STEAM REFORMING OF GASIFIED BIOMASS TAR FOR HYDROGEN PRODUCTION OVER NICKEL–DOLOMITE BASED CATALYST

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DEDICATION

This thesis is wholeheartedly dedicated to my beloved parents for their unconditional love, endless support and encouragement during the challenges of my graduate school and life.

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ABSTRACT

Catalytic steam reforming is a promising approach to address tar formation and improve hydrogen (H_2) production from biomass gasification. In this research, multi-compound tar model (phenol, toluene, naphthalene, and pyrene) was steam reformed for H_2 production over various types of 10 wt.% dolomite promoted 10 wt.% nickel based catalysts supported on alumina, lanthana, ceria, and zirconia. The research aims to synthesize nickel-dolomite catalyst for steam reforming of gasified biomass tar for optimum H_2 production. The catalysts were characterized by thermogravimetric analysis, temperature programmed reduction, temperature programmed desorption, nitrogen physisorption, and X-ray diffraction. The results showed that the addition of dolomite promoter to the catalysts strengthened the metal-support interaction and basicity of the catalyst. Steam reforming for catalyst screening was carried out at 700 ^{o}C with steam to carbon (S/C) molar ratio of 1 and gas hourly space velocity (GHSV) of 20,453 mL/h g_{cat}. The Ni/dolomite/La_2O_3 (NiDLa) catalyst displayed mesoporous structure, high reducibility, and basicity, which lead to superior carbon conversion to gas (77.66 mol%) and H_2 yield (66.20 mol%). In addition, spent NiDLa exhibited the lowest amount of filamentous coke (110 mg/g_{cat}) formation after 5 hours of reaction compared to the other catalysts investigated. Findings on effect of reaction condition revealed that higher temperature (> 750 ^{o}C), S/C ratio that is close to the stoichiometric value (1), and moderate GHSV (12,000 – 18,000 h^{-1}) can improve carbon conversion to gas and H_2 yield. The optimum conditions were found to be 775 ^{o}C of temperature, 1.02 of S/C molar ratio, and 14,648 h^{-1} of GHSV which resulted in 99.94 mol% of carbon conversion to gas and 82.84 mol% of H_2 yield. This finding is close to the predicted 98.96 mol% of carbon conversion to gas and 82.00 mol% of H_2 yield by response surface method.

ABSTRAK

Pembentukan semula stim bermangkin merupakan kaedah yang berpotensi untuk menangani pembentukan tar dan meningkatkan penghasilan hidrogen (H_2) daripada penggasan biojisim. Dalam kajian ini, model tar pelbagai sebatian (fenol, toluena, naftalena, dan pirena) telah digunakan untuk penghasilan H_2 melalui pembentukan semula stim dengan menggunakan pelbagai jenis 10 % berat nikel berasaskan mangkin yang digalakkan dengan 10 % berat dolomit di sokong alumina, lanthana, ceria, dan zirkonia. Kajian ini bertujuan untuk mengsintesis mangkin nikel-dolomit untuk pembentukan semula stim biojisim tar dan bergas bagi penghasilan H_2 yang optimum. Mangkin-mangkin dicirikan menggunakan analisis termogravimetrik, pengurangan pengaturcaraan suhu, nyaherapan pengaturcaraan suhu, pejerapan fizikal nitrogen, dan pembelaun sinar-X. Keputusan menunjukkan bahawa penambahan penggalak dolomit kepada mangkin memperkuatkan interaksi logam-sokongan dan meningkatkan sifat beralkali mangkin. Penapisan mangkin dijalankan melalui pembentukan semula stim pada suhu 700 ^{o}C, nisbah molar stim kepada karbon (S/C) 1, dan halaju ruang gas setiap jam (GHSV) 20,453 mL/h g_{cat}. Mangkin Ni/dolomit/La_2O_3 (NiDLa) mempunyai struktur mesoporous, kebolehturunan tinggi dan sifat beralkali tinggi menyebabkan penukaran karbon ke gas (77.66 mol%) dan hasil H_2 (66.20 mol%) yang unggul. Tambahan pula, mangkin NiDLa menyebabkan pembentukan filamen karbon kok yang paling rendah (110 mg/g_{cat}) selepas 5-jam tindakbalas berbanding dengan mangkin lain yang kaji. Dapatan kajian kesan keadaan tindakbalas menunjukkan bahawa suhu yang tinggi (> 750 ^{o}C), nisbah S/C yang hampir dengan nilai stoikiometri (1), dan GHSV yang sederhana (12,000 − 18,000 h^{-1}) bermanfaat untuk penukaran karbon kepada gas dan hasil H_2. Keadaan optimum didapati pada suhu 775 ^{o}C, nisbah molar S/C 1.02, dan GHSV 14,648 h^{-1} yang menghasilkan 99.94 mol% penukaran karbon ke gas dan 82.84 mol% hasil H_2. Dapatan ini menghampiri keputusan yang diramalkan oleh kaedah permukaan sambutan iaitu 98.96 mol% penukaran karbon ke gas dan 82.00 mol% hasil H_2.

