

SYNGAS PRODUCTION FROM ETHANOL DRY
REFORMING OVER La AND Ce PROMOTED
Co/Al₂O₃ CATALYSTS

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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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 kering etanol telah muncul sebagai laluan yang berpontesi dalam menukarkan etanol diperbaharui dan gas rumah hijau yang tidak diingini (CO_2) kepada syngas yang diiktiraf oleh industri. Syngas boleh digunakan sebagai bahan mentah bagi pengeluaran metanol hiliran dan sintesis Fischer-Tropsch. Walau bagaimanapun, pemendapan karbon semasa proses EDR menyebabkan penyahaktifan pemangkin. Oleh itu, pemangkin berasaskan Co telah disediakan dengan penggalak La dan Ce menggunakan kaedah pengisitepuan basah dan menyelidik sifat fizikokimia $10\% \text{Co}/\text{Al}_2\text{O}_3$ serta menilai kesan parameter operasi terhadap aktiviti pemangkin untuk tindak balas pembaharuan kering etanol dalam reaktor kuarza turus tetal. Di samping itu, kesan kandungan ceria yang berlainan (dari 2% hingga 5%) telah dinilai dalam tindak balas pembaharuan kering etanol pada 973 K dan juga pada keadaan stoikiometri. Hasilnya menunjukkan bahawa 3% pemangkin Ce-penggalak menunjukkan aktiviti dan rintangan tertinggi terhadap pemendapan karbon. Pemangkin dengan penggalak 3%Ce dibandingkan dengan pemangkin dengan penggalak 3%La dan pemangkin tanpa penggalak pada nisbah $\text{C}_2\text{H}_5\text{OH}:\text{CO}_2$ berbeza iaitu 2.5:1-1:2.5 dan pada suhu tindak balas 923 hingga 973 K. 3%La terhadap $10\% \text{Co}/\text{Al}_2\text{O}_3$ telah meningkatkan penyebaran logam yang tinggi (kira-kira 16.6%) dan kadar pengurangan (98.3%). Selain itu, penukaran $\text{C}_2\text{H}_5\text{OH}$ dan CO_2 meningkat sehingga 150.6% dan 55.5%, masing-masing terhadap peningkatan suhu tindak balas dari 923 hingga 973 K disebabkan oleh sifat endotermik tindak balas pembaharuan kering etanol. Di samping itu, penukaran kedua-dua reaktan telah meningkat dengan peningkatan tekanan separa CO_2 daripada 20 hingga 50 kPa untuk semua pemangkin, sementara, penukaran reaktan menurun dengan peningkatan tekanan separa $\text{C}_2\text{H}_5\text{OH}$. Dalam pembaharuan kering etanol, nisbah H_2/CO selalu lebih tinggi daripada satu disebabkan adanya tindak balas sampingan (penyahhidrogenan etanol). Tanpa mengira keadaan tindak balas, pemangkin dengan penggalak La menjadi pemangkin optimum dari segi penukaran $\text{C}_2\text{H}_5\text{OH}$ dan CO_2 . Pertukaran reaktan meningkat dalam susunan; $10\% \text{Co}/\text{Al}_2\text{O}_3 < 3\% \text{Ce}-10\% \text{Co}/\text{Al}_2\text{O}_3 < 3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ pemangkin pada semua keadaan operasi. Pemangkin $10\% \text{Co}/\text{Al}_2\text{O}_3$ dan $3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ telah diuji untuk ujian lanjutan masa dalam pembaharuan kering etanol dan menunjukkan bahawa pemangkin $3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ mempamerkan aktiviti yang tinggi berbanding pemangkin tanpa penggalak pada keadaan stoikiometri untuk 72 h dan 973 K. Tambahan pula, pemangkin dengan penggalak La telah diperbaharui dengan tiga kitaran dan diplot dengan masa pada komposisi suapan stoikiometri selama 90 h dan $T = 973 \text{ K}$. Hasilnya mendapati bahawa 3%La mempamerkan prestasi pemangkin tertinggi dari segi aktiviti dan pemendapan karbon berbanding pemangkin yang tanpa penggalak. Sifat heterogen bagi karbon mendap (karbon nanofilament dan grafit) pada permukaan pemangkin yang telah digunakan adalah jelas. Selain itu, penggalak 3%La mengurangkan pembentukan karbon dari 51.49% kepada 30.06%. Di samping itu, pengiraan undang-undang kuasa mendapati bahawa tenaga pengaktif untuk pemangkin dengan penggalak Ce dan penggalak La (kira-kira 98 kJ mol^{-1} untuk penggalak Ce dan 93 kJ mol^{-1} untuk penggalak La) adalah lebih kecil berbanding $10\% \text{Co}/\text{Al}_2\text{O}_3$ tanpa penggalak dengan lebih kurang 108 kJ mol^{-1}). Ekspresi kadar Langmuir-Hinshelwood juga mencadangkan bahawa kedua-dua reaktan ($\text{C}_2\text{H}_5\text{OH}$ dan CO_2) dikaitkan secara berserat pada satu tapak pemangkin dengan tenaga pengaktifan sebanyak 106 kJ mol^{-1} . Kajian ini menunjukkan bahawa syngas yang dihasilkan daripada pemangkin berasaskan Co dengan nisbah H_2/CO yang wajar boleh digunakan secara langsung dalam sintesis Fischer-Tropsch tanpa keperluan penyesuaian komposisi bahan.

