adalah merupakan satu cara yang mesra alam dan baru untuk penghasilan gas sintetik kerana bukan hanya menggunakan bioethanol tetapi menawarkan manfaat tambahan dengan menggunakan gas rumah hijau (CO_2) yang tidak diingini. Walaubagaimanapun, penghasilan karbon semasa proses ECR menyebabkan penyahaktifan pemangkin. Oleh itu, matlamat tesis ini adalah untuk mengkaji kesan CeO_2 dan La_2O_3 sebagai penggalak dan kadar kuantiti penggalak terhadap sifat-sifat fizikokimia bagi pemangkin 10%Cu/Al_2O_3. Pemangkin 10%Cu/Al_2O_3 dengan penggalak 3%Ce- dan 3%La-pemangkin disediakan melalui kaedah pengisitepuan basah baru berturutan manakala, pemangkin tanpa penggalak disediakan melalui teknik pengisitepuan basah baru. Pemangkin dicirikan menggunakan penjerapan fizik N_2 (BET), penurunan suhu berprogram (TPR), pengoksidaan suhu berprogram (TPO), analisis pembelauan sinar-X (XRD), spektroskopi elektron sinar (XPS), mikroskop elektron pengimbasan (SEM), mikroskop elektron transmisi resolusi tinggi (HRTEM) dan spektroskopi Raman. Proses tersebut dikaji di dalam reaktor keluli tahan karat dengan perbezaan suhu dari 948 hingga 1023 K dan nisbah CO_2:C_2H_5OH ratios of 2.5:1 to 1:2.5 di bawah tekanan atmosfera. Luas permukaan BET pemangkin adalah diantara 93.4 to 98.5 m^2 g^{-1} manakala purata saiz kristal CuO berkurangan dari 32.4 hingga 27.4 nm dengan penambahan La_2O_3 dan CeO_2 disebabkan oleh kesan pencairan. Penambahan penggalak menguatkan interaksi diantara metal dan sokongan, dibuktikan melalui analisis H_2-TPR. Peningkatan pengambilan H_2 dengan penggalak semasa H_2-TPR menunjukkan peningkatan tahap pengurangan $CuO \rightarrow Cu^0$. Kedua-dua penukaran reaktan meningkat dengan peningkatan suhu dari 948-1023 K untuk semua sampel disebabkan oleh sifat endoterma ECR. Peningkatan tekanan separa CO_2 dari 20 to 50 kPa menambah baik penukaran reaktan untuk semua pemangkin manakala tekanan separa C_2H_5OH yang optimum diperhatikan pada 40 kPa. Kehadiran endotermik serentak dehidrogenasi etanol di dalam ECR meningkatkan nisbah H_2/CO dari 1.46-1.91 sesuai dengan sintesis Fischer-Tropsch. Pengukuran XRD oleh pemangkin yang telah digunakan membuktikan fasa aktif Cu^0 dikekalkan semasa ECR dan pemangkin menentang pengoksidaan semula di dalam bahan mentah yang mengandungi CO_2. Walaupun pembentukan karbon dikesan di atas pemangkin yg telah digunakan melalui ukuran XRD, TPO dan HRTEM, penurunan yang besar dalam jumlah pembentukan karbon dari 40.04% hingga 27.55% telah dicapai dengan penambahan penggalak dengan turutan La-penggalak < Ce- penggalak < pemangkin tanpa penggalak Cu/Al_2O_3. 3%La-Cu/Al_2O_3 adalah pemangkin terbaik untuk kajian dengan penukaran C_2H_5OH (94.64%), penukaran CO_2 (73.21%), hasil H_2 (68.32%) dan hasil CO (32.06%) disebabkan oleh kebolehan kitaran redox, ciri asas dan mempunyai pengambilan H_2 yang tertinggi dan saiz kristal yang paling kecil.

ABSTRACT

Ethanol CO_2 reforming (ECR) is an eco-friendly and novel way for syngas production as it not only consumes bio-ethanol but also offers an additional benefit by utilizing the unwanted greenhouse gas (CO_2). Nevertheless, the carbonaceous deposition during ECR process leads to deactivation of the catalyst. Hence, the aim of this thesis was to investigate the effect of CeO_2 and La_2O_3 as promoters and promoter loading on the physicochemical properties of 10%Cu/Al_2O_3. 3%Ce- and 3%La-promoted 10%Cu/Al_2O_3 catalysts were synthesized via sequential incipient wetness impregnation approach meanwhile, unpromoted catalysts were prepared by incipient wetness impregnation technique. The catalysts were characterized using N_2 physisorption (BET), temperature-programmed reduction (TPR), temperature programmed oxidation (TPO), X-ray diffraction (XRD) analysis, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM) and Raman spectroscopy. The processes were evaluated in stainless steel fixed-bed reactor at varying temperature from 948 to 1023 K and CO_2:C_2H_5OH ratios of 2.5:1 to 1:2.5 under atmospheric pressure. The BET surface area of these catalysts was about 93.4 to 98.5 m^2 g^{-1} whereas the calculated average CuO crystallite size declined from 32.4 to 27.4 nm with La_2O_3 and CeO_2 additions because of the diluting effect. Promoter addition enhanced metal-support interaction as evidenced in H_2-TPR analyses. The increasing H_2 uptake with promoters during H_2-TPR was indicative of enhancing degree for CuO $\rightarrow$ Cu^0 reduction. Both reactant conversions increased substantially with rising temperature from 948-1023 K for all samples due to ECR endothermic nature. The increase in CO_2 partial pressure from 20 to 50 kPa improved reactant conversions for all catalysts whilst the optimal C_2H_5OH partial pressure was observed at 40 kPa. The existence of concurrent endothermic ethanol dehydrogenation in ECR increased H_2/CO ratio from 1.46 to 1.91 suitable for Fischer-Tropsch synthesis. XRD measurements of spent catalysts proved that Cu^0 active phase was maintained during ECR and catalysts resisted to re-oxidation in CO_2-containing feedstock. Although carbonaceous formation was detected on spent catalysts by XRD, TPO and HRTEM measurements, the significant decline in total carbon deposition from 40.04% to 27.55% was achieved by promoter addition in this order; La-doped < Ce-doped < unpromoted Cu/Al_2O_3. 3%La-Cu/Al_2O_3 is the best catalyst for this research with C_2H_5OH conversion (94.64%), CO_2 conversion (73.21%), H_2 yield (68.32%) and CO yield (32.06%) due to redox cycle ability, basic properties, possessed highest H_2 consumption and smallest crystallite size.