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LIST OF ABBREVIATIONS

Al_2O_3	-	Alumina
ANOVA	-	Analysis of variance
ATR	-	Autothermal reforming
Au	-	Gold
BBD	-	Box-Behnken design
BET	-	Brunauer Emmett Teller
BJH	-	Barret-Joyner-Halenda
CaO	-	Calcium oxide
$CaMg(CO_3)_2$	-	Dolomite
CaO/MgO	-	Calcined state of dolomite
Ce	-	Cerium
CeO_2	-	Ceria
CH_4	-	Methane
CO	-	Carbon monoxide
Co	-	Cobalt
CO_2	-	Carbon dioxide
CO_2-TPD	-	Temperature programmed desorption of carbon dioxide
Cu	-	Copper
CuO	-	Copper oxide
$C_4H_6NiO_4$	-	Nickel acetate
C_6H_6O	-	Phenol
C_7H_8	-	Toluene
$C_{10}H_8$	-	Naphthalene
$C_{16}H_{10}$	-	Pyrene
DOE	-	Design of Experiment
Fe	-	Iron
FE-SEM	-	Field emission scanning electron microscopy
Fe_2O_3	-	Iron oxide
GC-TCD	-	Gas chromatography with thermal conductivity detector

GHSV	-	Gas hourly space velocity
HC	-	Hydrocarbon
He	-	Helium
H_2	-	Hydrogen
H_2-TPR	-	Temperature programmed reduction of hydrogen
IEA	-	International Energy Agency
K_2O	-	Potassium oxide
La_2O_3	-	Lanthana
Mg	-	Magnesium
MgO	-	Magnesium oxide
Mn	-	Manganese
Ni	-	Nickel
$NiCl_2$	-	Nickel chloride
NiD	-	Ni/dolomite
NiDAl	-	Ni/dolomite/Al_2O_3
NiDCe	-	Ni/dolomite/CeO_2
NiDLa	-	Ni/dolomite/La_2O_3
NiDZr	-	Ni/dolomite/ZrO_2
NiO	-	Nickel oxide
$Ni(NO_3)_2$	-	Nickel nitrate
$Ni(NO_3)_2 \cdot 6H_2O$	-	Nickel nitrate hexahydrate
NiS	-	Nickel sulfide
O_2	-	Oxygen
Pd	-	Palladium
PLOT	-	Porous layer open tubular
POX	-	Partial oxidation
Pt	-	Platinum
Rh	-	Rhodium
RSM	-	Response surface methodology
Ru	-	Rutherium
SBA-15	-	Well-order hexagonal mesoporous silica
SEM	-	Scanning electron microscopy
SiC	-	Silicon carbide
SD	-	Standard deviation

SiO	-	Silica oxide
SR	-	Steam reforming
S/C	-	Steam/carbon
TGA	-	Thermogravimetric analysis
TiO_2	-	Titanium dioxide
TPO	-	Temperature programmed oxidation
VP-SEM	-	Variable-pressure scanning electron microscopy
WGS	-	Water gas shift
WHSV	-	Weight hourly space velocity
XRD	-	X-ray diffraction
ZrO_2	-	Zirconia
Zn	-	Zinc

LIST OF SYMBOLS

D	-	Dispersion
d_{Ni}	-	Crystallite size e
d_p	-	Pore size
p/p_o	-	Relative pressure
R^2	-	Correlation coefficients value
S_{BET}	-	Brunauer Emmett Teller surface area
V_{total}	-	Total pore volume
X	-	Gaseous product (H_2, CO, CO_2, CH_4)
x	-	Coded values of variable, temperature (x_1), S/C ratio (x_2), space time (x_3)
x	-	Conversion fraction of, tar (x_{tar}), phenol (x_{phenol}), toluene ($x_{toluene}$), naphthalene ($x_{naphthalene}$), pyrene (x_{pyrene})
y	-	Predicted response, conversion (y_1), H_2 yield (y_2)
θ	-	Diffraction peak angle
λ	-	Wavelength of incident x-ray
β	-	Coefficient, constant (β_0), linear (β_i), quadratic (β_{ii}), interaction (β_{ij})
β	-	Half width of diffraction peak
ε	-	Statistical error term

