SYNTHESIS AND CHARACTERIZATION OF La-PROMOTED Ni/SBA-15 CATALYSTS FOR METHANE DRY REFORMING

OSAZE OMOREGBE

Master of Science

UNIVERSITI MALAYSIA PAHANG

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.

_____

(Supervisor's Signature)

Full Name : Dr Dai-Viet N. Vo

Position : Senior Lecturer

Date :

_____

(Co-supervisor's Signature)

Full Name : Dr Sumaiya Bt Zainal Abidin

Position : Senior Lecturer

Date :

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.

_____

(Author's Signature)

Full Name : Osaze Omoregbe

ID Number : MKC15016

Date : 07-06-2017

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Dedicated to all members of my family including my late aunt, Mrs Esther, for their love and support

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LIST OF SYMBOLS

β	Full width at half maximum (FWHM) measured in cps (count per second)
c	A constant characteristic of adsorbate (unitless)
C_{Ab}	Bulk gas-phase concentration of component A (mol m^{-3})
C_P	Specific heat capacity (J kg^{-1} K^{-1})
D_{eff}	Effective diffusivity (m^2 s^{-1})
μ_i	Viscosity of component *i* in gas mixture (Kg m^{-1} s^{-1})
E_A	Activation energy (J mol^{-1})
h	Heat transfer coefficient between gas mixture and catalyst (J m^{-2} s^{-1} K^{-1})
h_w	Heat transfer coefficient of reactor tube wall (J m^{-2} s^{-1} K^{-1})
j_D	Colburn's mass transfer factor (unitless)
k_c	Mass transfer coefficient (m s^{-1})
l_p	Crystallite size (m)
n_m	Number of molecules adsorbed (mol m^{-2})
N	Avogadro's number (mol^{-1})
n	Reaction order (unitless)
M_{ad}	Molecular weight of adsorbate (g)
P	Gas pressure (Pa)
Pr	Prandtl number (unitless)
P_s	Saturation pressure of adsorbed gas (Pa)
R	Universal gas constant (J mol^{-1} K^{-1})
$-r_{exp}$	Rate of reaction (mol g_{cat}^{-1} s^{-1})
R_p	Catalyst particle radius (m)
r_p	Actual radius (m)
d_p	Average catalyst particle diameter (m)
d_t	Reactor tube diameter (m)
S_A	Total surface area of sample (m^2)
Sc	Schmidt number (unitless)
T_b	Boiling point (K)
t_{ads}	Thickness of the adsorbed layer (m)
U	Superficial gas velocity (m s^{-1})
V	Volume of adsorbed gas (m^3)

$\dot{v}$	Volumetric flowrate ($m^3\ s^{-1}$)
W_{cat}	Weight of the catalyst (g)
λ	Wavelength (m)
λ_i	Thermal conductivity of component *i* (($J\ m^{-1}\ s^{-1}\ K^{-1}$)
θ	Bragg angle (°)
ρ_b	Bulk density of catalyst bed ($kg\ m^{-3}$)
ρ_g	Density of gas mixture ($kg\ m^{-3}$)
ρ_c	Density of catalyst pellet ($kg\ m^{-3}$)
ω_p	Catalyst pellet porosity (unitless)
σ_c	Construction factor (unitless)
$\tilde{\tau}$	Tortuosity (unitless)
ε	Void fraction (unitless)
β	Heating rate ($K\ s^{-1}$)
ΔH_r	Heat of reaction ($J\ mol^{-1}$)

LIST OF ABBREVIATIONS

ATR	Autothermal reforming
BET	Brunauer-Emmett-Teller
BR	Boudouard reaction
CGS	Carbon gasification by steam
CPOR	Catalytic partial oxidation reforming
CTS	Cool Tip Swirl burner
EPA	Environmental Protection Agency
FESEM	Field-emission scanning electron microscope
FTS	Fischer-Tropsch synthesis
GHSV	Gas hourly space velocity
I.D.	Inner diameter
LPG	Liquefied Petroleum Gas
MC	Methane cracking
MDR	Methane dry reforming
POXR	Partial oxidation reforming
RF	Radiative forcing
RBR	Reverse Boudouard reaction
RWGS	Reverse water–gas shift
SEM	Scanning electron microscopy
MSR	Methane steam reforming
TCD	Thermal conductivity detector
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
TPC	Temperature-programmed calcination
TPD	Temperature-programmed desorption
TPO	Temperature-programmed oxidation
TPR	Temperature-programmed reduction
XRD	X-ray diffraction

