

SYNGAS PRODUCTION FROM METHANE  
DRY REFORMING OVER CeO<sub>2</sub> PROMOTED  
Ni/CaFe<sub>2</sub>O<sub>4</sub> CATALYST

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DOCTOR OF PHILOSOPHY

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## **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 Doctor of Philosophy.

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## **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.

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## ABSTRACT

There is a growing interest in methane dry reforming as a technological route for producing synthesis gas (syngas) due to the benefits of utilizing methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>), the two principal components of greenhouse gases as feed stocks to Fischer-Tropsch synthesis of liquid hydrocarbons and oxygenates. Nevertheless, one of the major constraints of the methane dry reforming reaction is catalyst deactivation by sintering and carbon deposition. Approaches such as the synthesis of metal-based catalysts on suitable supports and the use of appropriate promoters have been reported to enhance catalyst resistance to deactivation and also improve their activities. This study is aimed at investigating the effect of using calcium ferrite (CaFe<sub>2</sub>O<sub>4</sub>) as support and cerium oxide (CeO<sub>2</sub>) as promoter on the activities and stabilities of Ni catalyst in methane dry reforming. Both the unpromoted and the CeO<sub>2</sub>-promoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalysts were synthesized using wet impregnation method. The as-synthesized catalysts were subsequently characterized for their physicochemical properties by thermogravimetric analysis (TGA), X-ray diffraction (XRD), N<sub>2</sub>-physisorption analysis, Scanning electron microscope-Energy dispersive X-ray (SEM-EDX), Transmission electron microscopy (TEM), NH<sub>3</sub> and CO<sub>2</sub> temperature programmed desorption (TPD-NH<sub>3</sub> and TPD-CO<sub>2</sub>) and H<sub>2</sub>-temperature programmed reduction (TPR). The catalytic activities and stabilities of the catalysts were investigated in methane dry reforming to syngas in a fixed bed stainless steel reaction at temperature range of 973-1073 K, under atmospheric pressure. In addition, the modelling and optimization study of the syngas production by methane dry reforming over the Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst was conducted. The catalysts characterization shows that both the promoted and the unpromoted catalysts with pore diameter of 3.2 nm and 3.08 nm, respectively possessed mesopores (pore diameter > 2 nm) suitable for catalytic reaction such methane dry reforming. Moreover, the H<sub>2</sub>-TPR as well as the TPD-NH<sub>3</sub> and TPD-CO<sub>2</sub> show good reducibility of the nickel oxides and the presence of both acidic and basic sites. The CeO<sub>2</sub> promoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst displayed better catalytic activity compared to the unpromoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst due to the preservation of the catalyst active site by the promotional effect of the CeO<sub>2</sub>. At feed (CH<sub>4</sub>: CO<sub>2</sub>) ratio of 1, and reaction temperature of 1073 K, the highest CH<sub>4</sub> and CO<sub>2</sub> conversions of 92.5%, and 91.2% respectively were obtained compared to 90.4% and 83.5% values obtained as CH<sub>4</sub> and CO<sub>2</sub> conversions from the methane dry reforming over the unpromoted catalyst. The deactivation of the unpromoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst can be attributed to carbon deposition evidenced from the characterization of the used catalysts. The fitting of the rate data obtained from the methane dry reforming over the CeO<sub>2</sub>-promoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst in LH model yielded an activation energy of 22.58 kJ/mol, which is comparable to those obtained from the literature. This implies that the kinetic model that fit the data is based on single site dissociative adsorption of both CH<sub>4</sub> and CO<sub>2</sub> with bimolecular surface reaction. The response surface methodology (RSM) studies showed that the process parameters (reaction temperature, feed ratio and GHSV) significantly influence the four responses (CH<sub>4</sub> conversion, CO<sub>2</sub> conversion, H<sub>2</sub> yield and CO yield). Moreover, the interactions between these parameters were also found to significantly affect the responses. The optimization studies over the CeO<sub>2</sub>-promoted Ni/CaFe<sub>2</sub>O<sub>4</sub> catalyst using RSM revealed that optimum conditions (Temperature = 1019 K, feed ratio= 0.98, GHSV= 28000 mL g<sup>-1</sup> h<sup>-1</sup>) resulted in the maximum values of 93.76, 92.45, 77.54 and 79.17% for the CH<sub>4</sub> conversion, CO<sub>2</sub> conversion, CO yield, and H<sub>2</sub> yield, respectively.