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CHAPTER 1

INTRODUCTION

1.1   Background Study

The rapid depletion of fossil fuels and associated environmental issues such as global warming and climate change are becoming global concerns. However, the worldwide energy demand is continuously increasing at an alarming rate year after year. According to the finding of International Energy Agency (IEA), global energy demand increased by 2.1 % in 2017 which is more than twice the rate of previous year [1]. With regard to electricity generation in 2017, fossil fuel was responsible for 81 % of the total world energy as compared to other energy sources including renewables and nuclear [1]. Globally, fossil fuel-related carbon dioxide (CO_2) emissions showed an increment of 1.4 % in 2017 [1]. Consequently, the upsurge in fossil fuel demand infers higher socio-economic and environmental cost. Therefore, the exploitation of alternative energy sources to replace conventional fossil fuels is indispensable.

Currently, hydrogen (H_2) gas is considered as crucial commodity to sustainably generate electricity in 21st century. Among the existing fuels and energy carriers, H_2 has the highest energy density and its energy yield is up to 122 kJ/kg [2]. Its energy yield is approximately 2.75 times higher than most hydrocarbon (HC) fuels [2]. By employing H_2 gas, the crises of supply disruption and the impact of greenhouse gas emissions associated with conventional fossil fuel-based energy systems can be avoided. Basically, H_2 utilization generates only water vapor as a by-product with zero greenhouse gas emissions, during H_2 gas combustion with oxygen (O_2) in internal engine or electrochemically converting H_2 gas in fuel cell [3].

However, H_2 does not occur naturally on earth but commonly exists as part of other substances in nature such as water, alcohol, natural gas, biomass, coal, and HC. Consequently, it can only be obtained from H_2-containing resources through chemical reaction processes. In recent years, numerous technologies including thermochemical conversion [4-6], electrolysis [7], and photolysis [8] are under investigation for H_2 production. Among these possible options, biomass gasification is considered a promising and economical technology [9, 10]. Biomass gasification technology encompasses thermochemical process that converts organic substances from agriculture and forestry into syngas rich in H_2 and carbon monoxide (CO) along with a small amount of CO_2 and methane (CH_4).

The presence of impurities in the syngas such as tar, ash, nitrogen-, and sulfur-containing compounds is highly unacceptable especially tar. This is because tar is a complex mixture of condensable aromatic and oxygenated HCs that condenses at low temperature and subsequently lead to process-related problems. For instance, filter clogging, plugging of downstream equipment, and coke deposition on the downstream catalyst [11, 12]. More importantly, the formation of tar represents a decrease in conversion efficiency since biomass is converted to tar instead of syngas. Hence, the physical removal and further reduction/oxidation of tar is essential in order to improve the production of syngas.

Generally, reforming techniques are categorized into 3 types: steam reforming (SR), partial oxidation (POX), and autothermal reforming (ATR). However, SR is reported to have the superior H_2 yield [13]. It is the most developed and attractive technique providing a conversion mechanism for liquid HCs. This is because SR offers higher concentration of H_2 in the reformate, which is about 70 to 80 vol.% on a dry basis compared to other reforming technologies (40-50 vol.%) [14]. In addition, it produces about 100,000 Nm^3/hr of H_2 gas on an industrial scale [15]. For comparison the resulting cost of H_2 by conventional steam methane reforming is less than \$ 2.00/kg at comparable natural gas in year 2017 [16]. Furthermore, based on the higher heating value, SR of CH_4 had achieved up to 85% of thermal efficiency. Whereas, only 60 to 75% of thermal efficiency was achieved by both ATR and POX [17].

In the present study, tar which is the major undesired by-product derived from biomass gasification was converted into H_2 gas by SR over dolomite promoted Ni-based catalysts. The components of tar model selected are the major chemical composition contained in the gasified biomass tar as reported by Singh et al. [18]. The representatives selected are phenol for phenolic and heterocyclic HCs, toluene for one-ring aromatic HCs, naphthalene for two-ring aromatic HCs and pyrene for four-ring and higher HCs.