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LIST OF SYMBOLS

A	Pre-exponential factor
B	The line broadening at half the maximum intensity (FWHM)
B_{iw}	Biot number
C	Constant characteristic of adsorbate
C_{Ab}	Bulk gas-phase concentration of component A
C_{As}	Concentration of ethanol on catalyst surface
C_D	Percentage of amorphous carbon
C_{pg}	Specific heat capacity of feed gas mixture at constant pressure
C_γ	Carbides
C_v	Vermicular carbon (whisker-like)
D_{eff}	Effective diffusivity
D_g	Diffusivity
D_p	Average pore diameter
d_{CuO}	Average crystallite size of CuO phase
d_p	Diameter of catalyst particle
d_t	Diameter of reactor tube
E_a	Activation energy
E_l	The excitation of laser energy
F	Molar flow rates
h	Heat transfer coefficient between gas mixture and catalyst
h_w	Heat transfer coefficient of reactor tube wall
j_D	Colbourn's mass transfer factor
j_H	J-factor for heat transfer
k_c	Mass transfer coefficient

L_a	Crystallite size
M_{ad}	Molecular weight of adsorbate
n_m	Number of molecules adsorbed
n	Reaction order
p	Partial pressure of gaseous species
P_r	Prandtl number
P_s	Saturation pressure of adsorbed gas
R	Ideal gas constant
R_t	Radius of reactor tube
R_p	Particle radius of catalyst
r	Production of formation rates
$(-r_{exp})$	Rate of reaction
S_A	Total surface area of sample
S_C	Schmidt number
T_b	Reactant gas bulk temperature
T_s	Reactant gas bulk temperature
T_w	Tube wall temperature
t_{ads}	Thickness of adsorbed layer
U	Superficial gas velocity
V_a	Volume of gas adsorbed
V_p	Total pore volume
W_{cat}	Catalyst weight
X_i	Reactant conversion
Y_i	Yield of product
λ	Wavelength

λ_m	Thermal of catalyst material
λ_p	Thermal conductivity of catalyst pellet
θ	Bragg angle
ε	Void fraction in catalyst bed
τ	Tortuosity
σ_c	Construction factor
ω_p	Porosity of catalyst pellet
μ_g	Viscosity of gas mixture
ρ_b	Bulk density of catalyst bed
ρ_c	Density of catalyst pellet
ρ_g	Density of gas mixture
ΔH	Heat of reaction
ΔG	Gibbs free energy
Al_2O_3	Aluminium oxide
Ce	Ceria
CeO_2	Cerium oxide
Cu	Copper
CuO	Copper oxide
CO_2	Carbon dioxide
Ni	Nickel
Pt	Platinum
Ru	Ruthenium
Rh	Rhodium
Ir	Iridium
Sm	Samarium
Pm	Promethium

LIST OF ABBREVIATIONS

ATR	Autothermal reforming
BET	Brunauer-Emmett-Teller
CNF	Carbon nanofilament
DME	Dimethyl ether
DOE	Department of Energy
ECR	Ethanol CO_2 reforming
EDX	Energy dispersive X-ray
ESR	Ethanol steam reforming
FTS	Fischer-Tropsch synthesis
GC	Gas chromatography
GHSV	Gas hourly space velocity
GTL	Gas to liquid
HRTEM	High resolution transmission electron microscopy
H_2-TPR	H_2-temperature programmed reduction
I.D	Inner diameter
JCPDS	Joint committee on powder diffraction standards
MDR	Methane dry reforming
MT	Metric ton
MTBE	Methyl tert-butyl ether
O.D	Outer diameter
POX	Partial oxidation
SEM	Scanning electron microscopy
SR	Steam reforming
TCD	Thermal conductivity detector
TGA	Thermogravimetric analysis
TPO	Temperature-programmed oxidation
TPR	Temperature-programmed reduction
WGS	Water-gas shift
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

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