UNIVERSITI PUTRA MALAYSIA

ENHANCEMENT OF DRY REFORMING OF METHANE FOR SYNGAS PRODUCTION OVER M/$Mg_{1-X}M'_xO$ CATALYSTS (M=Ni, Pd, AND Pt., M'=Ce^{3+},Ce^{4+}, AND Ni^{2+})

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

January 2016

Dedicated to

My parent's soul

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

ENHANCEMENT OF DRY REFORMING OF METHANE FOR SYNGAS PRODUCTION OVER M/$Mg_{1-X}M'_xO$ CATALYSTS, (M=Ni, Pd, AND Pt, M'=Ce^{3+}, Ce^{4+}, AND Ni^{2+})

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January 2016

Chairman: Professor Taufiq Yap Yun Hin, PhD
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In this study, the conversion of methane and carbon dioxide to synthesis gas using dry reforming of methane over Ni, Pd, and Pt/MgO catalysts in different concentrations of Ce^{3+}, $Ce^{4+}$and Ni^{2+} was prepared via the co-precipitation of Mg and Ce nitrates, followed by impregnation with 1 wt.% each of Ni, Pd, and Pt metals to form Pt/$Mg_{1-x}Ni_xO$, Pd/$Mg_{1-x}Ce^{3+}{}_xO$, Pd/$Mg_{1-x}Ce^{4+}{}_xO$ and Ni,Pd,Pt/$Mg_{1-x}Ce_xO$ catalysts.

The goal of this study was to prepare a catalyst with high activity and selectivity which prevent the carbon deposition onto the catalyst during the syngas production. The performance of metal / magnesia-promoter catalysts in syngas production and the factors influencing carbon deposition during reaction were also investigated. The produced catalysts were characterized using various kinds of analytical techniques.

The Ni,Pd,Pt/Mg_{1-x} $Ce_x{}^{3+}O$ catalyst with cubic structure was synthesized using the co-precipitation method which showed good selectivity for dry reforming of methane reaction with CO_2 and CH_4 conversion rates of 99% and 84%, respectively, at ratio CO_2:CH_4, 1:1 at 900 °C. They also showed good thermal stability for the first 200h. and also, great potential for use in fuel processing. This catalyst also increased the activity & stability of DRM reaction by adding a small concentration of O_2, which resulted in a combination of combustion and reforming reaction that made the overall process thermo-neutral and helped in limiting carbon formation.

Abstrak tesis yang dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PENAMBAHBAIKAN PROSES PEMBENTUKAN SEMULA METANA KERING UNTUK PENGHASILAN SYNGAS DENGAN MENGGUNAKAN MANGKIN M/$Mg_{1-X}M'_xO$, (M=Ni, Pd, DAN Pt, M'=Ce^{3+}, Ce^{4+}, DAN Ni^{2+})

Oleh

FARIS ABDULRIDHA JASSIM ALDOGHACHI

Pengerusi : Profesor Taufiq Yap Yun Hin, PhD
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Dalam kajian ini, penukaran gas metana dan karbon dioksida kepada gas sintesis melalui proses pembentukan semula metana kering dengan menggunakan mangkin Ni, Pd dan Pt/MgO dalam kepekatan Ce^{3+}, Ce^{4+} dan Ni^{2+} berbeza disediakan dengan kaedah ko-mendakan Mg dan Ce nitrat, diikuti dengan kaedah pengisitepuan bagi setiap 1wt% logam Ni, Pd, dan Pt untuk membentuk mangkin Pt/$Mg_{1-x}Ni_xO$, Pd/$Mg_{1-x}Ce^{3+}{}_xO$, Pd/$Mg_{1-x}Ce^{4+}{}_xO$ dan Ni,Pd,Pt/$Mg_{1-x}Ce^{3+}{}_xO$.

Tujuan kajian ini adalah untuk menyediakan mangkin yang mempunyai aktiviti dan pemilihan yang tinggi yang mampu menghalang pemendapan karbon di permukaan mangkin semasa penghasilan syngas. Prestasi mangkin logam/magnesia dalam penghasilan syngas dan faktor-faktor yang mempengaruhi pemendapan karbon semasa tindakbalas juga dikaji. Mangkin yang dihasilkan telah dicirikan dengan menggunakan pelbagai jenis teknik analisis.