Among the various existing catalysts, Ni-based catalysts have been extensively employed for SR because of their low price and pronounced performance in O-H, C-H, and C-C bonds rupture [19, 20] along with the additional activity for water gas shift (WGS) reaction [19, 21]. However, Ni-based catalysts are prone to active sites deactivation as a result of coke formation [22]. Dolomite which serves as the promoter was added to catalyst to suppress the deposition of coke and improve the catalytic activity per unit surface area. Furthermore, dolomite plays an essential role in CO_2 sorption to promote the water-gas shift (WGS) reaction, which results in the production of H_2 rich fuel gas [23]. The oxide supports that were used in this study include alumina (Al_2O_3), lanthana (La_2O_3), ceria (CeO_2), and zirconia (ZrO_2).

1.2   Problem Statement

The presence of unacceptable levels of tar in the syngas is the primary challenge of biomass gasification. This implies that less biomass is converted into syngas and consequently reduce the total H_2 production. Furthermore, the production of tar in biomass gasification also contributed to syngas end-use problems. These problems typically include blockages and corrosion in downstream filters, fuel line, engine nozzles, and turbines [11]. Typically, tar content in syngas produced from biomass gasification ranges from 0.5 to 100 g/Nm^3, depending on the biomass feedstock, operating condition, and gasifier [18, 24]. However, the tolerance limit of

tar in syngas for various applications is 1, 5, and 100 mg/Nm^3 in fuel cells, gas turbines, and internal combustion engines, respectively [24]. Although physical separation considerably removes tar from product gas, it has great potential to create secondary pollution. Thus, SR is a promising technique to convert the separated tar into valuable H_2 rich gas.

To date, most research studies deal with the SR based on an individual tar model compound, typically phenol, benzene, toluene or naphthalene over a variety of supported metal catalysts. Nevertheless, the composition of real biomass tar is complex and each component possesses a mutual influence on SR performance. Josuinkas et al., [25] investigate the effect of feedstock composition on catalytic performance during SR. They reported that the mixture of toluene and naphthalene altered the catalytic activity and reduce the feed conversion as compared to individual feed compound. Therefore, a research that can reflect the real condition of biomass tar SR is necessary. In the present research, the tar model was made up of phenol, toluene, naphthalene, and pyrene.

Owing to the presence of catalyst provides a more efficient SR, several kinds of catalytic reforming have been developed. However, the extraordinary difficulty is to obtaining a high stability of catalyst which is selective for H_2 and also resistance to coke deposition [22, 26]. Ni-based catalysts have been extensively used in steam reforming but it is prone to deactivation of its active sites by coke formation [22, 27]. Recently, it has been reported that addition of alkaline earth metal oxides such as MgO and CaO as a promoter could neutralise the acidity of the catalyst and improve steam-carbon reaction, which in turn increases the coke suppression rate and catalytic stability [28-30]. Low cost and abundance naturally occurring minerals such as dolomite that contains both CaO and MgO. In addition, dolomite also give a positive impact on H_2 production by adsorbing CO_2 to shift the thermodynamic equilibrium of WGS reaction towards H_2 production [23]. Therefore, this study is conducted to develop oxide supported Ni-based catalysts using dolomite as promoter for SR of multi-compound tar model. To the best of my knowledge, there is no study has been reported utilizing a dolomite as promoter.

The SR parameters such as temperature, steam to carbon (S/C) ratio, and space velocity have been reported as major factors that influent the tar conversion and H_2 yield [22, 31, 32]. Besides, previous study also mentioned that the effect of operating parameters on tar conversion and H_2 yield is associated with catalyst used. For instance, Furusawa et al., [33] found that SR over Ni/MgO operated at high S/C ratio produced low amount of H_2 as compared to Ni/Al_2O_3. Therefore, one of the aim of this research is to study the effect of the operating parameters on carbon conversion to gas and H_2 yield over the studied catalyst. In order to ensure the optimum carbon conversion to gas and H_2 yielded from the tar SR over the studied catalyst, the optimization of operating parameters using response surface methodology (RSM) should be taken into account.

1.3 Objectives of Study

The aim of this research is to develop Ni-based catalyst for high carbon conversion to gas with maximum H_2 yield via SR of multi-component gasified biomass tar model. To achieve this aim, the following objectives have been planned:

(a) To synthesize and characterize the Ni-based catalysts on several catalyst supports (Al_2O_3, La_2O_3, CeO_2, and ZrO_2) and dolomite as a catalyst promoter for hydrogen production via steam reforming of gasified biomass tar.

(b) To determine the effect of steam reforming parameters on hydrogen production in terms of temperature, steam to carbon ratio, and gas hourly space velocity.

(c)   To optimize the operating parameters for hydrogen production via steam reforming of gasified biomass tar.

