CO_2-CH_4 REFORMING OVER LANTHANIDE PROMOTED COBALT/MESOPOROUS ALUMINA CATALYST FOR SYNGAS PRODUCTION

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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 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 CO_2-CH_4 telah menarik perhatian kerana teknologi ini mampu menukar gas yang menipiskan ozon yang tidak diingini, CO_2 dan CH_4 sebagai bahan suapan kepada syngas ekuimolar yang diinginkan untuk sintesis Fischer-Tropsch. Pada masa ini, masih ada cabaran dalam menghasilkan pemangkin yang sangat stabil dan aktif untuk pembaharuan CO_2-CH_4 serta ketahanan yang lebih baik terhadap pemendapan karbon. Oleh itu, idea utama kerja kami untuk menghasilkan sokongan mesopore alumina (MA) menggunakan Teknik pembentukan sendiri Bersama hidroterma (SAHA) sebelum diimpregnasi dengan Co. Kemudian, hubungan antara parameter operasi, seperti suhu (923-1073 K) dan tekanan separa reaktan (10-40 kPa) terhadap prestasi pemangkin dan pembentukan karbon dinilai dalam kerja ini, serta kinetik dan mekanisme bagi pemangkin 10%Co/MA dalam pembaharuan CO_2-CH_4. Kesan pengalak lantanida (lanthanum (La), cerium (Ce), ytrrium (Y) dan samarium (Sm)) dan kuantiti pengalak (1, 2, 3 dan 5wt.%) terhadap sifat fizikokimia pemangkin 10%Co/MA juga dikaji dalam projek ini. 10%Co/MA menunjukkan prestasi pemangkin yang bagus (penukaran CH_4 = 70.9%, penukaran CO_2 = 71.7% dan kadar penyahaktifan = 1.3%), disebabkan oleh penyebaran zarah Co yang baik kedalam pori MA, interaksi sokongan-logam yang kuat, dan kebolehan pengurungan MA. Penyebaran zarah Co pada sokongan MA terbukti bertambah baik setelah penggabungan pengalak, menghasilkan ukuran kristalit yang lebih kecil dan pengumpulan Co yang lebih rendah. Penukaran reaktan bertambah baik mengikut urutan YCo/MA > LaCo/MA > CeCo/MA > SmCo/MA > Co/MA, sementara jumlah pemendapan karbon dicatatkan dengan urutan Co/MA > SmCo/MA > LaCo/MA > CeCo/MA > YCo/MA. Selain itu, pemangkin YCo/MA mencapai prestasi pemangkin terbaik (penukaran CH_4 = 85.8%, penukaran CO_2 = 92.2%, Kadar penyahaktifan = 0.57%) dan memiliki pemendapan karbon terendah (7.02%) kerana penyebaran zarah Co yang bagus, saiz Co yang kecil dengan interaksi Co-MA yang kuat dan keupayaan penyimpanan oksigen yang lebih tinggi. Nisbah H_2/CO diperoleh dalam 0.78-0.86, sedikit lebih rendah daripada 1 akibat peralihan air-gas terbalik. Prestasi pemangkin unggul ditunjukkan oleh pemuatan 3wt.%Y_2O_3 (penukaran CH_4 = 85.8%, dan penukaran CO_2 = 90.5%), diikuti oleh 2wt.% > 5wt.% >1wt.% > 0wt.% muatan Y_2O_3. Hasil ini disebabkan sifat pemangkin yang bagus oleh 3%Y-10%Co/MA termasuk saiz zarah Co kecil, penyebaran Co tinggi, jumlah nisbah atom yang tinggi (Co/Al), dan jumlah kekosongan oksigen kisi yang tinggi. Lebihan kandungan Y_2O_3 (>3wt.%) menyebabkan halangan terhadap aktif Co yang tidak dapat dielakkan dan mengakibatkan penurunan prestasi pemangkin. Pemuatan 3wt.%Y_2O_3 mencatat karbon pemendapan paling rendah (7.0%) kerana kekosongan oksigen tertinggi (78.1%) berbanding 1, 2 dan 5wt.%Y_2O_3. Kesimpulannya, penggunaan sokongan MA dan penambahan penggalak Y_2O_3 (3wt. %) berkesan dalam meningkatkan prestasi Co dalam pembaharuan CO_2-CH_4 termasuk menghalang permendapan karbon diatas permukaan pemangkin berbanding penggalak yang lain (Ce, La, dan Sm) dan kandungan Y_2O_3 (1, 2, 5wt. %) disebabkan penambahbaikkan terhadap struktuk dan sifat fizikimia pemangkin.