Mangkin Ni,Pd,Pt/$Mg_{1-x}Ce_x{}^{3+}O$ dengan struktur kubik yang telah disintesis menggunakan kaedah ko-mendakan menunjukkan pemilihan dan kestabilan terma yang baik untuk tindakbalas pembentukan semula metana kering dan juga mempunyai potensi yang baik dalam proses penghasilan bahan api. Mangkin ini juga menunjukkan peningkatan aktiviti dan pemilihan tindak balas pembentukan semula metana kering dengan sedikit penambahan kepekatan O_2, menghasilkan kombinasi tindak balas pembakaran dan pembentukan semula yang mewakili keseluruhan proses neutral-terma dan membantu menghadkan pembentukan karbon.

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I certify that a Thesis Examination Committee has met on 21 January 2016 to conduct the final examination of Faris Abdulridha Jassim on his thesis entitled "Enhancement of Dry Reforming of Methane for Syngas Production Over $M/Mg_{1-x}M'_xO$ Catalysts (M=Ni, Pd, and Pt, $M'=Ce^{3+}$, Ce^{4+}, and Ni^{2+})" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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CHAPTER 1

INTRODUCTION

1.1. Background of the study

Numerous research is conducted to convert methane into liquid fuels or higher hydrocarbons. Bradford & Vannice (1999) were investigating the production of methanol, formaldehyde, propanol, benzene and other aromatics by direct oxidative conversion of methane. Unfortunately, all the aforementioned processes produce low yields or they are not recommended on an industrial-scale (Claridge *et al.*, 1998). Today, various technologies are available for the production of synthesis gas (syngas) from natural gas (NG) (Li *et al.*, 2011). This gas is a building block for valuable liquid fuels and chemicals such as Fischer-Tropsch oil, methanol and dimethyl ether (Lunsford, 2000). The three processes that draw industrial attention are steam reforming of methane (Eq. 1.1), partial oxidation of methane with oxygen or air (Eq. 1.2) and dry reforming of methane with carbon dioxide (Eq. 1.3) (Asencios & Assaf 2013).

$$CH_4 + H_2O \rightarrow CO + 3H_2 \qquad \Delta H^{\circ}_{298K} = 225.0\ kJ/mol \tag{1.1}$$

$$CH_4 + 1/2\, O_2 \rightarrow CO + 2H_2 \qquad \Delta H^{\circ}_{298K} = -22.6\ kJ/mol \tag{1.2}$$

$$CH_4 + CO_2 \rightarrow 2\, CO + 2H_2 \qquad \Delta H^{\circ}_{298K} = 247.0\ kJ/mol \tag{1.3}$$

The technologies involved in reforming of methane through steam or partial oxidation of methane with oxygen or air are well-established with both advantages and disadvantages (Djinović *et al.*, 2012). As for the steam reforming of methane, it produces a higher ratio of syngas (H_2/CO =3) (Gangadharan *et al.*, 2012) compared to the requirement for Fischer-Tropsch or methanol synthesis (H_2/CO =2) (Olah *et al.*, 2012). This process consumes much energy due to its endothermic nature; hence it is a highly capital intensive investment (Nieva *et al.*, 2014). Since a higher H_2O/CH_4 ratio is required for the production of higher yields of hydrogen, the process of steam reforming of methane is not energetically favorable. This leads to the deactivation of the catalyst (Carvalho, 2009). Moreover, steam reforming is prone to corrosion problems. As a result, it requires a desulfurization unit (Djinović *et al.*, 2012). The partial oxidation of methane is suitable for the production of heavier hydrocarbons and naphtha (Larimi & Alavi, 2012). The benefits of this process include high rates of conversion, high selectivity and brief residence time (Ruckenstein & Hu, 1999). There are drawbacks in the exothermic nature of the reaction. For one, it induces hot spots on catalyst arising from poor heat removal rate and the other is the difficulty in the control of operation (Asencios & Assaf, 2013). Desulfurization unit is not required in the partial oxidation of methane, but a cryogenic unit is necessary for the separation of oxygen from the air (Djinović *et al.*, 2012). Dry reforming of methane offers valuable environmental benefits that include the utilization of biogas (Kohn *et al.*, 2014), removal of greenhouse gases (GHG) (methane and carbon dioxide) and conversion of NG with high carbon dioxide content to valuable syngas (Lunsford, 2000). The dry reforming of methane produces a lower syngas ratio ($H_2/CO = 1$), that is appropriate for the synthesis of oxygenated chemicals (Wurzel *et al.*, 2000) and hydrocarbons from the synthesis of Fischer-Tropsch. Syngas, from dry reforming is also considered for storage of solar or nuclear energy (Levy *et al.*, 1992) through the chemical energy transmission system (CETS). Solar energy which can convert feed gases (methane and carbon dioxide) to syngas can be exported to places where energy sources are scarce. The energy stored in the syngas is released by the backward reaction for utilization as an energy source (Fraenkel *et al.*, 1986).