1.4   Scope of Study

In order to achieve the research objectives, the scope of this research was designed and listed as follows:

(a)   Catalytic SR was adopted to convert gasified biomass tar into H_2 rich gas over dolomite promoted Ni-based catalyst. The tar model was made up of 15 wt.% phenol, 50 wt.% toluene, 30 wt.% naphthalene, and 5 wt.% pyrene. The selected components are the major chemicals contained in the gasified biomass tar as reported by Singh et al., [18] and their composition are represents to their chemical family.

(b)   5 types of 10 wt.% dolomite promoted 10 wt.% Ni-based catalysts were prepared using co-impregnation method. The oxide supports include Al_2O_3, La_2O_3, CeO_2, and ZrO_2. The catalysts were designated as Ni/dolomite (NiD), Ni/dolomite/Al_2O_3 (NiDAl), Ni/dolomite/La_2O_3 (NiDLa), Ni/dolomite/CeO_2 (NiDCe), and Ni/dolomite/ZrO_2 (NiDZr). The selection of 10 wt% Ni loading is due the best performance reported by previous literature as discussed in Section 2.7.1. Generally, the promoter loading is not more than 5 wt.% [34-36]. Since dolomite used contains 57.3 wt.% $CaCO_3$ and 41.8 wt.% $MgCO_3$, 10 wt.% of dolomite loading was selected in this research.

(c)   All of the catalysts were characterized by thermogravimetric analysis (TGA) for thermal stability, temperature programmed reduction of hydrogen (H_2-TPR) for reducibility of active metal, temperature programmed desorption of carbon dioxide (CO_2-TPD) for basicity properties, nitrogen physisorption for textural properties, and X-ray diffraction (XRD) for crystalline properties.

(d)   For catalyst screening, 0.8 g of catalyst was reformed in a fixed bed reactor at 700 °C, S/C molar ratio of 1, and gas hourly space velocity (GHSV) of 20,453 mL/h g_{cat} under atmospheric pressure.

(e)   The most promising catalyst was selected based on the catalyst screening, and was used to study the effect of SR parameters on carbon conversion to gas and hydrogen yield. The operating parameters that influence the SR of gasified biomass tar were considered in this study. The chosen parameters where temperature (500-900 °C), S/C molar ratio (0.5-2.5), and GHSV (8,000-22,000 h^{-1}).

(f)   The parameters were further optimized by RSM in order to obtain maximum carbon conversion to gas with highest H_2 yield. A second-order factorial design called Box-Behnken design (BBD) was adopted to design the experiment by varying the SR parameters. The variables studied were temperature (600-800 °C), S/C moalr ratio (1-2), and GHSV (12,000-22000 h^{-1}), while the response were carbon conversion to gas and H_2 yield. The range of these three variables was determined based on the parametric study.

(g)   The coke formation of spent catalysts was evaluated by TGA, XRD, variable-pressure scanning electron microscopy (VP-SEM) or field emission scanning microscopy (FE-SEM).

1.5   Significance of Research

Following a great development and deployment in biomass gasification for H_2 production, there is a need to explore the SR of tar over a reliable catalyst. To date, there are not many studies focused on SR of multi-compounds tar model. Therefore, through this research, the real conditions of biomass tar SR can be reflected by employing a tar model made up of main representative of gasified biomass tar. This is importance in prediction and understanding the catalytic performance in real biomass tar SR process.

Catalyst with high activity, high thermal stability, high coking resistance and high mechanical strength are key elements in the reaction. To the best of my knowledge, no study is reported in the open literature using dolomite promoted Ni-based catalysts on catalyst support of Al_2O_3, La_2O_3, CeO_2, and ZrO_2 in SR of gasified biomass tar. In this research, dolomite was selected as an attractive catalyst promoter because of its numerous advantages such as environmental friendly, readily available, economic feasibility of material, high thermal stability, relatively mechanically resistant and higher resilience to catalyst poison [37]. Therefore, the catalyst in this research has a lower cost compared to noble metal based or promoted catalysts, which paves way for its application in large scale SR process.

Generally, reaction conditions also play crucial role in H_2 production in SR of gasified biomass tar. From this research, an optimal reaction conditions in terms of temperature, S/C ratio, and GHSV over the most promising catalyst was provided. This result can be applied in industrial SR of gasified biomass tar. By doing so, the new developed catalyst of this research has a great potential as an alternative catalyst for commercial SR of gasified biomass tar. Besides, this work would benefit in a number of particular areas such as reduction of energy consumption, catalyst usage, and also minimise expenses of feedstock. Thus, the result from the present research is expected to expand the frontier of knowledge in the field of SR catalyst.

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