ABSTRACT

CO_2-CH_4 reforming has caught significant attention since this technology is able to convert undesirable ozone-depleting gases, CO_2 and CH_4, as feedstocks into the desired equimolar syngas for Fisher-Tropsch synthesis. At present, there is still a challenge in developing the highly stable and active catalysts for CO_2-CH_4 reforming as well as better resistance to carbon deposition. Therefore, the main idea of our work is to synthesize mesoporous alumina (MA) support using self-assembly hydrothermal approach (SAHA) technique before being impregnated with cobalt (Co). Then, the relationship between operating parameters, such as reforming temperature (923–1073 K) and reactant partial pressure (10-40 kPa) on catalytic performance and coke formation was evaluated in this work. The effect of lanthanide promoters (lanthanum (La), cerium (Ce), yttrium (Y), and samarium (Sm)) and promoter loading (1, 2, 3, and 5wt.%) on the physicochemical properties of 10%Co/MA catalyst was also studied in this project. 10%Co/MA exhibited great catalytic performance (CH_4 conversion = 70.9%, CO_2 conversion = 71.7% and Rate of deactivation = 1.3%), credited to the well dispersed Co within pore MA, strong metal-support interaction, and MA confinement ability. The Co particle dispersion on MA support evidently improved after promoter incorporation, resulting in smaller crystallite size and lesser Co agglomeration. The reactant conversions improved in the order of YCo/MA > LaCo/MA > CeCo/MA > SmCo/MA > Co/MA, while the amount of carbon deposit was recorded with the sequence of Co/MA > SmCo/MA > LaCo/MA > CeCo/MA > YCo/MA. Additionally, YCo/MA catalyst attained the highest catalytic performance (CH_4 conversion = 85.8%, CO_2 conversion = 92.2%, Rate of deactivation = 0.57%) and possessed the lowest carbon deposition (7.02%) due to great Co dispersion, small Co particle size with strong Co-MA interaction and higher oxygen storage capacity. H_2/CO ratios were obtained within 0.78-0.86, which is slightly lower than 1 due to the reverse water-gas shift. A superior catalytic performance was shown by 3wt.% Y_2O_3 loading (CH_4 conversion = 85.8%, and CO_2 conversion = 90.5%), followed by 2wt.% > 5 wt.% > 1wt.% > 0 wt.% Y_2O_3 loading. This result was attributed to the favorable catalytic properties of 3%Y-10%Co/MA including small Co particle size, high Co dispersion, high amount of atomic ratio (Co/Al), and a high number of lattice oxygen vacancies. The excess Y_2O_3 addition (>3 wt.%) led to inevitably blocked Co active sites and resulted in decreasing catalytic performance. The 3wt.% Y_2O_3 promoter loading recorded the lowest carbon deposited (7.0%) due to the highest oxygen vacancies (78.1%) as compared to 1, 2 and 5 wt.% Y_2O_3. As a conclusion, the employment of MA support and incorporation of Y_2O_3 (3wt.) effectively boosted the Co performance in CO_2-CH_4 reforming along with suppressing the deposition of carbon on the catalyst surface as compared with other promoted catalysts (Ce, La, and Sm) and Y_2O_3 loadings (1, 2, 5wt.) owing to the improvement in catalysts structure and physicochemical attributes.

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LIST OF SYMBOLS

P_{CH_4}	Partial pressure of CH_4
P_{CO_2}	Partial pressure of CO_2
ΔG	The changes in the standard Gibbs free energy
n	Number of moles
D_d	Deactivation degree
wt%	weight percent
C_{Ab}	Bulk gas-phase concentration
c_p	Specific heat capacity
C_d	Carbon deposition
k_c	Mass transfer coefficient
h_w	Heat transfer coefficient
R	Ideal gas constant
μ	Viscosity
D_{eff}	Effective diffusivity
D_g	Diffusivity in mixture
E_A	Activation energy
$-\Delta H_r$	Heat of reaction
r	Rate of reaction
R_p	Catalyst particle radius
T	Temperature
V	Volume
ε	Void fraction on the catalyst bed
ρ_c	Density of catalyst pellet
ρ_b	Bulk density of catalyst bed
σ_c	Construction factor
$\tilde{\tau}$	Tortuosity
ω_p	Catalyst pellet porosity
n	Reaction order
j_D	Colburn's mass transfer factor
Sc	Schmidt number
U	Superficial gas velocity
$\dot{n}$	Molar flow rates
$W_{cat.}$	Weight of catalyst
β	full width at half maximum of peaks
λ	XRD wavelength
θ	Bragg angle
C_α	Adsorbed. atomic carbon (surface carbide)
C_β	Polymers, amorphous films
C_γ	Ni carbide (bulk)
C_v	Vermicular filaments/ whiskers
C_c	Graphite (crystalline) platelets film
X_i	Conversion

LIST OF ABBREVIATIONS

BET	Brunauer-Emmett-Teller
BP	British Petroleum
Ce	Cerium
Co	Cobalt
CTAB	Cetyl-trimethyl ammonium bromide
EISA	Evaporation-induced self-assembly
FTS	Fischer-Tropsch synthesis
FWHM	Full width at half maximum of peaks
GHSV	Gas hourly speed velocity
HRTEM	High-resolution transmission electron microscopy
I.D.	Inner diameter
Ir	Iridium
IUPAC	International Union of Pure and Applied Chemistry
IWI	Incipient wetness impregnation
JCPDS	Joint Committee on Powder Diffraction Standards
La	Lanthanum
MA	Mesoporous alumina
MCM-41	Mobil Composition of Matter No. 41
MSR	Methane steam reforming
n.a.	Not available
O.D.	Outer diameter
Pd	Palladium
PPO	Polypropylene oxide
Pt	Platinum
RBR	Reverse Boudouard reaction
Rh	Rhodium
Ru	Ruthenium
RWGS	Reverse water gas shift
SA	Surface area
SAHA	Self-assembly hydrothermal approach
SBA-15	Santa Barbara Amorphous-15
S:C	Steam to carbon ratio
SDA	Structure directing agent
SEM	Scanning electron microscopy
SIWI	Sequential incipient wetness impregnation
Sm	Samarium
SOFCs	Solid oxide fuel cells
TPR	Temperature-programmed reduction
TPO	Temperature-programmed oxidation
US	United States
WGS	Water gas shift
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

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