In Malaysia and Indonesia, the most abundant bio-renewable resource is the palm oil biomass. It has a great potential for the sustainable production of chemicals and fuels. An effluent from palm oil mills, also referred to as palm oil mill effluent (POME), is always considered as highly polluted wastewater (Chin *et al.*, 2013). Digestion, an aerobically treatment is widely used in the oil palm industry as a primary treatment for POME. However, biogas, is flared off. Biogas contains two GHG; about 60-70% of methane and 30-40% of carbon dioxide with traces of hydrogen sulfide (Chin *et al.*, 2013). Methane and carbon dioxide, are the key contributors to climate-change (Noor *et al.*, 2013). The concentrations of methane and carbon dioxide in 2011 were recorded at 1.813 ppm and 390.5 ppm, respectively, based on the assessment of the environment by the World Meteorological Organization (WMO) (Tarasova *et al.*, 2011). Even though the concentration of methane in the atmosphere was recorded to be lower than carbon dioxide, (Warmuzinski, 2008), it has kicked in about 20 % of overall global warming (Popa *et al.*, 2014). There are two sources of methane emission that have been identified. The first source is from naturally occurring activities such as termite, grasslands, coal beds, lakes, wetlands and wildfires while the second source stems from anthropogenic activities such as landfills, oil and gas processing, wastewater treatment plants, coal mining, rice production, cattle ranching and agricultural activities (Yusuf *et al.*, 2012). It has been estimated that the production of methane amounts to 6875 million metric tons, CO_2 equivalent (MMT CO_2) from all anthropogenic sources in 2010 (Johari *et al.*, 2012). In fact, methane, a major component of NG contains low balances of other hydrocarbons that comprise of ethane, propane, and butane. NG also contains inert diluents such as molecular nitrogen (N_2) and carbon dioxide (Karavalakis *et al.*, 2012).

By looking at the negative impact of methane and carbon dioxide, their concentrations must be decreased to prevent the concentration of greenhouse gases that result in adverse environmental conditions and the increase of temperature.

1.2. Reaction chemistry and carbon deposition

Syngas that is produced from the dry reforming of methane is affected by the simultaneous occurrence of reverse water gas shift (RWGS) reaction (Eq. 1.4) resulting in a syngas ratio that is less than unity (Bradford & Vannice, 1999).

$$CO_2 + H_2 \rightarrow CO + H_2O \qquad \Delta H^{\circ}_{298K} = 41.0\ kJ/mol \tag{1.4}$$

Despite being environmentally beneficial, the dry reforming of methane suffers from the problem of carbon deposition that is caused by methane decomposition (Eq. 1.5) and the disproportionation of CO (Boudouard reaction) (Eq. 1.6).

$$CH_4 \rightarrow C + 2H_2 \qquad \Delta H^{\circ}_{298K} = 75.0\ kJ/mol \tag{1.5}$$

$$2CO \rightarrow C + CO_2 \qquad \Delta H^{\circ}_{298K} = -172.0\ kJ/mol \tag{1.6}$$