## ABSTRAK

Teknologi pembaharuan kering dijadikan keutamaan dalam penghasilan (singas) (campuran gas antara karbon monoksida dan hidrogen) pada masa hadapan. In kerana banyak kebaikan menggunakan mentana ( $\text{CH}_4$ ) dan karbon dioksida ( $\text{CO}_2$ ) sebagai stok suapan untuk sintesis Fischer-Tropsch cecair hidrokarbon dan pengoksigenan. Walaubagaimanapun, salah satu halangan utama adalah pendeaktifan pemangkin oleh pensinteran dan pengendapan karbon. Pendekatan seperti sintesis pemangkin berasaskan logam untuk sokongan dan penggunaan penggalak yang sesuai dapat meningkatkan ketahanan pendeaktifan pemangkin dan meningkatkan aktivitinya. Tujuan kajian ini ialah untuk menyiasat kesan penggunaan kalsium ferit ( $\text{CaFeO}_4$ ) sebagai sokongan dan penggalak cerium oksida ( $\text{CeO}_2$ ) terhadap aktiviti dan kestabilan pemangkin Ni dalam pembaharuan semula mentana kering. Pengalok dan bukan pengalok  $\text{CeO}_2$  pemangkin Ni/ $\text{CaFeO}_4$  disintesis menggunakan kaedah pengisitepuan basah. Psikokimia pemangkin disintesis telah dikaji dengan menggunakan analisis termogravimetrik (TGA), Pembelahan sinar-X (XRD), analisis  $\text{N}_2$ - penjerapan fizik, Memindai mikroskop electron-X-ray dispersif tenaga (SEM-EDX), Mikroskop elektron penghantaran (TEM),  $\text{NH}_3$  dan  $\text{CO}_2$ - desorpsi diprogram suhu ( $\text{CO}_2$ -TPD) dan  $\text{H}_2$ - pengurangan suhu yang diprogramkan (TPR). Kestabilan dan tindak balas aktiviti untuk kedua-dua pemangkin telah dikaji dalam tindak balas keluli tahan karat lapisan tetap pada suhu antara 973-1173 K, dibawah tekanan atmosfera. Selain itu, kajian pemodelan dan pengoptimuman untuk penghasilan singas daripada pembaharuan mentana kering terhadap pemangkin Ni/ $\text{CaFe}_2\text{O}_4$  telah dijalankan. Keputusan faktor pencirian menunjukkan kedua-dua penggalak dan bukan penggalak pemangkin dengan saiz diameter 3.2 dan 3.08 nm, adalah sesuai sebagai pemangkin untuk tindak balas seperti pembaharuan mentana kering. Tambahan lagi,  $\text{H}_2$ -TPR dan  $\text{CO}_2/\text{NH}_3$  TPD menunjukkan kebolehturunan yang baik terhadap nikel oksida dengan kehadiran kedua-dua keasidan dan kebesan. Ujian kestabilan menunjukkan penggunaan  $\text{CeO}_2$  sebagai penggalak telah meningkatkan kestabilan pemangkin Ni/ $\text{CaFe}_2\text{O}_4$  terhadap masa dalam arus oleh penggasan daripada pembentukan karbon semasa pengaktifan  $\text{CH}_4$  dengan oksigen keluar dari  $\text{CeO}_2$ . Tambahan lagi, penggalak  $\text{CeO}_2$  untuk pemangkin Ni/ $\text{CaFe}_2\text{O}_4$  menunjukkan aktiviti tindak balas yang lebih baik berbanding dengan bukan penggalak pemangkin Ni/ $\text{CaFe}_2\text{O}_4$  kerana pemuliharaan tapak aktif oleh kesan promosi penggalak  $\text{CeO}_2$ . Nisbah suapan ( $\text{CH}_4$ :  $\text{CO}_2$ ) adalah 1, suhu tindak balas adalah 1173K, penukaran tertinggi  $\text{CH}_4$  dan  $\text{CO}_2$  adalah 92.5% dan 91.2% telah diperolehi berbanding dengan 90.4% dan 83.5% diperolehi sebagai penukaran  $\text{CH}_4$  dan  $\text{CO}_2$  daripada pembaharuan semula mentana kering terhadap pemangkin bukan penggalak. pendeaktifan pemangkin bukan penggalak Ni/ $\text{CaFe}_2\text{O}_4$  menyumbang kepada pengendapan karbon dan dapat dibuktikan melalui ujian faktor pecirian pemangkin. Data yang diperolehi dari kajian bagi pemangkin penggalak Ni/ $\text{CaFe}_2\text{O}_4$  dalam model LH telah menghasilkan tenaga pengaktifan sebanyak 22.58 kJ/mol berbanding data yang diperolehi dari *literature*. Kajian metodologi permukaan tindak balas (RSM) menunjukkan bahawa parameter proses (suhu tindak balas, nisbah makanan dan GHSV) mempengaruhi empat tindak balas (penukaran  $\text{CH}_4$ , penukaran  $\text{CO}_2$ , hasil  $\text{H}_2$  dan hasil CO). Selain itu, interaksi antara parameter ini juga didapati memberi kesan yang signifikan kepada respons. Kajian pengoptimuman terhadap  $\text{CeO}_2$  untuk pemangkin Ni/ $\text{CaFe}_2\text{O}_4$  pemangkin menggunakan kaedah RSM memperoleh keadaan optimum (Suhu = 1019 K, nisbah suapan = 0.98 GHSV= 28000 mL  $\text{g}^{-1} \text{h}^{-1}$ ) dalam nilai yang maksimum 93.76, 92.45, 77.54 dan 79.17% untuk penukaran  $\text{CH}_4$ , penukaran  $\text{CO}_2$ , hasil CO dan  $\text{H}_2$ .

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## LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ATR	Autothermal Reforming
BBD	Box-Behnken Design
BET	Brunauer-Emmett-Teller
BJH	Berreth-Joyner-Halenda
CCD	Central Composite Design
CO	Carbon Monoxide
DMD	Doehlert Matrix Design
DRM	Dry Reforming of Methane
EDX	Energy Dispersive X-ray Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FTS	Fischer Tropsch Synthesis
GTL	Gas To Liquid Technology
JCPDS	Joint Committee on Powder Diffraction Standard
MFC	Mass Flow Controller
PBD	Plackett Burman Design
POM	Partial Oxidation of Methane
RSM	Response Surface Methodology
RWGS	Reverse Water Gas Shift
SEM	Scanning Electron Microscopy
SMR	Steam Methane Reforming
TCD	Thermal Conductivity Detector
TEM	Transmission Electron Microscopy
TGA	Thermal Gravimetry Analysis
WGS	Water Gas Shift
XRD	X-Ray Diffraction
XPS	X-Ray Photoelectron Spectroscopy

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