ABSTRACT

Ethanol dry reforming has been emerged as a promising route for converting the renewable ethanol and undesirable greenhouse gas (CO_2) to industrially recognized syngas. It can also be used as feedstock for downstream methanol production and Fischer-Tropsch synthesis. However, the carbonaceous deposition during ethanol dry reforming process leads to deactivation of the catalyst. Therefore, the Co-based catalysts were prepared with La and Ce promoters using a wet impregnation method and investigated the physicochemical attributes of $10\% \text{Co}/\text{Al}_2\text{O}_3$ as well as evaluated the effect of operating parameters on the catalytic activity of ethanol dry reforming reaction in a quartz fixed-bed reactor. In addition, the effect of different ceria loading (from 2% to 5%) was evaluated for ethanol dry reforming reaction at 973 K and stoichiometric conditions. The results revealed that the 3% Ce-promoted catalyst showed the highest activity and resistance from coke deposition. The 3%Ce-promoted catalyst was compared with 3%La-promoted and unpromoted catalyst at different $\text{C}_2\text{H}_5\text{OH}:\text{CO}_2$ ratios of 2.5:1-1:2.5 and reaction temperature of 923 to 973 K. The 3%La over bare $10\% \text{Co}/\text{Al}_2\text{O}_3$ significantly improved the metal dispersion (about 16.6%) and degree of reduction (98.3%). Besides, $\text{C}_2\text{H}_5\text{OH}$ and CO_2 conversions increased up to 150.6% and 55.5%, respectively with growing reaction temperature from 923 to 973 K due to the endothermic character of ethanol dry reforming reaction. In addition, both reactant conversions increased with rising CO_2 partial pressure from 20 to 50 kPa for all catalysts while, the decreasing reactant conversions with increasing $\text{C}_2\text{H}_5\text{OH}$ partial pressure. In ethanol dry reforming runs, H_2/CO ratio was always higher than unity due to the presence of side reaction (ethanol dehydrogenation). Irrespective of reaction conditions, La-promoted catalyst seemed to be the best catalyst in terms of both $\text{C}_2\text{H}_5\text{OH}$ and CO_2 conversions. Reactant conversions of catalysts increased in the order; $10\% \text{Co}/\text{Al}_2\text{O}_3 < 3\% \text{Ce}-10\% \text{Co}/\text{Al}_2\text{O}_3 < 3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ catalysts for all operating conditions. The $10\% \text{Co}/\text{Al}_2\text{O}_3$ and $3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ catalysts were examined for longevity tests in ethanol dry reforming and showed that the $3\% \text{La}-10\% \text{Co}/\text{Al}_2\text{O}_3$ catalyst exhibited the high catalytic activity than that of unpromoted catalyst at stoichiometric condition for 72 h and 973 K. Furthermore, La-promoted catalyst was regenerated with three cycles and plotted with time-on-stream at stoichiometric feed composition for 90 h and $T = 973$ K. The results found that the 3%La exhibited the highest catalytic performance in terms of activity and carbon deposition compared to the counterpart unpromoted catalyst. The heterogeneous nature of deposited carbons (carbon nanofilament and graphite) on spent catalyst surface was evident spent catalyst characterizations. Additionally, the 3% La promoter reduced the carbon formation from 51.49% to 30.06%. Furthermore, from the power law expression found that the activation energy for Ce- and La-promoted catalysts (about 98 kJ mol^{-1} and La-promoted 93 kJ mol^{-1} , respectively) was smaller compared to unpromoted $10\% \text{Co}/\text{Al}_2\text{O}_3$ catalyst (about 108 kJ mol^{-1}). The Langmuir-Hinshelwood rate expressions also suggested that both reactants ($\text{C}_2\text{H}_5\text{OH}$ and CO_2) were associatively adsorbed on single-site of catalyst with corresponding activation energy of about 106 kJ mol^{-1} . This study suggests that the syngas produced over Co-based catalysts with desirable H_2/CO ratios could be used directly in Fischer-Tropsch synthesis without the requirement of adjusting feedstock composition.