The tendency towards carbon deposition can be estimated by the ratio of O/C and H/C in the feed gas. The higher tendency towards carbon deposition is observed in lower O/C and H/C ratio (Li *et al.*, 2011), which is the case in the dry reforming of methane (CH_4/CO_2 =1/1) with O/C =1 and H/C =2. However, the reverse was observed for the steam reforming of methane (CH_4/H_2O =1/1) whereby the readings are high for O/C =1 and H/C = 6. Similarly, the partial oxidation of methane ($CH_4/0.5O_2$) shows a rather high ratio, O/C =1 and H/C =4. Considering the aforementioned ratios, it is clear that the tendency towards carbon deposition is higher for dry reforming of methane compared to steam reforming and partial oxidation of methane (Edwards & Maitra, 1995). The thermodynamic studies on dry reforming reaction revealed that spontaneous reaction cannot be achieved for temperature below 640 °C (Tsang *et al.*, 1995) and for side reactions (Eq. 1.5) and (Eq. 1.6) that take place at a significant rate of between 633 °C and 700 °C. Therefore, a higher temperature of (T≥700 °C) is employed to minimize the effects of side reactions, which can lead to reactions or blockages and reductions in activities (San José-Alonso *et al.*, 2013) (Zhang *et al.*, 2007).

1.3. Problem Statement

There is little to no previous research study of data of reaction kinetics on product formation using the dry reforming of methane over Ni,Pd,Pt/MgO with promoter NiO, CeO_2 and Ce_2O_3 catalyst.

1.4. Scope of study

The scope of study is the development and characterization of a catalyst, Ni,Pd,Pt/MgO with promoter NiO, CeO_2 and Ce_2O_3 in the dry reforming of methane. The technology of dry reforming is commonly employed for hydrocarbon species to obtain lower H_2:CO ratios. This dry reforming technology utilizes biogas from the biodiesel industry for the production of syngas. This can resolve the problems of greenhouse gases as well as reduce the market price of syngas, which is currently a costly gas. At the same time, the production of syngas is an alternative in mitigating the over-dependence on fossil fuel consumption.

The contribution of this study lies in the development of a new basic catalyst, Ni,Pd,Pt/MgO with promoter NiO, CeO_2 and Ce_2O_3. This new catalyst is designed to be more coke-resistance due to the addition of a promoter which is an alkaline compound.

Also, CO_2 and CH_4 as a feedstock for the dry reforming of methane reduce the effects of the global warming phenomenon.

1.5. Objectives

The general objective of this study was to determine the appropriate method for the preparation of Ni,Pd,Pt/$Mg_{1-x}Ce^{3+}{}_xO$ catalysts to enhance the selectivity, activity, and stability in DRM reaction. The specific objective are to:

1. Synthesize different series of catalyst by co-precipitation method.

2. Characterize the catalyst prepared using FESEM, BET, XRD, XRF, TGA, FTIR, TPR, TEM, XPS and TPO, that attempt to relate physicochemical attributes towards the carbon coking tendency of catalysts and production of syngas.

3. Evaluate the effects of reactant feed ratio, CO_2 and CH_4 feed ratio in the range of 1:1 to 1:2, and the effects of temperature that is in the range of 700 ^oC to 900 ^oC on the production of syngas as well as the conversion of CO_2 and CH_4.

4. Evaluate the stability of the reaction of catalysts over 200 h (8 days) and the physicochemical properties of the used catalyst using TPO, FESEM, TEM, FTIR and XRD.

5. Improve the conversion of methane by using a low stream of Oxygen on the dry reforming of methane

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LIST OF PUBLICATIONS

Al-Doghachi, F. A., Islam, A., Zainal, Z., Saiman, M. I., Embong, Z., & Taufiq-Yap, Y. H. (2016). High Coke-Resistance Pt/$Mg_{1-x}Ni_xO$ Catalyst for Dry Reforming of Methane. *PloS one*, ***11***(1).

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