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ABSTRAK

Kajian ini mengkaji pengaruh parameter operasi termasuk tekanan separa bahan mentah dan suhu terhadap aktiviti pemangkin 10%Ni/SBA-15 dalam tindak balas pembaharuan kering metana (MDR). Selain itu, kesan penggalak La kepada prestasi pemangkin 10%Ni/SBA-15 MDR telah dikaji. Kedua-dua pemangkin 10%Ni/SBA-15 dan 3%La-10%Ni/SBA-15 telah disintesis melalui kaedah penjejalan secara basah di peringkat awal proses dan dinilai menggunakan kaedah BET, XRD, FESEM, TEM, FTIR, EDX, H_2-TPR, NH_3-TPD dan analisis Raman. Selepas itu, pemangkin yang telah disintesis diuji untuk MDR dalam reaktor tiub yang berisi kapas *quartz* di bawah tekanan atmosfera pada tekanan separa CO_2 dan CH_4 yang berbeza di antara 20-60 kPa dan suhu tindak balas yang berbeza di antara 923-1023 K. Kedua-dua pemangkin tanpa penggalak dan mengandungi penggalak La memiliki luas permukaan BET yang tinggi dalam lingkungan 303-445 $m^2 g^{-1}$. Keputusan FESEM dan TEM mendedahkan bahawa zarah Ni telah diagihkan di dalam pemangkin dengan penggalak La manakala sebahagian kelompok terdapat pada pemangkin tanpa penggalak 10%Ni/SBA-15. Penilaian aktiviti pemangkin 10%Ni/SBA-15 dalam MDR pada 923 K memberikan penukaran CH_4 dan penukaran CO_2 sebanyak 65.3% dan 70.3%, yang turun dalam masa 4 jam sepanjang dalam aliran pada perbezaan peratusan sebanyak 26.1% dan 17.6%, bagi setiap bahan mentah. Penyahaktifan diperhatikan bagi pemangkin tanpa penggalak sepanjang masa dalam aliran disebabkan oleh tindak balas Boudouard yang cenderung untuk berlaku yang membawa kepada pembentukan karbon. Walau bagaimanapun, aktiviti pemangkin 10%Ni/SBA-15 bertambah baik dengan kestabilan yang ketara apabila suhu tindak balas meningkat daripada 923 K kepada 1023 K pada peratusan kenaikan sebanyak 47.5% dan 39.6% bagi penukaran CH_4 dan CO_2 masing-masing. Peningkatan dalam aktiviti pemangkin 10%Ni/SBA-15 pada suhu yang tinggi telah disifatkan kepada penyerapan haba dalam tindak balas MDR dan tekanan daripada tindak balas Boudouard. Tambahan pula, penukaran CH_4 bagi pemangkin 10%Ni/SBA-15 meningkat daripada 91.1% kepada 98.8% apabila tekanan separa CO_2 (P_{CO_2}) dinaikkan dari 20 kPa 50 kPa. Apabila P_{CO_2} dinaikkan dari 50 kPa kepada 60 kPa, penukaran CH_4 menurun sedikit kepada 98.1%. Selain itu, penukaran CO_2 menurun daripada 94.4% kepada 58.7% apabila P_{CO_2} dinaikkan dari 20 kPa kepada 60 kPa. Begitu juga dengan nisbah H_2/CO yang menurun daripada 0.98 kepada 0.54 dengan kenaikan P_{CO_2} dari 20 kPa kepada 60 kPa yang secara logiknya berlaku kerana tindak balas peralihan air gas yang berbalik (RWGS) yang cenderung untuk berlaku. Menariknya, kedua-dua penukaran CO_2 dan penukaran CH_4 menurun dengan ketara daripada 94.4% kepada 76.3% dan 91.1% kepada 34.4%, masing-masing, apabila tekanan separa CH_4 dinaikkan daripada 20 kPa kepada 60 kPa. Trend yang berlaku ini disifatkan kepada pemendapan karbon yang cenderung untuk berlaku melalui penguraian CH_4 dalam keadaan CH_4 yang berlebihan. Ujian ketahanan pemangkin menunjukkan sedikit penurunan dalam aktiviti pemangkin tanpa penggalak 10% Ni/SBA-15 dalam tempoh 24 jam sepanjang dalam aliran dengan peratusan penyahaktifan sebanyak 8.7% dan 4.4% dari segi penukaran CH_4 dan penukaran CO_2, manakala penukaran CH_4 dan CO_2 untuk pemangkin dengan penggalak La 10%Ni/SBA-15 masing-masing secara relatif stabil pada 92.1% dan 94.7% dalam tempoh 24 jam sepanjang dalam aliran. Tambahan pula, kajian menunjukkan La loading optimum untuk aktiviti pemangkin yang terbaik adalah 3% berat. Post-reaksi analisis mendedahkan kehadiran dua jenis spesies karbon.