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LIST OF SYMBOLS

A	Pre-exponential factor
B	The line broadening at half the maximum intensity in radian (FWHM)
b	Inert solids fraction of catalyst bed
c	The adsorbate constant
C_{Ab}	Bulk gas phase concentration of component A
C_{AS}	Concentration of C_2H_5OH on the catalyst surface
C_D	Amorphous carbon percentage
C_p	Specific heat capacity of gases
C_{pg}	Specific heat capacity of feed gas mixture at constant pressure
C_α	Carbon formed via decomposition of hydrocarbon
C_β	Carbon formed via dissociation of CO
C_C	Graphitic carbon
C_γ	Carbides
C_v	Whisker-like or vermicular carbon
D	Dispersion
D_{eff}	Effective diffusivity
D_g	Diffusivity
d_p	Average particle diameter
E_A	Activation energy
F	Flow rate
h	Heat transfer coefficient
j_D	Colburn's mass transfer factor
k_c	Mass transfer coefficient
L_a	Size of crystallite

n_m	Number of molecules adsorbed
N	Avogadro's number
n	Reaction order
M_{ad}	The adsorbed molecular weight
P	Pressure of gas
Pr	Prandtl number
P_s	Saturation pressure of adsorbed gas
R	Universal gas constant
R_t	Reactor tube radius
r	Rate of production
r_{exp}	Reaction rate
r_p	Actual radius
R_p	Catalyst particle radius
S_A	Total surface area of catalyst
Sc	Schmidt number
T_b	Boiling point
t_{ads}	Adsorbed layer thickness
U	Superficial gas velocity
V_a	Volume of gas adsorbed
W_{cat}	Weight of the catalyst
y	Mole fraction of component gases
λ	Wavelength
θ	Bragg angle
ρ_b	Bulk density of catalyst bed
ρ_c	Catalyst pellet density
ρ_g	The gas mixture density
σ_c	Constriction factor

μ_g	Viscosity of the gas mixture
ω_p	Catalyst pellet porosity
$\tilde{\tau}$	Tortuosity
λ_p	Thermal conductivity of catalyst pellet
ε	Void fraction
ΔH	Heat of reaction
ΔG	Gibbs free energy

LIST OF ABBREVIATIONS

ATR	Autothermal reforming
BET	Brunauer-Emmett-Teller
CNF	carbon nanofilament
EDR	Ethanol dry reforming
EDX	Energy-dispersive x-ray
ESR	Ethanol steam reforming
FTS	Fischer-Tropsch synthesis
MARI	Most abundant reactive species
MDR	Methane dry reforming
MT	Metric ton
GC	Gas chromatography
GHSV	Gas hourly space velocity
GTL	Gas to liquid
O.D.	Outer diameter
OSRE	Oxidative steam reforming of ethanol
POX	Partial oxidation
SEM	Scanning electron microscopy
SR	Steam reforming
TCD	Thermal conductivity detector
TEM	Transmission electron microscopy
TPC	Temperature-programmed calcination
TPD	Temperature-programmed desorption
TPR	Temperature-programmed reduction
TOS	Time-on-stream
TPO	Temperature-programmed oxidation
TGA	Thermogravimetric analysis
WGS	Water-gas shift
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

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