ABSTRACT

Methane dry reforming (MDR) is one of the practicable ways of synchronously converting two greenhouse gases (CO_2 and CH_4) into syngas which is a valuable feedstock for chemical processes such as Fischer–Tropsch synthesis, but the process suffers from catalyst deactivation caused by active metal sintering and carbon formation. Thus, this research investigated the influence of operating parameters including reactant partial pressure and temperature on the activity of 10%Ni/SBA-15 catalyst in MDR reaction. Additionally, the effect of La-promoter on the performance of 10%Ni/SBA-15 catalyst for MDR was studied. Both the 10%Ni/SBA-15 and 3%La-10%Ni/SBA-15 catalysts were synthesised by the incipient wetness impregnation method and characterised using BET surface area, XRD, FESEM, TEM, FTIR, EDX, H_2-TPR, NH_3-TPD, and Raman analyses. Subsequently, the synthesised catalysts were tested for MDR in a quartz fixed-bed tubular reactor under atmospheric pressure at varying CO_2 and CH_4 partial pressure of 20–60 kPa, and a reaction temperature of 923-1023 K. Both the unpromoted and La-promoted catalysts exhibited high BET surface area in the range of 303-445 m^2 g^{-1}. FESEM and TEM results revealed that the Ni particles were well distributed in the La-promoted catalyst while some clusters were formed on the unpromoted 10%Ni/SBA-15 catalyst. Evaluation of the catalytic activity of the 10%Ni/SBA-15 catalyst in MDR at 923 K gave CH_4 and CO_2 conversions of 65.3% and 70.3%, which declined within 4 h on-stream at percentage deviations of 26.1% and 17.6%, respectively. The observed deactivation of the unpromoted catalyst with time-on-stream was attributed to favoured Boudouard reaction leading to carbon formation. However, the 10%Ni/SBA-15 catalyst activity improved with appreciable stability as reaction temperature increased from 923 K to 1023 K at percentage increment of 47.5% and 39.6% in CH_4 and CO_2 conversions, respectively. The improvement in activity of 10%Ni/SBA-15 catalyst at high temperature was ascribed to the endothermicity of MDR reaction and the suppression of Boudouard reaction. Furthermore, CH_4 conversion of the 10%Ni/SBA-15 catalyst increased from 91.1% to 98.8% as the CO_2 partial pressure (P_{CO_2}) was raised from 20 kPa to 50 kPa. When P_{CO_2} was raised from 50 kPa to 60 kPa, CH_4 conversion declined slightly to 98.1%. On the other hand, CO_2 conversion decreased steadily from 94.4% to 58.7% when P_{CO_2} was raised from 20 kPa to 60 kPa. Similarly, the H_2/CO ratio declined from 0.98 to 0.54 with rising P_{CO_2} from 20 kPa to 60 kPa which is logically due to favoured reverse water-gas shift (RWGS) reaction. Interestingly, both CO_2 and CH_4 conversions decreased substantially from 94.4% to 76.3% and 91.1% to 34.4%, respectively, when CH_4 partial pressure was raised from 20 kPa to 60 kPa. These trends were ascribed to the favoured carbon deposition through CH_4 decomposition in excess CH_4 environment. Longevity tests showed slight decline in the activity of the unpromoted 10%Ni/SBA-15 catalyst within 24 h on-stream with percentage deactivation of 8.7% and 4.4% in terms of CH_4 and CO_2 conversions, whilst the CH_4 and CO_2 conversions for La-promoted 10%Ni/SBA-15 catalyst were relatively stable at 92.1% and 94.7%, respectively, within the 24 h on-stream. The study on the effect of La-promoter loading revealed that the optimum La loading, in terms of CO_2 and CH_4 conversions, was 3wt% La and above this loading, there was a significant decline in the catalyst activity reasonably due to pore blockage at higher La loading. Post-reaction analyses by XRD, TPO, SEM, TEM, and FTIR revealed the presence of two different types of carbonaceous species, viz., carbon filament (more reactive) and moss-like (less reactive) carbon.

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